Influence of Light Energy Density, Composite Type, Composite Thickness, and Postcuring Phase on Degree of Conversion of Bulk-fill Composites

Abstract

Context: Achieving a high degree of conversion (DC) is one of the major concerns during photopolymerization of bulk-fill composites. **Aims:** To evaluate the effect of light energy densities (11.2 J/cm² and 20 J/cm²) on the DC and variation of DC in the 24-h postcuring of four bulk-fill composites: SDR, Venus Bulk Fill, MI FIL, and Tetric N-Ceram Bulk Fill at simulated clinically relevant filling depths. **Settings and Design:** This was an *in vitro* comparative study. **Subjects and Methods:** A total of twenty samples were prepared using a teflon mold. VALO curing light was used with two light intensity modes of 1000 mW/cm² for curing time of 20 s and 1400 mW/cm² for curing time of 8 s. The energy density was calculated as follows: energy density (J/cm²) is the light intensity (mW/cm²) applied during a certain time (s) divided by 1000. The DC was measured at two time intervals: immediately postcure and after 24-h storage in artificial saliva using an Fourier-transform infrared spectroscopy equipped with attenuated total reflectance accessory. **Statistical Analysis Used:** ANOVA and Bonferroni test at $P < 0.05$. **Results:** High energy density (20 J/cm²) leads to higher DC. Thickness, type of composites, and postcuring phase strongly influence the DC. DC values of the top surface for all the bulk-fill materials investigated were found significantly greater ($P < 0.005$) than those of their bottom surface. Among composites, SDR showed highest DC. DC strongly increased after 24-h postcure by 32% on top surface and 76% on bottom surface. **Conclusions:** Energy density more than 20 J/cm², derived by increasing curing time and low power density, helps obtain a high DC of bulk-fill composites for adequate clinical performance.

Keywords: *Bulk-fill composites, curing, degree of conversion, energy density, post, thickness*

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Introduction

Over the past few years, bulk-filled composite has caused a dynamic transition in placement techniques of direct posterior composite restorations. Because of limited depth of cure of conventional resin-based composites, incremental layering technique with a maximum of 2-mm thickness has been accepted as a standard for the placement.[1] However, layering technique is time consuming, and the possibility of incorporating voids between layers and chances of contamination is much higher[2,3] Characteristic feature of bulk-fill composites is its increased depth of cure up to 4 mm. Furthermore, various studies have concluded that bulk-fill composites exhibit reduced polymerization shrinkage stress.[4,5] These properties may be addressed to modification in their organic matrix and/or filler size and content, incorporation of different photoinitiators (Tetric N-Ceram Bulk Fill) and higher translucency (SureFil SDR).[6,7] Other characteristics are reduced cuspal deflection in Class II cavities,[8] good marginal adaptation,[9] and acceptable creep deformation.[2]

Degree of conversion (DC) is an important parameter to know the optimal clinical performance of resin-based composite materials. It has been stated that DC is directly related to physical and mechanical properties such as strength, hardness, solubility, color changes, and biocompatibility of resin-based composites.[10-12] Theoretically, during photopolymerization, all monomers would be converted to polymers, that is, C=C converted to C-C. However, in clinical situation, dimethacrylate monomers exhibit some amount of residual bonds (C=C) in the final product.[13] Factors affecting the DC are chemistry of organic matrix, filler type, amount, size, distribution, photoinitiators used, shade of composite resin, curing time, light intensity, curing mode, light tip size, etc.[1,10,14] There is increasing evidence of a

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gradual decrease in DC of resin composites with increasing distance from the irradiated surface.[15] The reported DC values for conventional composite resins are in the range of 52%–75%.[10] Ilie *et al*. reported 41.4% and 43.8% DC of Tetric EvoCeram and X-tra base Bulk Fill composite resins, respectively, using an Fourier-transform infrared (FTIR) spectrometer.[7] Another study done by Leprine *et al*. reported a range of 43.6%–76.5% DC of nine bulk-fill composites.[6]

Manufacturers have always focused on increasing the curing efficiency of resin-based composite by variation in irradiation intensity, time, and curing mode, keeping the assertion that an adequate polymerization might be reached at short exposure times (5 s or less) at high irradiances. However, another important parameter to consider is total energy delivered by light curing unit, that is, energy density measured in J/cm^2.[11,14] Emami *et al*. stated that a fixed amount of blue light energy rather than high light intensity output is the key factor that should be considered when we try to optimize the clinical properties of light-cured dental composites.[14] Recommended energy density for adequate polymerization of conventional composite resin at 2-mm depth is 21–24 J/cm^2.[11] Various *in vitro* studies have reported that bulk-filled composites can achieve an optimal DC at 4-mm thick layers. However, limited data are available on energy density required for the optimal DC of bulk-fill composites.

Therefore, this study aims to evaluate the effect of light energy densities on the DC and variation of the DC in the 24 h following photoactivation of four bulk-fill resin-based composites: SDR, Venus Bulk Fill, MI FIL, and Tetric N-Ceram Bulk Fill at simulated clinically relevant filling depths. The null hypothesis tested was that energy density, composite thickness, type of bulk-fill composites, and postcuring phase have an impact on the DC.

Subjects and Methods

Four bulk-fill composites were investigated [Table 1] by assessing the DC at 6-mm distance away from the light tip as a function of energy density (11.2 J and 20 J), depth of cure (2 mm and 4 mm), and at two time intervals (immediate postcure and 24-h postcure). Artificial saliva is used as storage media.

The DC was measured using an FTIR spectrometer equipped with attenuated total reflectance (ATR) accessory. The FTIR spectrometer was operated under the following conditions: 4000–500 cm^{-1} wavelength, 6 cm^{-1} resolution, and 32 scans.

Immediate postcure degree of conversion

The uncured composite material was placed on ATR crystal, making sure that the crystal was completely covered by the material (*n* = 5), and then the FTIR spectra of the uncured samples were collected. A total of 20 samples were prepared using a teflon mold, 10 for each thickness (2 mm and 4 mm).

Curing protocol

VALO curing light (Ultradent) was used with two light intensity modes of 1000 mW/cm^2 (Standard mode) for curing time of 20 s and 1400 mW/cm^2 (high power mode) for curing time of 8 s. Output of curing unit was verified with Bluephase Meter II (IvoclarVivadent, Schaan, Liechtenstein). The energy density was calculated as follows: energy density (J/cm^2) is the light intensity (mW/cm^2) applied during a certain time (s) divided by 1000.

Ten samples, 5 for each thickness, were cured at 11.2 J, while remaining 10 were cured at 20 J energy density. Light tip was kept at a distance of 6 mm from the sample. The FTIR spectra of cured samples were then collected immediately.

24-h postcure degree of conversion

Samples were stored in artificial saliva at the 37°C for 24 h. After 24 h, each sample was carefully placed on an ATR crystal plate and FTIR spectrum of the sample was then collected.

Degree of conversion calculation

For all samples, DC was measured using Origin 8 software (OriginLab corporation, Northampton, MA, USA) by assessing the variation in the ratio of the absorbance intensities of aliphatic C=C peak at 1638 cm^{-1} and that of an internal standard peak of aromatic C=C at 1608 cm^{-1} of the uncured and cured samples. For SureFil SDR, the reference peak was set at 1600 cm^{-1} due to the absence of the aromatic carbon bond

Table 1: Compositions of bulk-fill composite resins tested in the study

Materials	Manufacturer	Organic matrix	Filler	Filler loading (weight %)
SureFil SDR	DENTSPLY	Modified UDMA, EBPADMA, TEGDMA	Ba-Al-F-B silicate glass, Sr-A-F silicate glass	68
Venus Bulk Fill	Heraeus	UDMA, EBPADMA	Ba-Al-F-silicate glass,	65
MI FIl	GC Japan	UDMA, Bis-MEPP, TEGDMA	Silica- Nanofiller (16 nm), Sr-doped nanofiller (200 nm)	69
Tetric N-Ceram Bulk Fill	Ivoclar Vivadent	BisGMA, UDMA	Ba glass, Prepolymer, Ytterbium trifluoride and mixed oxide	75-77

DC (%) =100 × (1 − R cured/R uncured)

where R = band height at 1638 cm⁻¹/band height at 1608 cm⁻¹

Statistical analysis

Data were entered into statistical software SPSS version17.0 (SPSS Inc.,Chicago,IL,USA). An analysis of variance (ANOVA) was performed to evaluate DC values among composites, between energy densities, thickness, immediate cure, and 24-h postcure. *Post hoc* multiple comparisons (Bonferroni test) among the composites were performed. *P* <0.05 was considered statistically significant.

Results

The Mean and standard deviation of the DC recorded immediately after cure and at 24-h postcure in top and bottom surfaces are presented in Table 2. The results from ANOVA depicting significant differences among composites, between energy densities and thickness, and

their interactions are presented in Table 3 ($P < 0.001$, $P = 0.002$). Variation in DC between immediate cure and 24-h postcure is presented in Table 4. About 32% on top surface and 76% on bottom surface increase in DC was observed after 24 h of curing.

Immediate postcure mean DC values of different materials were in the following order: SDR = Venus Bulk Fill > MI FIL > Tetric N-Ceram Bulk Fill and 24h postcure: SDR > Venus Bulk Fill > MI FIL > Tetric N-Ceram Bulk Fill. DC values of top surface for all the bulk-fill materials investigated were found significantly greater ($P < 0.005$) than those of their bottom surface. The greatest mean DC was obtained when all the materials of thickness 2 mm and 4 mm were irradiated with 20 J/cm² energy density.

Discussion

Adequate photopolymerization is one of the most crucial aspects for clinical success of composite resin

Table 2: Mean and standard deviation of the degree of conversion recorded immediately after cure and at 24-h postcure in top and bottom surfaces

Composite	Energy density	Thickness	Degree of conversion			
			Mean±SD			
			Immediate cure		24-h postcure	
			Top surface	Bottom surface	Top surface	Bottom surface
SDR	20J	2 mm	60.12±1.52	46.48±2.53	76.45±1.83	67.37±1.55
		4 mm	56.95±2.56	39.7±2.21	74.81±1.62	57.67±2.82
	11.2J	2 mm	52.6±2.33	33.86±2.1	70.27±0.84	59.53±1.94
		4 mm	46.23±3.48	18.77±3.81	63.22±2.58	47.98±2.57
Venus	20J	2 mm	59.61±2.72	44.54±1.66	79.79±2.77	60.58±1.32
		4 mm	52.06±0.91	35.41±3.55	64.89±1.79	56.08±2.03
	11.2J	2 mm	52.55±2.62	33.06±2.98	67.6±2.65	55.03±2.59
		4 mm	44.5±3.2	17.67±2.32	56.59±1.47	44.46±3.11
MI Fill	20J	2 mm	54.14±1.59	31.81±2.2	73.67±1.76	52.16±4.09
		4 mm	44.72±0.86	24.67±1.72	60.78±2.34	39.7±1.6
	11.2J	2 mm	44.02±2.46	18.54±3.43	64.4±1.71	42.75±1.92
		4 mm	34.4±2.04	10.96±2.41	53.56±1.87	31.44±2.29
Tetric N	20J	2 mm	48±1.56	17.8±0.97	62.35±3.55	38.84±1.95
		4 mm	46.3±1.75	24.12±1.08	52.75±1.11	34.13±1.55
	11.2J	2 mm	33.02±2.47	11.6±1.57	44.51±2.15	28.74±1.81
		4 mm	24.96±1.24	10.84±1.61	35.81±2.37	25.06±1.69

SD: Standard deviation

Table 3: Results from analysis of variance depicting significant differences among composites, between energy densities and thickness, and their interactions

	df	Immediate cure				24-h postcure			
		Top surface		Bottom surface		Top surface		Bottom surface	
		F	Significant	*F*	Significant	*F*	Significant	*F*	Significant
Energy density × Thickness	1	20.25	<0.001	9.838	<0.05	116.47	<0.001	31.36	<0.001
Energy density × Composite	3	12.59	<0.001	30.71	<0.001	5.636	<0.05	2.481	0.069*
Thickness × Composite	3	3.666	0.01	8.5	<0.001	14.883	<0.001	9.671	< 0.001
Energy density × Thickness × Composite	3	14.74	<0.001	11.04	<0.001	19.888	<0.001	2.098	0.109*
Error	64	…	…	…	…	…	…	…	…
Total	79	…	…	…	…	…	…	…	…

*Denotes significant difference

restoration.[16] In general, the DC in the commercially available composites has been found between 52% and 75%. However, the minimum DC for clinically acceptable restoration has not yet been exactly recognized.[1] Various authors have reported that, for occlusal restorative layers, DC values below 55% are not recommended.[17,18] Several methods (direct and indirect) have been used to assess the DC, among them FTIR spectroscopy has been used widely as an appropriate and reliable method. FTIR spectrometer detects C=C stretching vibrations directly before and after curing of materials.[19]

Table 4: Increase in degree of conversion between immediate cure and 24-h postcure

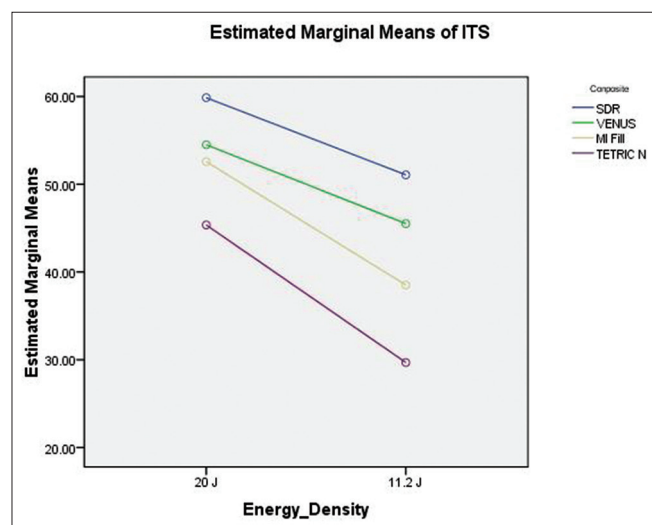|	Immediate curing	After 24 h	DC increase after 24 h (%)
Top surface	47.13±9.72	62.58±11.72	32.70
Bottom surface	26.23±11.7	46.34±12.5	76.60

DC: Degree of conversion

Figure 1: (a and b) Interaction plot shows interaction of energy density and degree of conversion of immediate cure (top surface and bottom surface). (ITS: Immediate top surface, IBS: Immediate bottom surface)

The present *in vitro* study has proven that energy density plays a pivotal role in adequate photopolymerization of bulk-fill composite resins. High energy density (20 J/cm^2) derived from both increased curing time and low power of the curing device results in a faster release of free radicals and, consequently, a higher DC of the material and increased depth of cure [Figure 1].[20] These results concur with those obtained from Gritsch *et al.*, who demonstrated that an increased curing time associated with low power improved the DC and depth of cure due to delay in the formation of rigid grid links between the polymer chains.[21]

The results of this *in vitro* study also indicated that both composite thickness ($P < 0.001$) and type of bulk-fill composite ($P < 0.001$) were significantly correlated with the DC. All bulk-fill composites showed increased DC at 2 mm when compared to those of 4mm thick sample [Figure 2]. The reason for this variation is due to polymerization process initiated by external light. Therefore, variations in the transmission and attenuation of incident light between specimens of different thickness can have a range of outcomes.[22] The tests of all four commercial bulk-fill composites showed that they behave differently with the increase in the energy density used. The difference is mainly because of their chemical composition and filler content. Highest mean DC at top surface and bottom surface was noted with SureFil SDR and Venus Bulk Fill followed by MI FIL, and the least DC was reported in Tetric N-Ceram Bulk Fill. This difference in DC may be attributed to variation in the chemistry of resin matrix as well as the ratio and concentration of monomer used. Initial monomer viscosity and flexibility are the two most important characteristics of a monomer that can affect the DC.[4] Sideridou *et al.* reported that DC of different dimethacrylate monomer systems increase in the order BisGMA <BisEMA (EBPDMA) <UDMA <TEGDMA.[23] Even though additional Ivocerin photointiator is added in Tetric N-Ceram Bulk Fill, it showed least DC. The reason may be BisGMA in resin matrix and filler system as compared with regular nanohybrid Tetric N-Ceram Bulk Fill.[10] BisGMA is highly viscous and least flexible monomer because of strong
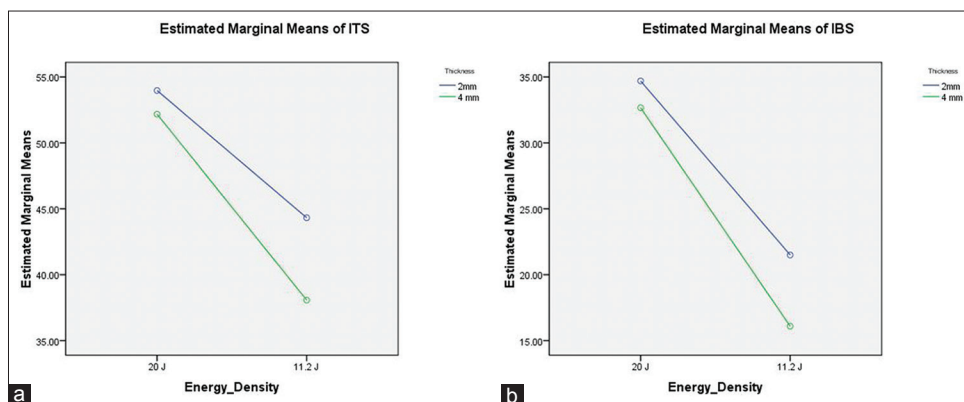

Figure 2: (a and b) Interaction plot shows interaction of energy density and thickness (2 mm and 4 mm) of immediate cure (top surface and bottom surface). (ITS: Immediate top surface, IBS: Immediate bottom surface)

intermolecular hydrogen bonding between pendant hydroxyl groups and rigid aromatic ring based on bisphenol A that negatively affects DC.[4,24] SDR, MI FIL, and Venus Bulk Fill contain mainly UDMA monomer in different ratio with other methacrylate monomers (EBPADMA, TEGDMA, and Bis-MEPP). UDMA is less viscous and has greater flexibility because it contains imino (-NH-) group which forms weaker intermolecular hydrogen bonds compared to hydroxyl group.[25] Furthermore, imino group provides an alternative path for continuation of polymerization by means of chain transfer reactions.[10] Ether (C-O-C) linkages in the TEGDMA molecule lead to decrease in viscosity and more DC.[23] SDR contains a polymerization modulator in the center of the SDR™ monomer backbone which lowers the polymerization shrinkage stress by the formation of more homogeneous copolymer networks. This may be the reason that SDR has shown the highest DC in the present study.[10,26]

Various studies have been reported that the amount of light available to excite the photoinitiator dramatically decreases from the top surface to the bottom surface as a result of light absorption and scattering by the composite itself or surrounding tissue.[14,28,29] Similar results have been reported in the present study. Another finding of the present study was that higher energy density derived from longer curing time leads to increased DC at the bottom surface. This phenomenon can be explained by the effect of light energy incident on the surface of a material, during the attempt to cross it, by a coefficient of attenuation proportional to the physical characteristics of the material itself.[20]

The evaluation of the DC by keeping light curing unit at a 6-mm distance from the top surface of the specimen is clinically relevant because it simulates what happen to DC in difficult-to-access posterior composite restorations. Ilie *et al.* reported that DC influenced by the light guide tip distance at 0 mm and 7 mm, respectively. Increased distance between the curing unit and sample surface leads to decrease in the amount of light energy to activate polymerization.[7] It has also been reported that the DC progression increases during the postcuring phase. Most postcure reaction occurs within a few minutes or 1 h after removal of irradiation source and continues up to 24 h.[10,30,31] The statistical analysis of the present study showed that DC strongly increased after 24-h postcure by 32% on top surface and 76% on bottom surface. Some amount of radicals generated during photopolymerization are trapped within heterogeneous network because as polymerization reaction progresses, both propagation and termination reactions of polymerization process become diffusion limited and also the creation of highly cross-linked polymer network restricts the mobility of the reacting system. These trapped radicals can persist for extended periods, resulting in additional conversion.[14,27] This provides the rationale for further investigation of the mechanical properties of bulk-fill composite resin during the postcuring phase.

Conclusion

The null hypothesis tested that energy density, composite thickness, type of bulk-fill composite, and postcuring phase influence the DC has to be accepted. Moreover, higher energy density increases the DC at the bottom surface. Within the limitation of our study, more than 20 J/cm^2 energy density derived from low power output of curing device and increased curing time can be recommended for adequate clinical performance of bulk-fill composite resins.

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Conflicts of interest

There are no conflicts of interest.

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