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Laboratory Research

Applicability of Exposure Reciprocity Law for Fast Polymerization of Restorative Composites Containing Various Photoinitiating Systems

A Tichy • P Bradna

Clinical Relevance

The irradiation time of composite materials should not be shortened based on the exposure reciprocity law. Irradiation times shorter than 10 seconds increase the risk of insufficient polymerization at the bottom surface of a composite increment even when a high-power LED lamp is used.

SUMMARY

Objectives: The exposure reciprocity law (ERL) has been used to calculate the optimal irradiation time of dental composites. This study examined the applicability of ERL for fast polymerization of restorative composites containing various photoinitiating systems using a high-power multi-peak light-emitting diode (LED) lamp.

Methods: Three commercial composites differing in photoinitiating systems were tested: Filtek Ultimate Universal Restorative (FU) with a camphorquinone-amine (CQ-A) photoinitiating system, Tetric EvoCeram (TEC) with CQ-A and (2,4,6-trimethylbenzoyl)phosphine oxide (TPO), and Estelite Σ Quick (ESQ) with CQ and a radical amplified photopolymerization (RAP) initiator. Specimens 2-mm thick were polymerized using a high-power multipeak LED lamp (Valo) at 3 pairs of radiant exposures (referred to as low, moderate, and high) ranging from 15.8-26.7 J/cm^2. They were achieved by different combinations of irradiation time (5-20 seconds) and irradiance (1300-2980 mW/cm^2) as determined with a calibrated spectrometer. Knoop microhardness was measured 1, 24, and 168 hours after polymerization on specimen top (irradiated) and bottom surfaces to characterize the degree of polymerization. The results were

*Antonin Tichy, DDS, Institute of Dental Medicine, First Faculty of Medicine, Charles University and General University Hospital in Prague, Prague, Czech Republic

Pavel Bradna, MA, PhD, Institute of Dental Medicine, First Faculty of Medicine, Charles University and General University Hospital in Prague, Prague, Czech Republic

*Corresponding author: Karlovo namesti 32, Prague, 121 11, Czech Republic; e-mail: antonin.tichy@lf1.cuni.cz

http://doi.org/10.2341/20-112-L

statistically analyzed using a three-way analysis of variance and Tukey's post hoc tests, α = 0.05.

Results: Microhardness increased with radiant exposure and except for ESQ, top-surface microhardness was significantly higher than that on bottom surfaces. Combinations of high irradiance and short irradiation time significantly increased the top-surface microhardness of TEC at low and moderate radiant exposures, and the bottom-surface microhardness of FU at a low radiant exposure. In contrast, the microhardness of ESQ on both surfaces at high radiant exposure increased significantly when low irradiance and long irradiation time were used. With all tested composites, bottom-surface microhardness obtained at low radiant exposure was below 80% of the maximum top-surface microhardness, indicating insufficient polymerization.

Conclusion: Combinations of irradiance and irradiation time had a significant effect on microhardness, which was affected by photoinitiators and the optical properties of composites as well as spectral characteristics of the polymerization lamp. Therefore, ERL cannot be universally applied for the calculation of optimal composite irradiation time. Despite high irradiance, fast polymerization led to insufficient bottom-surface microhardness, suggesting the necessity to also characterize the degree of polymerization on the bottom surfaces of composite increments when assessing the validity of ERL.

INTRODUCTION

The shortening and simplification of composite restoration procedures is one of the main goals of contemporary dental materials research. Significant progress has been made with the development of self-etch adhesives that have a reduced number of application steps, which has decreased the time necessary for composite placement and the risk of errors compared with traditional etch-and-rinse systems. Further acceleration was accomplished by optimizing composite optical properties and the development of high-power light-emitting diode (LED) polymerization lamps. In addition, more efficient photoinitiating systems than camphorquinone-amine (CQ-A), such as (2,4,6-trimethylbenzoyl)phosphine oxide (TPO) or dibenzoyl germanium compounds[1-3] were incorporated in some products. This progress enabled the polymerization of so-called bulk-fill composites in 4-5 mm increments or the reduction of irradiation time of the standard 2-mm increments to 10 or even less than 5 seconds.

The theoretical basis for the reduction of irradiation time using high-power polymerization lamps is the validity of the exposure reciprocity law (ERL). ERL states that the degree of monomer double-bond conversion (DC) in light-cured composites depends on radiant exposure E (J/cm^2), ie, light energy delivered to the composite surface in the region of photoinitiator absorption wavelengths. Radiant exposure is controlled by 2 independent variables, irradiation time t (seconds) and irradiance I (mW/cm^2), which is the radiant flux incident on cm^2 of the composite surface per second. Based on this definition, the same DC could be achieved at a given radiant exposure either through a shorter irradiation time and proportionally increased irradiance, or a prolonged irradiation time and lower irradiance. However, ERL has its limitations in complex free-radical polymerization of (meth)acrylic monomers. Using simplified models of photoinitiated polymerization, it was derived that radiant exposure should correspond to irradiance to the 0.5-1 power, depending on the termination mechanism.[4,5]

The validity of ERL would allow clinicians and manufacturers to simply estimate the required irradiation time for various irradiances. Several studies[1,6-22] used infrared spectroscopy[1,5-9,12-14,16,17,19-22] or mechanical properties such as hardness[18,19,21] and elastic modulus[9,10,12,16,18,20] to verify ERL. Some of them confirmed its validity,[6-9,13,15,21] but often at clinically inappropriately long irradiation times and low irradiances. Other studies, however, reported that ERL validity depends on photoinitiating systems,[1,16] fillers,[1,14] or composite viscosity.[4,14] They showed a violation of ERL at short irradiation times combined with high irradiance, especially for composites with CQ-A initiating systems.[1,5,10-12,14,16,20] In such a case, the application of ERL could result in insufficient polymerization of the composites, which would adversely affect their mechanical, aesthetic, and biological properties. The majority of these aforementioned results were achieved using quartz tungsten halogen, plasma arc, or low-power LED polymerization lamps. Although modern high-power multipeak LED lamps were used in some recent studies,[18-21] their effect on the validity of ERL with fast curing composites containing various photoinitiating systems has not been fully clarified.

To examine ERL validity, several conditions should be met. Firstly, it is necessary to determine irradiance of the specimen using a calibrated spectrometer. In addition, if composite materials with various photoinitiating systems are polymerized using multipeak LED lamps, it is important to measure not only the total irradiance but also irradiance at wavelengths corresponding to each peak to determine the effective light energy delivered in the light absorption range of the photoinitiators used. Secondly, the DC should be determined using, eg, infrared spectroscopy or a hardness measurement[23] on both the top (irradiated) surface and the bottom surface, because the incident light is substantially attenuated when passing through a composite. Thirdly, all measurements should be performed not only immediately after irradiation but also when the composite properties stabilize, as postirradiation polymerization[24,25] may alter outcomes.

The aim of this study was to investigate whether ERL could be applied to the polymerization of commercial composite materials activated by various photoinitiators and recommended for fast polymerization using a multipeak LED lamp. The first null hypothesis stated that the microhardness of tested composites measured on the top and bottom surfaces would not depend on radiant exposure, while the second null hypothesis assumed that microhardness at each radiant exposure would not depend on polymerization conditions, ie, combinations of irradiation time and irradiance, regardless of the composite material and photoinitiators used.

METHODS AND MATERIALS

Composite materials included the nanocomposite Filtek Ultimate Universal Restorative (FU, shade A2 dentin; 3M Oral Care, St. Paul, MN, USA), the nanohybrid composite Tetric EvoCeram (TEC, shade A2; Ivoclar Vivadent, Schaan, Liechtenstein), and the "supra-nano-filled" composite Estelite Σ Quick (ESQ, shade A2; Tokuyama Dental, Tokyo, Japan). According to the manufacturers, polymerization of FU is initiated by a CQ-A system, TEC contains a mixture of CQ-A with TPO, and ESQ is based on CQ and a newly developed radical amplified photopolymerization (RAP) initiator that allows a faster and more effective polymerization.[26,27] The composition of these materials is summarized in Table 1.

Irradiance Measurement

A corded multipeak LED polymerization lamp (Valo; Ultradent Products, South Jordan, UT, USA) was used in this study. Valo offers polymerization in 3 modes: standard, high power, and plasma emulation with claimed light intensities[28] of 1000, 1400, and 3200 mW/cm^2, respectively (Table 2). Irradiance measurements in each mode were performed using a USB2000+ spectrometer connected via an optical fiber with a CC-3 cosine corrector (all Ocean Optics, Dunedin, FL, USA). Its 3.9-mm diffuser diameter corresponded to the diameter of composite specimens. Before the measurement, the system was calibrated with a traceable

Table 1: *An Overview of Composite Materials Used*

Composite (Abbreviation; Manufacturer)	Monomers	Fillers	Filler (Load)	Photoinitiating System	Shade (Batch Number)
Filtek Ultimate Universal Restorative (FU; 3M ESPE, St. Paul, MN, USA)	Bis-GMA, UDMA, Bis-EMA, TEGDMA, PEGDMA	SiO_2 and ZrO_2 nanoparticles and their agglomerates	63.3 vol% (78.5 wt%)	CQ-amine	A2 Dentin (N747514)
Tetric EvoCeram (TEC; Ivoclar Vivadent, Schaan, Liechtenstein)	UDMA, Bis-GMA, Bis-EMA	Ba glass, YF_3, oxide mix, prepolymerized filler	53-55 vol% (75-76 wt%)	CQ-amine TPO	A2 (U26271)
Estelite Σ Quick (ESQ; Tokuyama Dental, Tokyo, Japan)	Bis-GMA, TEGDMA	SiO_2 and ZrO_2 particles, composite filler	71 vol% (82 wt%)	CQ RAP initiator	A2 (E654)

Abbreviations: Bis-EMA, ethoxylated bisphenol-A glycol dimethacrylate; Bis-GMA, bisphenol-A-glycidyl methacrylate; CQ: camphorquinone; PEGDMA, polyethylene glycol dimethacrylate; RAP: radical amplified photopolymerization; SiO_2, silicon dioxide; TEGDMA, triethyleneglycol dimethacrylate; TPO, diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide; UDMA, urethane dimethacrylate; YF_3, yttrium(III) fluoride; ZrO_2, zirconium dioxide.

Table 2: *Comparison of Valo Light Intensity Declared by the Manufacturer and Irradiance Measured with a Cosine Corrector Under the Conditions of Specimen Polymerization, ie, Through a 1-mm Thick Microscope Slide and Transparent Plastic Foil*

Polymerization Mode	**Declared Light Intensity (mW/cm²)**	**Measured Irradiance Mean ± SD (mW/cm²)**		
		Total (380-515 nm)	**380-420 nm**	**420-515 nm**
Standard	1000	1300 ± 30	195 ± 5 (15.0%)	1105 ± 12 (85.0%)
High power	1400	1950 ± 28	333 ± 7 (17.1%)	1617 ± 10 (82.9%)
Plasma emulation	3200	2980 ± 30	613 ± 16 (20.6%)	2367 ± 15 (79.4%)

Abbreviation: SD, standard deviation.

light source HL-3P-CAL (Ocean Optics, Dunedin, FL, USA). The data were processed using the SpectraSuite Ocean Optics software. Irradiance was measured in the full range of wavelengths (380-515 nm) as defined by ISO/TS 10650:2019,[29] and in the violet (380-420 nm) and blue (420-515 nm) ranges, activating TPO and CQ, respectively. The measurements were performed 5 times per each mode under specimen polymerization conditions, ie, through a 1-mm thick microscope slide and transparent plastic foil. For all measurements, Valo was carefully centered over the cosine corrector using a custom-made guide and fixed using clamps to avoid any change in its alignment with the cosine corrector. The transmittance of composite specimens was calculated as the ratio of irradiance measured with and without a fully polymerized composite disc (6 mm diameter, 2 mm thickness) placed between the microscope slide and the cosine corrector. The Valo built-in timing intervals for each polymerization mode were measured 10 times with a calibrated stopwatch (Table 3).

Specimen Preparation and Microhardness Measurement

For each composite material, 6 experimental groups of cylindrical specimens (n=5) were prepared in Teflon molds (4 mm diameter, 2 mm thickness). The molds were placed on a microscope slide, laid on white filtration paper, and covered with a transparent plastic foil. The molds were slightly overfilled in 1 increment, covered with another transparent foil and microscope slide, and pressed with a finger to remove excess material. Polymerization at 6 predetermined modes was performed using Valo, with the tip placed concentrically and perpendicularly to the specimen's surface. Approximately 30 minutes after polymerization, top (irradiated) and bottom surfaces were slightly ground under water using P1000, P2500, and P4000 silicon carbide grinding papers and polished using the 3 μm MetaDi II diamond polishing paste on a nylon polishing cloth followed by the suspension of 1 μm MicroPolish II alumina oxide particles on the MicroCloth polishing cloth (all Buehler, Lake Bluff, IL, USA). The specimens were stored in the dark at room temperature.

Table 3: *Polymerization Conditions: Irradiance, Irradiation Time and Radiant Exposure*

Polymerization Setup	**Irradiance (mW/cm²)**	**Declared Irradiation Time**	**Real Irradiation Time(s)**	**Radiant Exposure (J/cm²) (Uncertainty Interval)**
2980/5	2980	5 s (3 s + 2 s)	5.3	15.8 (14.7-16.9) a
1950/8	1950	8 s (2×4 s)	8.5	16.6 (15.4-17.8) a
2980/6	2980	6 s (2×3 s)	6.5	19.4 (18.0-20.8) b
1950/10	1950	10 s (4 s + 2×3 s)	10.6	20.7 (19.2-22.2) b
1950/12	1950	12 s (3×4 s)	12.8	25.0 (23.2-26.8) c
1300/20	1300	20 s	20.5	26.7 (24.7-28.7) c

The uncertainty range of radiant exposures, which corresponds to a 95% confidence interval was calculated as the square root of the sum of squared standard deviations of irradiance (0.3%-0.7%), irradiation time (2%), uncertainties of the stopwatch (0.03%), and the traceable light source (3%) multiplied by 2. Different lowercase letters indicate statistically significant differences between radiant exposures.

Knoop microhardness was measured after 1, 24, and 168 hours (IndentaMet 1600-1105D; Buehler) at 5 locations on both the top and bottom surfaces of the specimen with a 25-g load and 5-second dwell time. The first location was in the center of the specimen, and the other locations on the perimeter in north, south, east, and west positions, approximately 1.5 mm from the middle of the specimen. With TEC and ESQ containing large prepolymerized filler particles of a size comparable with the length of indentations (up to approximately 100 μm), 2 or more indentations outside these particles had to be performed to obtain results varying less than 10%. If more indentations were performed at a single location, their arithmetic mean was calculated. The microhardness values from each location on a surface were then averaged to obtain mean surface microhardness, which was processed statistically.

Scanning Electron Microscopy Analysis

The structure of the composite materials was characterized using a scanning electron microscope ([SEM] JSM 5500-LV; Jeol Inc, Tokyo, Japan) in the backscatter electron mode. The composite surface was polished to a mirror gloss, as stated before, dried for 1 week at room temperature, and sputter coated with a thin layer of gold (JFC-1200 Fine Coater, Jeol Inc).

Statistical Analysis

The statistical analyses of microhardness data measured 1, 24, and 168 hours after irradiation were performed using a three-way analysis of variance (ANOVA), with factors of radiant exposure, postirradiation time, and specimen surface. Tukey's post hoc tests were used for pairwise comparisons. The analyses were performed at $\alpha = 0.05$ using the Statistica software (StatSoft 12, Tulsa, OK, USA). The expanded uncertainty of radiant exposures, which corresponded to a 95% confidence interval, was calculated as a square root of the sum of squared standard deviations of irradiance (0.3%-0.7%), irradiation time (2%), uncertainties of the stopwatch (0.03%), and the traceable light source (3%)[30] multiplied by 2. Differences between radiant exposures were assumed to be statistically significant if their values ± expanded uncertainty did not overlap.

RESULTS

Irradiance

Table 2 summarizes the light power intensities of Valo as declared by the manufacturer and irradiances measured with a cosine corrector under our experimental conditions in the wavelength range of 380-515 nm. The irradiances in standard, high power, and plasma emulation modes were 1300, 1950, and 2980 mW/cm^2, respectively. Based on the irradiances and irradiation times (Table 3), composite specimens were polymerized at clinically applicable conditions: 1300 mW/cm^2 for 20 seconds (referred to as 1300/20); 1950 mW/cm^2 for 12 seconds, 10 seconds, or 8 seconds (1950/12, 1950/10, and 1950/8, respectively); and 2980 mW/cm^2 for 6 seconds or 5 seconds (2980/6 and 2980/5, respectively). Radiant exposures corresponding to these polymerization conditions ranged from 15.8-26.7 J/cm^2. Given the 7.2%-7.4% expanded uncertainty in their determination, 3 pairs of statistically nonsignificantly different radiant exposures referred to as low (15.8 and 16.6 J/cm^2), moderate (19.4 and 20.7 J/cm^2), and high (25.0 and 26.7 J/cm^2) were achieved (Table 3). Due to the preset polymerization modes of Valo, the radiant exposures could not be matched more closely, which would have been optimal for the verification of ERL.

Table 2 shows that the radiant flux of individual diodes did not change proportionally with increasing irradiance but in favor of violet light. In the standard mode, violet light equaled 15% of total irradiance, while in the high power mode it rose to 17.1% and in the plasma emulation mode it increased to 20.6%. After passing through the composite specimens, the intensity of light decreased significantly, as shown in Table 4. The least amount of light was transmitted through FU (approximately 3% of the incident light), followed by TEC with 12% and ESQ with 13%. The attenuation was stronger in the violet region compared with the blue region. In the case of TEC, only ~1.5% of violet light was transmitted, while the transmittance of ESQ and FU for violet light was less than 1%.

Composite Microstructure

The SEM analysis revealed that FU contained a mixture of round polydisperse particles (up to approximately 5 μm in diameter), which are probably agglomerates of nanoparticles (Figure 1A). In contrast, large irregular particles (up to 30-50 μm) with a structure resembling microhybrid composites were observed in TEC (Figure 1B). Even larger filler particles (up to 100 μm) of a heterogeneous structure were found in ESQ (Figure 1C). Their amount seemed to be higher compared with that of TEC, and their distribution in the bulk material was less uniform.

Microhardness

In TEC and ESQ, the microhardness of large prepolymerized filler particles (70-80 KHN for TEC and 80-95 KHN for ESQ) was significantly higher than that of the surrounding composite (up to 51 KHN for

Table 4: *Irradiance Measured Through a 2-mm Thick Disc of Tested Composites (Mean±SD)*[a]

Polymerization mode	**Total (380-515 nm)**		**Violet region (380-420 nm)**		**Blue region (420-515 nm)**	
	Irradiance (mW/cm²)	**Transmittance (%)**	**Irradiance (mW/cm²)**	**Transmittance (%)**	**Irradiance (mW/cm²)**	**Transmittance (%)**
Filtek Ultimate Universal Restorative						
Standard	38 ± 2	2.9	0	0.0	38 ± 2	3.4
High power	55 ± 1	2.8	0	0.0	55 ± 1	3.4
Plasma emulation	91 ± 1	3.1	0.7 ± 0.5	0.1	90 ± 1	3.8
Tetric EvoCeram						
Standard	150 ± 9	11.5	2.6 ± 0.2	1.3	147 ± 8	13.3
High power	226 ± 4	11.6	4.9 ± 0.3	1.3	221 ± 4	13.7
Plasma emulation	380 ± 9	12.8	9.4 ± 0.1	1.5	371 ± 9	15.7
Estelite Σ Quick						
Standard	172 ± 3	13.2	0.1 ± 0.1	0.0	172 ± 3	15.5
High power	245 ± 5	12.6	1.2 ± 0.1	0.4	244 ± 5	15.1
Plasma emulation	405 ± 9	13.6	3.7 ± 0.6	0.6	401 ± 8	17.0

[a] *Transmittance was calculated as a ratio of irradiance measured with the disc to irradiance without the disc (see Table 2).*

both TEC and ESQ). If the Knoop indenter interfered with these particles, microhardness scatter increased markedly, which led to the loss of measurement sensitivity to the extent of matrix polymerization. For that reason, the tip of the Knoop indenter was directed outside these particles.

Tables 5-7 present top and bottom surface microhardness of the tested materials after 1, 24, and 168 hours from irradiation. The three-way ANOVA disclosed that all factors (radiant exposure, postirradiation time, specimen surface) were strongly significant ($p<0.001$). For FU and TEC, interactions among pairs of factors were also significant, whereas no significant interaction among factors was revealed for ESQ. Tukey's post hoc tests revealed that the microhardness of all tested composites increased with the time elapsed from light-curing. In the period between 1 and 24 hours, this increase was significant on both surfaces at all radiant exposures for FU and TEC, and at high radiant exposure for ESQ. Microhardness continued to increase between 24 and 168 hours, but the difference was not significant except for a few TEC and ESQ groups. Significantly higher microhardness was found on top surfaces compared with bottom surfaces in all groups of TEC ($p<0.001$) and in all groups of FU ($p<0.001$) except for the highest exposure of 26.7 J/cm² ($p>0.32$). In contrast, top and bottom surface microhardnesses of ESQ were not significantly different ($p>0.30$).

The effects of polymerization conditions — As microhardness increased during the first 24 hours after irradiation, the effect of polymerization conditions was evaluated after 168 hours when the values of microhardness stabilized. With FU (Table 5), top-surface microhardness at low radiant exposures (15.8 and 16.6 J/cm²) was significantly lower than at moderate radiant exposure (19.4 J/cm²) and at high radiant exposures (25.0 and 26.7 J/cm²). Comparisons of FU microhardness on the top surface at various combinations of irradiance and irradiation time 2980/5 vs 1950/8 at low radiant exposures, 2980/6 vs 1950/10 at moderate radiant exposures, and 1950/12 vs 1300/20 at high radiant exposures did not reveal any significant effect of polymerization conditions ($p=1$). On the bottom surface, microhardness increased significantly with radiant exposure except for 15.8 and 20.7 J/cm². The effect of polymerization conditions was significant at low radiant exposures ($p<0.01$), as microhardness at increased irradiance and shorter irradiation time 2980/5 was higher than at 1950/8.

The microhardness of TEC (Table 6) was lower in comparison with FU. On the top surface, microhardness at the low radiant exposure of 15.8 J/cm² and moderate radiant exposure of 19.4 J/cm² was not significantly different from high radiant exposures ($p>0.17$). At low and moderate radiant exposures, significantly higher microhardness was measured for combinations of increased irradiance and shorter irradiation time, ie, microhardness at 2980/5 and 2980/6 was significantly higher than at 1950/8 and 1950/10, respectively. On the bottom surface, microhardness increased gradually with radiant exposure and increased significantly between low and high radiant exposures ($p<0.01$). The

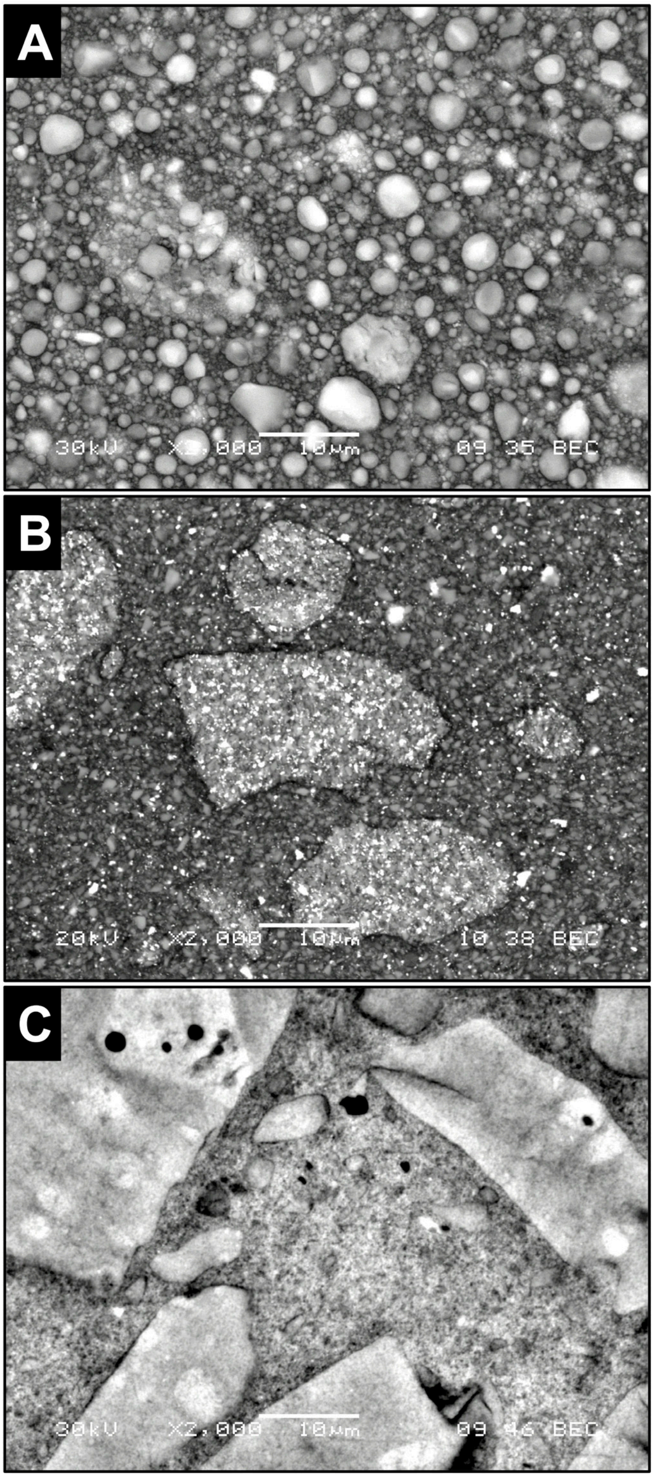

Figure 1. SEM analysis of composite microstructure. Backscatter electron images at 2000x magnification. (A): Filtek Ultimate Universal Restorative: agglomerates of ZrO_2 and SiO_2 nanoparticles. (B): Tetric EvoCeram: prepolymerized composite filler particles. (C): Estelite Σ Quick: heterogeneous prepolymerized composite filler particles. Abbreviations: SEM, scanning electron microscope; SiO_2, silicon di-oxide; ZrO_2, zirconium dioxide.

effect of polymerization conditions within each pair of radiant exposures was not significant (p>0.9).

The microhardness of ESQ (Table 7) increased gradually with radiant exposure on both surfaces, leading to significant differences between low and high radiant exposures (p<0.01). The effect of polymerization conditions was significant (p<0.03) at high radiant exposure on both surfaces, ie, significantly higher microhardness was obtained at mode 1300/20 compared with 1950/12.

Although the influence of radiant exposure on microhardness was material-dependent, none of the materials reached 80% of the maximal top-surface microhardness (62.7 KHN for FU, 40.7 KHN for both TEC and ESQ) or bottom surfaces at low radiant exposures (15.8 and 16.6 J/cm^2), suggesting insufficient polymerization.[31] The microhardness of ESQ at 15.8 J/cm^2 did not surpass the 80% threshold even on the top surface (Table 7).

DISCUSSION

The introduction of high-power polymerization lamps led to a discussion on whether irradiation time could be shortened when irradiance is proportionally increased. This would be applicable if ERL was upheld; however, in the opposite case, short irradiation times calculated using ERL may lead to a lower extent of polymerization, inferior mechanical properties,[24,32-35] an increased release of unreacted components,[36,37] or an increased susceptibility of composites to discoloration.

One of the essential requirements for the verification of ERL is the measuring of irradiance using a calibrated spectrometer or radiometer,[29] as noncalibrated spectrometers or even hand-held radiometers may provide misleading results. The measurement should be performed under the equivalent conditions as specimen polymerization to ensure the same amount of photons incident on the specimen surface as on the detector. Differences between the settings of irradiance measurements and specimen preparation, eg, in the distance of the lamp from the detector/specimen, may result in misinterpretation because irradiance varies with the distance between the tip of the lamp and specimen surface.[18] The detector and the specimen's irradiated surface should also be of a similar diameter, as spatial distribution of the emitted light may be heterogeneous.[38,39]

In this study, the irradiance of composite specimens was measured using a cosine corrector with a 3.9-mm diameter. This corresponded to the central area of the lamp's 9.6-mm tip diameter, where radiant flux is usually the highest.[38,39] In the standard and high power modes, irradiance surpassed the manufacturer's values

Table 5: *Filtek Ultimate Universal Restorative Microhardness Results*

Radiant Exposure (J/cm^2)	Top Surface			Bottom Surface		
	1 h	24 h	168 h	1 h	24 h	168 h
15.8 (2980/5)	61.6 ± 2.4 ABa*	69.3 ± 1.9 Ab*	71.7 ± 2.9 Ab*	52.0 ± 1.1 Ba*	58.7 ± 0.5 Bb*	61.3 ± 1.0 Bb*
16.6 (1950/8)	61.2 ± 0.8 Aa*	70.3 ± 1.2 Ab*	72.4 ± 1.1 Ab*	46.0 ± 0.8 Aa*	54.4 ± 1.3 Ab*	56.1 ± 0.8 Ab*
19.4 (2980/6)	65.9 ± 1.2 Ca*	74.5 ± 1.0 Bb*	76.1 ± 0.5 BCb*	59.1 ± 1.5 CDa*	65.8 ± 2.0 Cb*	67.6 ± 1.6 Cb*
20.7 (1950/10)	65.0 ± 1.3 BCa*	72.5 ± 2.7 ABb*	74.5 ± 1.1 ABb*	56.3 ± 1.2 Ca*	63.8 ± 3.0 Cb*	63.9 ± 1.0 BCb*
25.0 (1950/12)	66.9 ± 1.1 Ca*	75.2 ± 1.2 Bb*	78.0 ± 1.4B Cb*	62.2 ± 2.2 DEa*	69.8 ± 1.8 Db*	72.8 ± 2.1 Db*
26.7 (1300/20)	64.0 ± 0.4 ABCa	75.2 ± 1.0 Bb	78.4 ± 1.4 Cb	63.6 ± 0.8 Ea	73.2 ± 0.7 Db	75.3 ± 1.9 Db

Different superscript letters indicate significant differences, uppercase letters in columns and lowercase letters in rows (among storage times within each surface). Asterisks indicate significant differences between corresponding groups on top and bottom surfaces. Polymerization was considered insufficient if bottom-surface microhardness after 168 hours did not reach 62.7 KHN, corresponding to 80% of the maximal top-surface microhardness (78.4 KHN). Abbreviation: h, hours.

by 30%-40% (Table 2), possibly due to the difference in the light-collecting area between the 3.9-mm diameter in our measurement and the 13-mm diameter of the Demetron LED hand-held radiometer used by the manufacturer.[28] In contrast, the irradiance of 2980 mW/cm^2 measured in the plasma emulation mode was slightly lower than the claimed value of 3200 mW/cm^2 measured using an integrating sphere with a 4-mm aperture.[28] These results demonstrate that calculations of radiant exposure should not rely on the manufacturer's data. Firstly, the information may be misleading, and secondly, manufacturers specify radiant exitance (mW/cm^2) defined as radiant flux emitted by the entire lamp's tip surface per unit area, which is not equivalent to irradiance (mW/cm^2), ie, the radiant flux incident on a composite surface per unit area.[29,40] Radiant exitance is therefore a characteristic of the lamp, whereas irradiance represents the light energy actually received by the specimen, and it varies with the distance from the lamp's tip and several other factors. Irradiance equals radiant

Table 6: *Tetric EvoCeram Microhardness Results*

Radiant Exposure (J/cm^2)	Top Surface			Bottom Surface		
	1 h	24 h	168 h	1 h	24 h	168 h
15.8 (2980/5)	40.1 ± 0.4 BCa*	47.5 ± 0.9 Cb*	48.2 ± 0.8 BCb*	33.1 ± 0.6 Aa*	37.2 ± 1.1 ABb*	38.5 ± 0.4 Ab*
16.6 (1950/8)	35.6 ± 1.1 Aa*	40.9 ± 1.2 Ab*	44.1 ± 0.8 Ac*	31.8 ± 2.1 Aa*	36.4 ± 1.0 Ab*	39.2 ± 0.7 ABc*
19.4 (2980/6)	40.5 ± 1.3 BCa*	49.3 ± 1.0 Cb*	50.9 ± 0.6 Db*	36.0 ± 1.0 BCa*	40.5 ± 0.6 CDb*	42.1 ± 0.2 Cb*
20.7 (1950/10)	38.9 ± 0.9 ABa*	44.6 ± 0.9 Bb*	46.7 ± 0.2 Bb*	33.9 ± 1.2 ABa*	39.0 ± 0.5 BCb*	41.5 ± 0.1 BCc*
25.0 (1950/12)	41.5 ± 0.4 Ca*	48.5 ± 0.5 Cb*	50.3 ± 0.1 CDb*	37.6 ± 0.7 Ca*	41.7 ± 0.8 Db*	43.6 ± 1.6 Cb*
26.7 (1300/20)	41.7 ± 0.5 Ca*	49.4 ± 0.8 Cb*	49.3 ± 1.9 CDb*	36.3 ± 0.8 Ca*	40.7 ± 1.3 CDb*	42.2 ± 1.8 Cb*

Different superscript letters indicate significant differences: uppercase letters in columns and lowercase letters in rows (among storage times within each surface). Asterisks indicate significant differences between corresponding groups on top and bottom surfaces. Polymerization was considered insufficient if bottom-surface microhardness after 168 hours did not reach 40.7 KHN corresponding to 80% of the maximal top-surface microhardness (50.9 KHN). Abbreviation: h, hours.

Table 7: *Estelite Σ Quick Microhardness Results*

Radiant Exposure (J/cm^2)	**Top Surface**			**Bottom Surface**		
	1 h	**24 h**	**168 h**	**1 h**	**24 h**	**168 h**
15.8 (2980/5)	32.7 ± 0.7 Aa	34.6 ± 0.8 Aab	37.9 ± 0.7 Ab	31.8 ± 1.0 Aa	33.7 ± 0.4 Aa	35.9 ± 0.9 Aa
16.6 (1950/8)	35.6 ± 0.5 ABa	40.6 ± 1.1 Bb	41.7 ± 0.5 ABb	35.5 ± 0.6 ABa	37.8 ± 0.5 ABa	38.8 ± 0.5 ABa
19.4 (2980/6)	40.4 ± 1.4 CDa	44.6 ± 1.5 BCab	45.7 ± 0.6 BCb	37.7 ± 0.8 BCa	41.9 ± 0.5 BCab	43.8 ± 0.6 CDb
20.7 (1950/10)	37.5 ± 1.5 BCa	43.2 ± 0.9 BCb	44.6 ± 0.8 BCb	37.2 ± 2.1 Ba	40.6 ± 1. 0 Bb	42.0 ± 1.1 BCb
25.0 (1950/12)	40.6 ± 2.0 CDa	44.6 ± 3.0 Cb	47.0 ± 3.1 Cb	40.4 ± 2.1 CDa	44.0 ± 3.7 CDb	45.8 ± 3.8 Db
26.7 (1300/20)	43.2 ± 1.4 Da	49.9 ± 1.1 Db	50.9 ± 1.0 Db	41.8 ± 0.3 Da	47.0 ± 1.6 Db	50.1 ± 0.6 Ec

Different superscript letters indicate significant differences: uppercase letters in columns and lowercase letters in rows (among storage times within each surface). Polymerization was considered insufficient if bottom-surface microhardness after 168 hours did not reach 40.7 KHN corresponding to 80 % of the maximal top-surface microhardness (50.9 KHN). Abbreviation: h, hours.

exitance only at zero distance, ie, when the detector/specimen are in contact with the lamp's tip.[40]

When using various radiant exposures in the examination of ERL validity, the uncertainty in the measurement of irradiance and irradiation time should also be taken into account. In this study, 3 pairs of radiant exposures obtained using different combinations of irradiance and irradiation time were selected to clarify the effect of polymerization conditions on the validity of ERL (Table 3). They included the conventional irradiation time of 20 seconds at irradiance 1300 mW/cm^2 and shorter irradiation times (down to 5 seconds) at irradiance of up to 2980 mW/cm^2. Variations in spectral irradiance at predefined radiant modes of the Valo lamp equipped with 3 kinds of diodes might be another issue affecting polymerization degree.[41,42] This is important because light emitted in the 420-515 nm region activates CQ, whereas the 380-420 nm wavelength range is effective for TPO. Irradiance measured in these ranges showed that that the proportion of violet light (380-420 nm) increased in the modes with a higher irradiance (Table 2). This increased the number of photons in the violet region, which could yield more radicals from TPO in TEC and contribute to improved polymerization.

When verifying the validity of ERL for dental materials, many studies focused only on the irradiated surfaces or thin films, but it is also essential to evaluate polymerization on the bottom surface of a composite increment where the risk of insufficient polymerization is increased. It is known that transmitted light energy decreases exponentially with increasing composite thickness due to light absorption and scattering.[43] The measurement of irradiance (through a 2-mm thick resin disc) of each composite revealed that the transmittance of ESQ and TEC was approximately 12%-13%, which corroborates previous reports with these materials.[44-46] The transmittance of FU was markedly lower, only around 3%, presumably because a more opaque dentin shade was used. Such an intensive decrease in irradiance was previously observed with some nanohybrid composites.[45] These measurements also revealed that the transmission of violet light (380-420 nm) was lower than that of blue light (420-515 nm), which corresponds to results of several previous studies.[45,47,48] This is due to the Rayleigh scattering of light by particles whose size is much smaller than the wavelength of the incident light, because the amount of scattering is inversely proportional to the fourth power of the light wavelength.

Microhardness is a suitable method for the evaluation of polymerization quality, because it is correlated with DC.[23,31] However, in the case of TEC and ESQ, the measurements were complicated by the presence of large irregular prepolymerized composite filler particles (Figure 1) whose hardness (70-80 KHN for TEC and 80-95 KHN for ESQ) was markedly higher than that of the surrounding composite and apparently independent of the polymerization conditions. These particles interfered with microhardness measurements and could conceal the effect of different exposures on matrix DC, so indentations were performed between them. Nevertheless, due to their high load, especially in ESQ, interference with the indenter could not always be avoided, and it was necessary to perform multiple measurements to obtain symmetrical indentations and microhardness values, which did not vary by more than 10%. In the case of FU, the size of primary inorganic nanoparticles and their agglomerates was much smaller, up to approximately 5 μm in diameter, and they were homogenously distributed within the resin matrix, allowing for plastic deformation at indentation sites and thus more reliable microhardness measurements.

The microhardness of all tested composites increased significantly during the first day, indicating a high rate of postirradiation polymerization.[24,25] After 168 hours, the microhardness values stabilized and they were used to evaluate the influence of radiant exposure and polymerization conditions on the validity of ERL. Among the tested materials, the CQ-A-based nanocomposite FU exhibited the highest microhardness values. On the top surface, low radiant exposures resulted in significantly lower microhardness compared with higher exposures. In each pair of radiant exposures, the effect of different irradiance and irradiation time was not significant, suggesting the validity of ERL. On the bottom surface, microhardness was significantly lower compared with the top surface except for the highest radiant exposure (26.7 J/cm^2). This can be attributed to the aforementioned low transmittance of FU and hence fewer photons available for CQ photoactivation. Bottom-surface microhardness increased with radiant exposure as well. At low radiant exposures, however, significantly higher microhardness was obtained when higher irradiance was combined with shorter irradiation time, that is at 2980/5 compared with 1950/8, which contradicts ERL. A similar but nonsignificant difference was found between combinations 2980/6 and 1950/10 at moderate radiant exposure. Therefore, it can be speculated that higher irradiance resulted in faster photobleaching of CQ,[49,50] thus decreasing the absorbance of light and allowing an increased number of photons to reach deeper layers of the composite material. This result is contradictory to several previous studies that reported either a similar or lower quality of polymerization of CQ-based composites polymerized at higher irradiances and shortened irradiation times.[5,10-12,16,20] However, most of these studies did not examine polymerization quality in deeper layers of the composites, where the effect of photobleaching could manifest. The microhardness of ESQ increased gradually with radiant exposure on both surfaces, significantly between low and high radiant exposures. On the other hand, even at low exposures, there were no significant differences between top and bottom surface microhardness. This could be due to the transmittance of ESQ for blue light, which was the highest of all the tested composites, and the RAP initiating system, which is supposed to regenerate consumed CQ molecules and hence allow for a higher yield of radicals.[26,27] The regeneration of CQ could also prevent the photobleaching effect speculated for FU. As a consequence, combinations of higher irradiance and shorter irradiation time did not lead to higher microhardness. On the contrary, the influence of polymerization conditions was significant at high radiant exposures; microhardness obtained at 1300/20 was significantly higher than at 1950/12, suggesting that ERL was not upheld in this case.

Among the tested composites, TEC was the only one containing not only CQ-A but also TPO, a very efficient initiator with a high quantum yield. TPO does not require the presence of reducing agents (eg, tertiary amines), as it undergoes α-cleavage (Norrish type I reaction) and forms 2 free radicals.[51] Together with the increased proportion of violet light at the highest irradiance, highest irradiance, TPO presumably contributed to efficient polymerization of the top surface in the plasma emulation mode (2980 mW/cm^2). As a result, top-surface microhardness at low radiant exposure 15.8 J/cm^2 (2980/5) and moderate radiant exposure 19.4 J/cm^2 (2980/6) did not statistically differ from microhardness at high radiant exposures, where the proportion of violet light was lower. In the pairs of similar radiant exposures, microhardness values obtained at 2980/5 and 2980/6 were significantly higher compared with 1950/8 and 1950/10, respectively, which is contradictory to ERL. On the bottom surface, TPO could not be effectively activated because the proportion of violet light decreased to only about 2% of the transmitted radiant flux (Table 4). Consequently, microhardness decreased significantly compared with the top surface, which agrees with previous studies reporting a lower depth of cure of TPO-based composites. It was attributed to the increased scattering of violet light[45,47,48] and to the high molar absorptivity of TPO.[1,52] Microhardness on the bottom surface gradually increased with radiant exposure, and groups with low radiant exposure exhibited significantly lower values than groups with high radiant exposure, similar to FU and ESQ. No significant effect of polymerization conditions was observed in the pairs of similar radiant exposures, indicating that ERL was upheld. Overall, 5/18 groups (~28%) did not follow ERL, and based on these results, the first and second null hypotheses were rejected.

A ratio of bottom-to-top microhardness is an acknowledged criterion of polymerization quality of a 2-mm thick increment.[31] If this ratio is greater than 0.8, ie, the microhardness on the bottom surface surpasses 80% of the maximal microhardness measured on the top surface, the polymerization is regarded as sufficient.[31] In this study, the criterion was not met at low radiant exposures of 15.8 J/cm^2 (2980/5) and 16.6 J/cm^2 (1950/8) with all tested composites (Tables 5-7). Moreover, the bottom-surface microhardness was just above the 80% threshold even in groups with moderate radiant exposures, especially 20.7 J/cm^2 (1950/10). These findings are noteworthy because radiant exposures used in this study were much higher than the manufacturers' minimal requirements (9 J/cm^2 for ESQ, 10 J/cm^2 for FU and TEC). In clinical practice where polymerization conditions are not ideal, the risk of insufficient polymerization is even higher. The distance between the lamp's tip and the composite surface is usually larger, and their mutual orientation is neither perpendicular nor concentric, thus decreasing the number of photons incident on the composite surface.

CONCLUSION

Within the limitations of this study, it was concluded that ERL cannot be used as a universal rule to calculate irradiation time for preset light intensities of polymerization lamps because composite polymerization was dependent not only on radiant exposure but also on combinations of irradiance and irradiation time. The composite layer thickness should be taken into account as well, because light is substantially attenuated when passing through the material. Despite high irradiance, insufficient polymerization of the bottom surface of a standard 2-mm increment was observed at short irradiation times, suggesting that it is necessary to examine the polymerization of both surfaces in the assessment of ERL validity.

Acknowledgments

Supported by Charles University, project PROGRES Q29/1LF.

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.

(Accepted 25 September 2020)

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