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Laboratory Research

Bulk-Fill Composites: Effectiveness of Cure With Poly- and Monowave Curing Lights and Modes

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Clinical Relevance

For camphorquinone-based bulk-fill composites, photopolymerization with monowave light-emitting diode lights may be more efficient than polywave ones. Despite manufacturers' claims, not all bulk-fill composites can be effectively cured to depths of 4 mm.

SUMMARY

This study compared the effectiveness of cure of bulk-fill composites using polywave light-emitting diode (LED; with various curing modes), monowave LED, and conventional halogen curing lights. The bulk-fill composites evaluated were Tetric N-Ceram bulk-fill (TNC), which contained a novel germanium photoinitiator (Ivocerin), and Smart Dentin Replacement (SDR). The composites were placed into black polyvinyl molds with cylindrical recesses of 4-mm height and 3-mm diameter and photopolymerized as follows: Bluephase N Polywave High (NH), 1200 mW/cm^2 (10 seconds); Bluephase N Polywave Low (NL), 650 mW/cm^2 (18.5 seconds); Bluephase N Polywave soft-start (NS), 0-650 mW/cm^2 (5 seconds) → 1200 mW/cm^2 (10 seconds); Bluephase N Monowave (NM), 800 mW/cm^2 (15 seconds); QHL75 (QH), 550 mW/cm^2 (21.8 seconds). Total energy output was fixed at 12,000 mJ/cm^2 for all lights/modes, with the exception of NS. The cured specimens were stored in a light-proof container at 37°C for 24 hours, and hardness (Knoop Hardness Number) of the top and bottom surfaces of the specimens was determined using a Knoop microhardness tester (n=6). Hardness data and bottom-to-top hardness ratios were subjected to statistical analysis using one-way analysis of variance/Scheffe's post hoc test at a significance level of 0.05. Hardness ratios ranged from 38.43% ± 5.19% to 49.25% ± 6.38% for TNC and 50.67% ± 1.54% to 67.62% ± 6.96% for SDR. For both bulk-fill composites, the highest hardness ratios were obtained with NM and lowest hardness ratios with NL. While no significant difference in hardness ratios was observed between curing lights/modes for TNC, the hardness ratio obtained with NM was significantly higher than the hardness ratio obtained for NL for SDR.

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INTRODUCTION

Bulk-fill composites are not a new notion, and many products have come and gone over the past three decades. Potential advantages of bulk-fill composites include reduction of voids in material mass as well as faster and easier placement or curing, leading to improved chairside efficiency. Polymerization shrinkage may, however, be more pronounced, and cure in deep preparations might be inadequate.[1] Contemporary bulk-fill composites differ considerably from their predecessors. Through the use of novel photoinitiators, proprietary resins, special modulators, unique fillers, and filler distribution, current bulk-fill composites are claimed to have lower polymerization shrinkage and depth of cure of 4 mm or more. Studies pertaining to the depth of cure of bulk-fill composites have, however, been equivocal. While some authors have reported adequate cure at 4-mm depths,[2-5] others have reported otherwise.[6-8] The apparent discrepancies can be attributed to differences in testing methodologies, viscosity and translucency of the bulk-fill composites evaluated, and light-curing conditions.[9]

Light-cured composites set via radical photopolymerization. Light photons are absorbed by photoinitiators, and free radicals are formed in the presence of activators. The free radicals subsequently trigger the polymerization reaction, resulting in the conversion of monomers into polymers.[10] Camphorquinone (CQ) is the most widely used photoinitiator and has a sensitivity peak near 470 nm in the blue range of the visible light spectrum. Because of its intense yellow color, alternative lighter-colored initiators that completely bleach out after photopolymerization have been recently promoted. These include phenyl propanedione (PPD), acyl phosphine oxide (APO), and Ivocerin. While the absorption spectrum of PPD extends from the ultraviolet (UV) wavelength range to approximately 490 nm, APO such as Lucirin TPO mainly absorbs light in the UV range. The sensitivity peak of APO is approximately 370 nm, which is considerably lower than that of CQ. Ivocerin is a newly developed germanium photoinitiator that absorbs light at a higher wavelength range than APO and has a sensitivity peak of about 420 nm.

Until the 1990s, halogen or quartz tungsten halogen curing lights were the standard curing option. Because of their wide spectral output, halogen lights required band-pass filters to limit wavelengths of light between 370 nm and 550 nm for the absorption of CQ. Their wide range of wavelengths also allows for the curing of composites employing PPD and APO as photoinitiators. However, the curing efficacy of halogen lights is low, and the high temperatures generated require external cooling and limit the lifetime of bulbs, reflectors, and filters.[11] As such, the use of light-emitting diode (LED) curing lights, which emit blue light, began in the 2000s to address the problems associated with halogen curing lights. Instead of hot filaments, LED curing lights use a combination of two different doped semiconductors for light production. They consume less energy, do not require cooling fans, and have extended lifetimes without significant loss of light intensity.[11] The first-generation LED curing lights had low light intensities of approximately 400 mW/cm^2, while the second generation lights were able to achieve intensities of up to 1000 mW/cm^2. Both first- and second-generation LED lights used only one type of LED (monowave [single-peak] technology) and were unable to cure composites with PPD and APO initiator systems. Current third-generation LED curing lights feature higher light intensities, multiple curing modes (high, low, and soft start), and avoid wavelength-compatibility issues by deploying polywave (dual/multipeak) technology.

Research pertaining to the use of polywave LED curing lights on the effectiveness of cure of bulk-fill composites is still limited.[3,12,13] The curing efficacy in these studies was assessed using different methodologies, including micro-Raman spectroscopy, microhardness testing, and the ISO 4049 scraping test. Comparison of polywave and monowave LED curing lights was addressed in only one study.[12] Using the ISO scraping test, no significant difference in depth of cure was found between the two LED technologies for the bulk-fill composites evaluated (Tetric Evoceram and Filtek Bulk Fill). More studies are therefore required to address the current gaps in knowledge, especially with the increased use of PPD and APO as photoinitiators, particularly in bleached shade composites. Therefore, the objective of this study was to compare the effectiveness of cure of two bulk-fill composites with polywave LED, monowave LED, and conventional halogen curing lights using microhardness testing. Curing efficacy of the high-intensity, low-intensity, and soft-start modes of the polywave LED was also appraised. It was hypothesized that no difference in the effectiveness of cure existed between the polywave LED (and its various curing modes), monowave LED, and halogen curing lights if the total light energy was kept constant.

Table 1: *Technical Profiles of Bulk-Fill Composites Evaluated*

Material	Abbreviation	Shade/Batch Number	Composition	Filler % by Weight (Volume) and Filler Size	Recommended Thickness, mm	Recommended Curing Time and Light Intensity
Tetric N-Ceram Bulk Fill	TNC	Universal (IVA)/LOT S21119, Exp 2017-06	Resin: dimethacrylates Filler: barium glass, ytterbium trifluoride, mixed oxide and copolymers	80%-81% (55%-57%) 0.04-3 μm	4	20 s for ≥ 500 mW/cm^2 or 10 s for ≥ 1000 mW/cm^2
SDR Posterior Bulk Fill Flowable Base	SDR	Universal/ 1405000811, Exp 2016-04	Resin: modified UDMA, EBPADMA, TEGDMA Filler: barium-alumino-fluoro-borosilicate glass, strontium alumino-fluoro-silicate glass	68% (45%) Mean 4.2 μm	4	20 s, for ≥ 550 mW/cm^2

Abbreviations: Bis-MPEPP, 2,2-bis[(4-methacryloxy polyethoxy)phenyl)propane; EBPADMA, ethoxylated bisphenol A dimethacrylate; SDR, Smart Dentin Replacement; S-PRG, surface pre-reacted glass ionomer; TEGDMA, triethylene glycol dimethacrylate; TNC, Tetric N-Ceram bulk-fill; UDMA, urethane dimethacrylate.

METHODS AND MATERIALS

The technical profiles and composition of the bulk-fill composites evaluated are shown in Table 1. Tetric N-Ceram Bulk Fill (TNC; Ivoclar Vivadent, Schaan, Liechtenstein) uses CQ and Ivocerin as photoinitiators, giving it a wide absorption range of 370 nm to 460 nm.[14] According to the manufacturer's instructions, TNC can be cured reliably in 4-mm increments by LED lights of 1000 mW/cm^2 over 10 seconds. While TNC is a "sculptable" bulk-fill restorative, Smart Dentin Replacement (SDR; Dentsply-Caulk, Milford, DE, USA) is a "flowable" bulk-fill base material. The photoinitiator in SDR is CQ, and 4-mm increments of SDR can be cured by halogen lights of 550 mW/cm^2 over 20 seconds.

The composites were placed in a single increment into black polyvinyl molds with cylindrical recesses of 4-mm height and 3-mm diameter. Excess material was removed by compressing the molds between two glass slides (1-mm thick). The composites were then irradiated through the top glass slide using either a polywave LED with different curing modes (Bluephase N Polywave, Ivoclar Vivadent, Schaan, Liechtenstein), a monowave LED (Bluephase N Monowave, Ivoclar Vivadent), or a halogen (QHL75, Dentsply-Caulk) curing light. The polywave LED light offered several different curing modes including high-power, low-power, and soft-start polymerization, whereas the monowave LED light presented with only high-power curing. The exit windows of the light-curing tips were 8 mm in diameter for the three curing lights, and light intensity was verified with a radiometer (Demetron LED radiometer; Kerr Corporation, Middleton, WI, USA) prior to use to ensure consistency of energy output. The five curing light/mode combinations are detailed in Table 2. These were Bluephase N Polywave high power (NH), Bluephase N Polywave low power (NL), Bluephase N polywave soft start (NS), Bluephase N Monowave (NM), and QHL75 (QH). The total energy output (intensity × time) was standardized at 12,000 mJ/cm^2 for all curing lights/modes, with the exception of NS. Because of the preset soft-start curing profile, the closest total energy achievable for NS was 13,625 mJ/cm^2. Six specimens were fabricated for each composite for the various curing light/mode combinations. Immediately after light polymerization, the composite specimens were removed from their molds and stored in a light-proof container at 37°C in a humidified atmosphere for 24 hours. They were then subjected to microhardness testing with a Knoop hardness tester (FM-7, Future-Tech, Tokyo, Japan). A 10 g load was applied with a dwell time of 15 seconds to obtain the Knoop hardness number

Table 2: *Technical Profile of Curing Lights and Modes Evaluated*

Curing Light	Curing Mode	Recommended Curing Profile	Study Curing Profile
Bluephase N Polywave	High (NH)	1200 mW/cm^2 (10 s)	1200 mW/cm^2 (10 s)
Bluephase N Polywave	Low (NL)	650 mW/cm^2 (10 s)	650 mW/cm^2 (18.5 s)
Bluephase N Polywave	Soft start (NS)	0-650 mW/cm^2 (5 s) → 1200 (10 s)	0-650 mW/cm^2 (5 s) → 1200 (10 s)
Bluephase N Monowave	High (NM)	800 mW/cm^2 (15 s)	800 mW/cm^2 (15 s)
QHL-75	Normal (QH)	550 mW/cm^2 (20 s)	550 mW/ cm^2 (21.8 s)

Table 3: *Mean Top KHN, Bottom KHN, and Hardness Ratio (%) With the Different Lights/Modes for TNC and SDR*

Material	Curing Light	Curing Mode	Top KHN	Bottom KHN	Hardness Ratio, %
TNC	Bluephase N Polywave	NH	25.78 (3.56)	10.32 (1.04)	40.95 (8.44)
	Bluephase N Polywave	NL	30.10 (4.13)	11.42 (1.03)	38.43 (5.19)
	Bluephase N Polywave	NS	29.09 (4.74)	11.67 (1.01)	41.10 (8.14)
	Bluephase N Monowave	NM	22.82 (1.91)	11.24 (1.75)	49.25 (6.38)
	QHL-75	QH	30.31 (1.82)	13.43 (1.52)	44.27 (4.08)
SDR	Bluephase N Polywave	NH	15.21 (2.42)	8.74 (0.83)	58.37 (8.67)
	Bluephase N Polywave	NL	16.39 (1.37)	8.30 (0.69)	50.67 (1.54)
	Bluephase N Polywave	NS	17.16 (1.65)	9.98 (0.72)	58.44 (5.13)
	Bluephase N Monowave	NM	16.19 (1.49)	10.95 (1.43)	67.62 (6.96)
	QHL-75	QH	18.35 (1.41)	10.51 (0.82)	57.64 (6.97)

Abbreviations: KHN, Knoop hardness number; SDR, Smart Dentin Replacement; TNC, Tetric N-Ceram Bulk Fill.

(KHN) for both top and bottom surfaces of each specimen. The KHN corresponding to each indentation was determined by measuring the dimensions of the indentations using the following formula:

$$KHN = 14.2 \times (F/d2)$$

where F is the test load in kilograms and d is the longer diagonal length of an indentation in millimeters. For each surface, three readings were taken, and the mean KHN value was calculated. The KHN of the bottom was divided by the KHN of the top surface to establish the hardness ratios, which were subsequently converted to a percentage. KHN data and hardness ratios were subjected to statistical analysis using one-way Analysis of Variance and Scheffe's post hoc test at a significance level of 0.05.

RESULTS

The mean KHN and hardness ratios (%) of the various curing light/mode combinations for TNC are shown in Table 3. The mean KHN of the top surface ranged from 22.82 ± 1.91 to 30.31 ± 4.13 when irradiated using NM and QH, respectively. The mean KHN of the bottom surface ranged from 10.32 ± 1.04 to 13.43 ± 1.52 for NH and QH, respectively. The hardness ratio for TNC ranged from 38.43% ± 5.19% to 49.25% ± 6.38% for NL and NM, respectively. Results of statistical analysis for TNC are reflected in Table 4. At the top surface, the KHNs of QH and NL were significantly higher than NM, while for the bottom surface, the KHN of QH was significantly higher than that of NH. However, no significant difference in hardness ratio was observed.

The mean KHN and hardness ratios (%) of the various curing light/mode combinations for SDR are also shown in Table 3. The mean top KHN ranged from 15.21 ± 2.42 to 18.35 ± 1.41 for NH and QH, while the mean bottom KHN ranged from 8.30 ± 0.69 to 10.95 ± 1.43 for NL and NM, respectively. The hardness ratio for SDR ranged from 50.67% ± 1.54% to 67.62% ± 6.96% for NL and NM, respectively. Results of the statistical analysis for SDR are reflected in Table 4. At the top surface, the KHN of QH was significantly higher than that of NH. At the bottom surface, the KHNs for NM and QH were significantly higher than that of NL. The bottom KHN of NM was also significantly higher than that of NH. Unlike TNC, significant differences in hardness ratio for SDR were observed. The hardness ratio obtained with NM was significantly higher than with NL.

DISCUSSION

The effectiveness of cure of bulk-fill composites with polywave LED, monowave LED, and conventional

Table 4: *Statistical Comparison of Top KHN, Bottom KHN, and Hardness Ratio (%) for TNC and SDR*

Material	Variable	Result[a]
TNC	Top KHN	QH, NL > NM
	Bottom KHN	QH > NH
	Hardness ratio, %	NS
SDR	Top KHN	QH > NH
	Bottom KHN	NM > NH, NL
		QH > NL
	Hardness ratio, %	NM > NL

Abbreviations: KHN, Knoop hardness number; NS, no statistical significance; SDR, Smart Dentin Replacement; TNC, Tetric N-Ceram Bulk Fill.
[a] > denotes statistically significant differences. Results of one-way Analysis of Variance/Scheffe's post hoc test (p<0.05).

halogen curing lights was evaluated. The null hypothesis was rejected as significant differences in effectiveness of cure existed between the different lights and curing modes despite regulating the total light energy. Curing efficacy can be assessed by direct and indirect methods. Direct methods, such as infrared and Raman spectroscopy, are not routinely used as they are complex, expensive, and time-consuming to perform.[15] Indirect methods include visual appraisal, scraping (ISO 4049), and hardness testing. While visual appraisal does not offer scientific objectivity, the ISO scraping test generally results in greater depths of cure when compared with hardness testing.[6] Hardness is an indicator of the degree of polymerization, and a good correlation between Knoop hardness and infrared spectroscopy has been reported.[16,17] In view of its relative efficiency and popularity, Knoop hardness testing was selected to determine the effectiveness of composite cure. Hardness testing was done 24 hours after photopolymerization to allow for composite postcure.[18]

Adequate photopolymerization is essential for optimization of physicomechanical properties and clinical longevity of composite restorations.[19] In addition, inadequately cured composites are also cytotoxic because of residual monomers and other reactive components.[20,21] Composite restorations should ideally be equally cured throughout. The bottom-to-top hardness ratio of the 4-mm-thick bulk-fill specimens should approximate or equal 1 (100%). Many studies have, however, used a hardness ratio of 0.8 or 80% as the standard for satisfactory cure due to material and light-curing constraints.[22,23] Material factors affecting photopolymerization involve thickness, shade, opacity, and composition, while those related to curing lights include light intensity, wavelength, exposure time, size, location, and orientation of the light probes.[19] As thickness, materials, total light energy (intensity × time), and light probe variables including curing distance were controlled during the experiment, the results can be largely attributed to light type, spectral wavelengths, and light intensity modification during curing (ie, continuous versus soft-start curing).

At both top and bottom surfaces, the KHN of TNC was higher than SDR regardless of curing light/mode. Results corroborated those of similar studies concerning these materials and can be attributed to TNC's higher filler content when compared with SDR.[6,7] While TNC can be placed up to the surface of the overall composite resin restoration and functionally loaded, SDR requires a "capping layer" of conventional composite to sustain functional loads because of its lower filler loading. At the top surface, significant differences in KHN were observed between curing lights/modes. For TNC, curing with the halogen light and polywave LED (NL in particular) resulted in harder top surfaces than with the monowave LED light (NM). This finding may be attributed to the narrower spectral output of the NM. For SDR, significant differences in the top KHN was observed between the halogen (QH) and polywave LED in high-power mode (NH). The harder surface associated with QH could be contributed in part to a thermal effect and longer exposure time as emission spectrum and total energy output were similar.[24] Exposure time for QH was approximately double that of NH (21.8 vs 10 seconds), and heating of composites from halogen lights has been shown to increase hardness.[25]

As light passes through the bulk-fill composites, intensity is clearly reduced because of light absorption and scattering by the materials, attenuating the potential for photopolymerization.[26] At the bottom surface, photopolymerization of TNC with the halogen light (QH) resulted in significantly higher bottom KHN than with the polywave LED at high power (NH). Thermal effects are negligible at the bottom surface of restorations as composites are poor conductors of heat.[27] As emission spectrum and total energy output were comparable, differences may well be due to variations in light attenuation, light exposure time, and the ensuing polymeric network type. Light attenuation with NH may be greater, especially considering its short curing time. Previous studies have reported the need for longer curing times with LED lights when compared with halogen lights for achieving a similar depth of cure and mechanical properties.[28,29] Hardness of composites is dependent not only on the degree of conversion but also on the nature of and bonding between monomers.[16] Polymers with more cross-linked chains are harder than those with linear chains.[30] Theoretically, the use of high light intensity in the initial phase of curing should result in a greater number of growth centers and higher cross-link density.[31] The aforementioned may, however, be mitigated by the short curing time associated with NH. As such, the cross-link density of bulk-fill composites associated with the various lights/modes requires further investigation.

For SDR, the bottom KHN with the halogen light (QH) was significantly higher than the polywave LED in low-power mode (NL). Significant differences in bottom hardness were also observed between the

LED lights. Photopolymerization with the monowave light (NM) resulted in significantly higher bottom KHN than curing with the polywave LED in both high- and low-power modes (NH and NL). The incongruence in findings when compared with TNC may be attributed to variances in composite composition. In addition to photoinitiator and resin differences, SDR has a lower filler loading and larger filler particle sizes than TNC, resulting in a more translucent material. Monomer conversion has been found to be inversely proportional to filler loading owing to decreased light transmission.[32] Light scattering from smaller filler particles has been found to reduce depth of cure, especially when filler sizes are similar to the wavelength of the emitted light.[33] Despite comparable curing times, photopolymerization with the halogen light still resulted in harder bottom surfaces than with the polywave LED light, reinforcing the necessity for cross-link density studies. Photopolymerization with the monowave LED light resulted in significantly higher bottom surface KHN than the polywave light, with the exception of the soft-start mode. The better performance of the polywave soft-start mode could be attributed to its slightly higher total energy (13,625 mJ/cm^2). The total energy of the soft-start mode could not be harmonized to 12,000 mJ/cm^2 because of the manufacturer's programmed settings. The polywave LED curing offered no advantage over its monowave counterpart as SDR uses primarily CQ as its photoinitiator.

For both TNC and SDR, the hardness ratio was lower than 80% for all curing lights/modes. This can be attributed to attenuated irradiance reaching the bottom surfaces.[26] The highest hardness ratio achieved was 49.25% ± 6.38% and 67.62% ± 6.96% for TNC and SDR, respectively. Results corroborated a recent independent study by Yap and others involving the same materials.[6] To achieve a hardness ratio of 80%, TNC and SDR need to be limited to increments of 2.5 mm and 3 mm correspondingly.[6] Other authors have, however, reported a bottom-to-top hardness ratio of 80% or more.[34,35] The divergence in outcomes can be attributed to differences in bulk-fill materials evaluated, curing light, or parameters and methodologies employed. For both bulk-fill composites, the highest hardness ratio was obtained with NM and lowest with NL despite their identical total energy output. Ranking of hardness ratios differed slightly between TNC and SDR and were as follows: TNC – NM > QH > NS > NH > NL; SDR – NM > NS > NH > QH > NL. When only the LED curing lights were considered, the ranking of hardness ratio was similar for both bulk-fill composites. Photopolymerization with monowave LED gave the highest hardness ratio followed by the polywave LED light in soft-start, high-power, and low-power modes. The monowave LED light thus appears to be somewhat more effective than the polywave LED at 4-mm depths.

For TNC, no significant difference in hardness ratio was observed between the various curing lights/modes. Results supported those of Meenes and others[12] using the ISO 4049 scraping test and customized tooth molds. They found the influence of curing lights to be insignificant, but there was a relatively significant interaction between materials and mold types on composite depth of cure. The use of stainless steel molds led to a deeper depth of cure for Tetric Evoceram bulk-fill, rationalizing the use of unreflective black polyvinyl molds in the present study. For SDR, photopolymerization with the monowave LED resulted in a significantly higher hardness ratio than with the polywave LED at low-power mode despite standardization of the total energy (67.62% vs 50.67%). The irradiance of the polywave LED light even with a lower power mode was already 650 mW/cm^2, well above the traditionally recommended minimum light intensity of 400 mW/cm^2.[36] The 17% variance in hardness ratio between the two LED lights is of concern and warrants further investigation. It may be attributed in part to the lower percentage of light transmitted through composites offered by polywave LED lights when compared with monowave LED lights.[37] Clinically, the reduction in hardness ratio may be even higher as the curing light probe may be 8 mm or more from the composite surface. The latter has been shown to significantly reduce the degree of conversion at the bottom surface of restorations.[38] The effect of light probe distance on curing efficacy of poly- and monowave LED curing lights should also be explored.

CONCLUSION

Within the limitations of this *in vitro* study, the following conclusions can be made:

1. Top and bottom surface hardness achieved with the halogen curing light was usually higher than that obtained with the LED lights when total energy was controlled.
2. The ideal bottom-to-top hardness ratio of 0.8 (80%) was not achieved by either bulk-fill composite evaluated regardless of curing lights/modes.

3. For both bulk-fill composites, the highest hardness ratios were obtained with the monowave LED curing light and lowest with the polywave LED curing light in low-power mode.
4. Photopolymerization with monowave LED lights may be more effective than with polywave LED lights for camphorquinone-based bulk-fill composites.

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Conflict of Interest

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

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