

Efficacy of Modern Light Curing Units in Polymerizing Peripheral Zones in Simulated Large Bulk-fill Resin-composite Fillings

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Clinical Relevance

Bulk-fill restorations with resin composites are increasing in popularity, but sufficiency of curing in depth and in peripheral zones of large fillings is still questioned. Modern curing units, displaying more homogeneous light beam profiles, claim enhanced homogeneity of curing, also in large fillings.

SUMMARY

The variation in micro-hardness (HV) within simulated large cavities (10×6 mm) filled in one increment with three bulk-fill resin-based composites (BF-RBC) was assessed by means of a universal hardness device. Modern blue and violet-blue light curing units (LCUs) were applied in three different positions, by rotating the LCU in 120° steps. The exposure distance was 3 mm. One center and two peripheral (4-mm apart from the center) HV line profiles were measured in 0.5-mm steps at 24 hours postpolymerization to calculate the depth of cure (DOC). Incident light, irradiance, and spectral distribution were recorded. A

multivariate analysis (general linear model) assessed the effect of the varied parameters as well as their interaction terms on HV and DOC. The effect of LCU rotation was not significant ($p=0.109$). The DOC varied between 3.46 mm and 5.50 mm and was more strongly influenced by the BF-RBC ($p<0.001$, $\eta_p^2=0.774$), followed by the width of specimen ($p<0.001$, $\eta_p^2=0.554$), while the influence of the LCU was very low ($p<0.06$, $\eta_p^2=0.070$). Whether a BF-RBC filling is cured as well in the periphery as in the center depends more on the material than on the curing unit used.

INTRODUCTION

Bulk-fill resin-based composites (BF-RBC) have gained increased interest and acceptance as restorative materials in the daily clinical routine. It is recognized that a bulk-filling technique allows for both reduced chair time and a decrease in the risk of inducing defects or contaminants between layers when compared with an incremental placement technique.¹ Whether a bulk-filling technique reduces

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DOI: 10.2341/17-095-L

shrinkage stress development in an RBC restoration is still controversial.²⁻⁴

The large variation in mechanical properties identified in BF-RBCs⁵ determined their classification in low-filled (flowable) and high-filled (sculptable) BF-RBCs. Restorations with flowable BF-RBCs require an occlusal covering layer of a high-viscosity, sculptable RBC. Despite concerns raised by the very low modulus of elasticity of flowable BF-RBCs,⁵ the described restoration model is successful, as evidenced in a five-year clinical follow-up study.⁶ In contrast, sculptable BF-RBCs may be used in bulk-fill restorations without capping and are characterized by good mechanical properties that are comparable or even higher than values measured in conventional (cured in 2-mm increments), micro-hybrid RBCs.⁵ Long-term clinical studies assessing their longevity are missing so far.

Opponents of using BF-RBCs impugn the sufficiency of curing in deep layers⁷ or in large fillings. *In vitro* studies identified that BF-RBCs are sufficiently cured in a 4-mm increment by using light curing units (LCU) with moderate irradiances (~ 1000 mW/cm²) and exposure times of ~ 20 seconds that correspond to a radiant exposure of ~ 20 J/cm².^{8,9} Interestingly, these curing conditions are comparable to radiant exposure values identified in conventional RBCs for sufficient polymerization (21-24 J/cm²).^{10,11} Variances in radiant exposure are, nonetheless, concerning because there can be grave clinical implications if too little, or conversely too much, radiant exposure is delivered to a BF-RBC restoration. Insufficient polymerization was identified to affect negatively several properties in conventional RBCs, including wear,¹² quality of restoration margins,¹³ bond strength to tooth structure,¹⁴ depth of cure,¹⁴ mechanical properties,^{15,16} degree of conversion, or amount of eluted substances from polymerized specimens.¹⁷ In contrast, increasing the radiant exposure is clinically not a guarantor for adequate curing, owing to the increased risks of thermal damage to the soft tissues and pulp.^{18,19}

The impact of LCUs on BF-RBC properties is assessed *in vitro* preponderantly by using narrow specimen sizes or considering specimen regions that correspond to ideal curing conditions, such as regions close and perpendicular to the center area of the light guide. From a clinical point of view, a sufficient polymerization in peripheral zones of large fillings is of great relevance. There is evidence that several contemporary LCUs have different light output characteristics, and both blue and polywave light-emitting diode (LED) LCUs may provide a

highly inhomogeneous spectral emission and radiant exitance beam profile.²⁰ This aspect is directly reflected in an uneven resin polymerization, manifested in poor physical properties in RBCs.²¹ Particularly when using polywave LED LCUs, the impact of the location of different LED types on the polymerization quality and homogeneity is questioned. This potential negative effect might even be intensified in deeper and peripheral regions of a BF-RBC restoration, considering that the light reaching deeper RBC layers is dependent on the thickness of the layer as well as on the wavelength of light.²² It has been shown that merely 24%-44% of the incident blue light and 9%-14% of the incident violet light is transmitted through 2-mm BF-RBC increments. These values are yet further reduced in 4-mm increments (9%-24% and 3%-9%, respectively).²²

Therefore, the following null hypotheses were tested: 1) the LCU type, 2) the placement of the LCU (by rotation in 120°), 3) the location of measurement (center – peripheral or surface – depth), and 4) the BF-RBC have no effect on hardness (HV) and depth of cure (DOC).

METHODS AND MATERIALS

The effect of two blue and one violet-blue LED LCUs (Table 1) on the variation of hardness within simulated large (10-mm) cavities restored with three bulk-fill RBCs (Table 2) was assessed by means of a universal hardness device. The BF-RBCs were polymerized as recommended by the manufacturer (Table 2). Incident light, irradiance, and spectral distribution were recorded for each LCU.

Characterization of the LCUs

The irradiance and spectral distribution of the used LCUs were measured on a laboratory-grade NIST-referenced USB4000 Spectrometer (Managing Accurate Resin Curing [MARC] System, Bluelight Analytics Inc, Halifax, Canada). The distance between sensor and light guide tip was set at 3 mm to reproduce the exposure distance used for curing the analyzed BF-RBC specimens. The recorded irradiance corresponds thus to the irradiance received by the RBC specimen. The miniature fiber-optic spectrometer uses a 3648-element Toshiba linear CCD array detector and high-speed electronics. The spectrometer has been spectroradiometrically calibrated with Ocean Optics' NIST-traceable light source (300-1050 nm). The system uses a CC3-UV Cosine Corrector to collect radiation over a 180° field of view, thus mitigating the effects of optical interference associated with light collection sam-

Table 1: Characteristics of the Analyzed Light Curing Units			
LCU	Bluephase Style (With Updated Light Guide Tip)	Elipar Elipar DeepCure	Demi Ultra
Manufacturer	Ivoclar Vivadent	3M ESPE	Kerr
Series No.	1110002669	939133000012	787016098
Wavelength range, nm	385-515m	430-480	450-470

pling geometry. The detector of the spectrometer had a diameter of 4 mm. Consequently, irradiance reaching this area was considered. Irradiance at a wavelength range of 360 to 540 nm was individually collected at a rate of 16 records/s on five occasions. The sensor was triggered at 20 mW.

Hardness Profiles

To evaluate the variation in micro-hardness as a function of LCU and material, 6-mm-high disc-shaped specimens with a diameter of 10 mm were prepared by applying the BF-RBCs in one increment in metal molds (*n*=6 for each material and LCU). The material was light cured in one step according to the manufacturers' recommendation, as indicated in Table 2. The curing unit was placed my means of a mechanical arm at 3-mm distance²³ from the specimen's surface, simulating a clinically relevant exposure condition, and it was applied in three different positions, by rotating the LCU in 120° steps. The position of the blue and violet LEDs in the violet-blue LCU Bluephase Style is indicated in Figure 1 for each rotation in relation to the specimen's surface. The analyzed blue LED LCUs contained similar LEDs that were distributed symmetrically. However, the blue LCUs were also rotated in a similar way as the violet-blue LCU to allow for equivalent measurement conditions.

Specimens were stored after curing in 37 °C distilled water for 24 hours, sectioned prior to testing in the middle along the *z*-axis as indicated in Figure 1 with a slow-speed diamond saw (Isomet low-speed saw, Buehler, Germany) under water cooling, then ground with SiC paper until grit 4000 and polished

with a diamond suspension (mean grain size: 1 μm). Measurements were made with an automatic micro-hardness indenter (Fischerscope H100C, Fischer, Sindelfingen, Germany) along the *z*-axis in 500-μm steps, starting at 0.5 mm under the surface that has been exposed to the curing light. Hardness profiles were measured through the middle as well as at two peripheral positions, located 4 mm apart from the middle of the specimens. These profiles are indicated as 1 mm (peripheral left), 5 mm (center), and 9 mm (peripheral right).

The test procedure was carried out force controlled, while the test load increased for 20 seconds and decreased for 20 seconds with constant speed between 0.4 mN and 500 mN. Load and penetration depth of the indenter were continuously measured during the load-unload hysteresis. Universal hardness is defined as the test force divided by the apparent area of indentation under applied test force. From a multiplicity of measurements stored in a database supplied by the manufacturer, a conversion factor between Universal hardness and Vickers hardness (HV) was calculated and input into the software.

The DOC, usually acknowledged as the thickness of an RBC that is adequately cured or rather as the depth where HV equals the surface value multiplied by an arbitrary ratio, usually 0.8²⁴ (= HV-80%), was also calculated. Therefore, for each sample, HV in the depth was compared within each material to the 0.5-mm subsurface value (mean value of all specimens) and noted when it became less than 80%. This depth is defined as DOC.

Table 2: Characteristics of the Analyzed Bulk-Fill Resin-Based Composites			
RBCs	Filtek Bulk-Fill, FBF	Tetric Evo Ceram Bulk Fill, TEVOBF	SonicFill, SF
Manufacturer	3M ESPE	Ivoclar Vivadent	Kerr
Lot No.	N637888	T14294	5338297
Exposure time, s	20	10	20
Shade	A3	IVA	A3
Filler wt%	76.5	76-77	83.5
Filler Vol.%	58.4	53-54	n/a
Light-initiator system	Camphorquinone/amine	Camphorquinone/amine, germanium-based, acyl phosphine oxide	Camphorquinone/amine

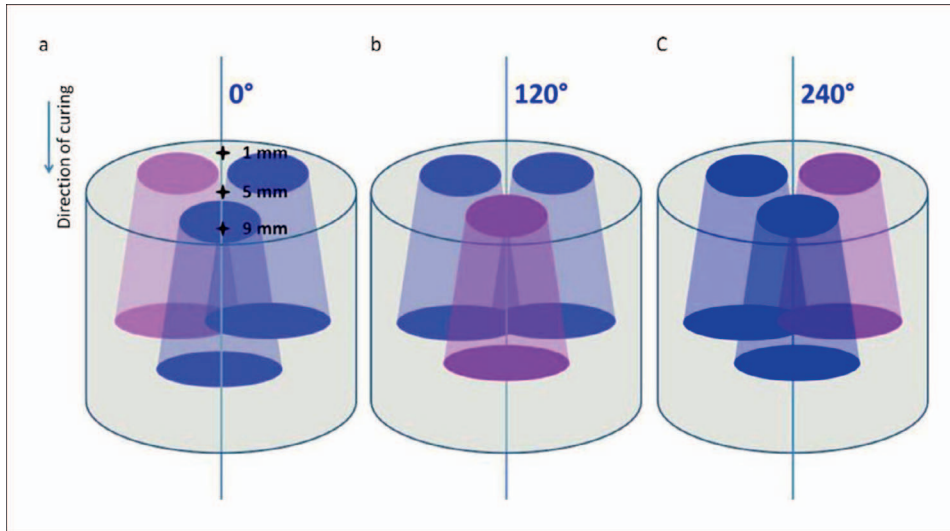


Figure 1. Projection of the light-emitting diodes (Bluephase Style) on specimens' surface (diameter = 10 mm, depth = 6 mm) during curing. The vertical line indicates the section plane; degree (0° , 120° , and 240°) indicates the rotation of the curing unit. The right half of the specimen was used for micro-hardness measurements. Three hardness line profiles were evaluated in depth in the section plane through the middle (5 mm) as well as at 4-mm left (1 mm from the specimen's margin = peripheral left) and right (9 mm from the specimen's margin = peripheral right) from the middle of the specimens.

Statistical Analyses

A Shapiro-Wilk test verified the normal distribution of the data. A multivariate analysis (general linear model) assessed the effect of the parameters LCU (three different LCUs), LCU-rotation ($3 \times 120^\circ$), RBC (three different BF-RBCs), depth (0.5 to 5.5 mm), and width (peripheral and center) as well as their interaction terms on HV and DOC. The partial eta-squared statistic reported the practical significance of each term, based on the ratio of the variation attributed by the effect. Larger values of partial eta-squared indicate a greater amount of variation accounted for by the model, which figure up to a maximum of 1.0. In addition, a one-way analysis of variance (ANOVA) with post hoc Tukey HSD test was used. In all statistical tests, p values < 0.05 were considered statistically significant when using SPSS Inc (version 23.0, Chicago, IL, USA).

RESULTS

The incident irradiance was identified as 1786.0 ± 56.9 mW/cm² for Elipar DeepCure, 1550.8 ± 50.3 mW/cm² for Demi Ultra, and 1184.6 ± 3.9 mW/cm² for Bluephase Style (Fig. 2a). Elipar DeepCure and Demi Ultra were identified as blue-LED LCUs, with a peak maximum located at 449 nm and 460 nm, respectively. Bluephase Style is a violet-blue LED-LCU, with two distinct peaks at 410 nm (violet) and 456 nm (blue; Figure 2b).

The effect of the parameter LCU rotation on HV was not significant ($p=0.109$) in any of the analyzed LCUs. Therefore, data of the three rotations of an LCU were pooled together. No significant difference was identified in HV for the positions peripheral-left and peripheral-right ($p=0.615$), while the difference

between peripheral-left and center ($p<0.001$) as well as peripheral-right and center ($p<0.001$) was significant. Consequently, the peripheral-left and peripheral-right data were merged as well. All other analyzed parameters, LCU, RBC, depth, and width (defined now as either peripheral or center), exerted a significant ($p<0.001$) effect on HV. The highest influence on HV was exerted by the parameter RBC ($p<0.001$; partial eta squared $\eta_p^2=0.805$), followed by the parameter depth ($p<0.001$; partial eta squared $\eta_p^2=0.788$; Figure 3). As a result, statistically similar HV values were identified in a depth of 0.5 mm for SonicFill (SF) and Filtek Bulk-Fill (FBF; $p=0.145$) that were significantly higher compared with values measured in Tetric Evo Ceram Bulk Fill (TEVOBF; $p<0.001$; Figure 4a). At a depth of 4 mm, SF and TEVOBF showed statistically similar HV values ($p=0.06$) that were lower compared with FBF ($p<0.001$; Figure 4b). The effect of width on HV ($p<0.001$; partial eta squared $\eta_p^2=0.292$; Figure 5a-c) was consistently lower and the effect of LCU was still significant but very low ($p<0.001$; partial eta squared $\eta_p^2=0.057$).

The DOC varied among 3.46 mm (SF, peripheral, LCU Bluephase Style) and 5.50 mm (FBF, center, all LCUs; Figure 6). The highest influence on DOC was exerted by the parameter RBC ($p<0.001$, $\eta_p^2=0.774$), followed by width ($p<0.001$, $\eta_p^2=0.554$), while the influence of the LCU was extremely low ($p<0.06$, $\eta_p^2=0.070$). The DOC measured peripheral or center was similar in FBF ($p=0.124$). DOC measured peripheral, compared with the DOC measured center, was at 1.12 ± 0.11 mm lower in TEVOBF ($p<0.001$) and at 1.04 ± 0.11 mm lower ($p<0.001$) in SF.

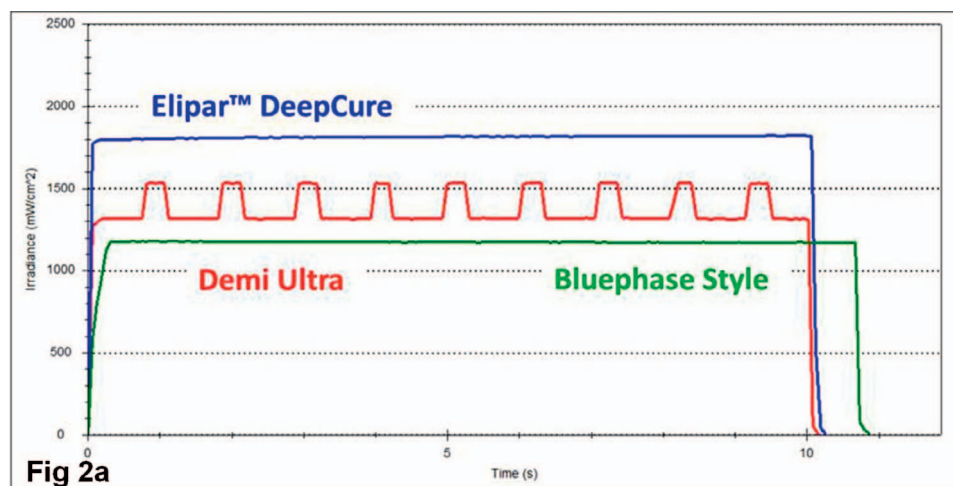
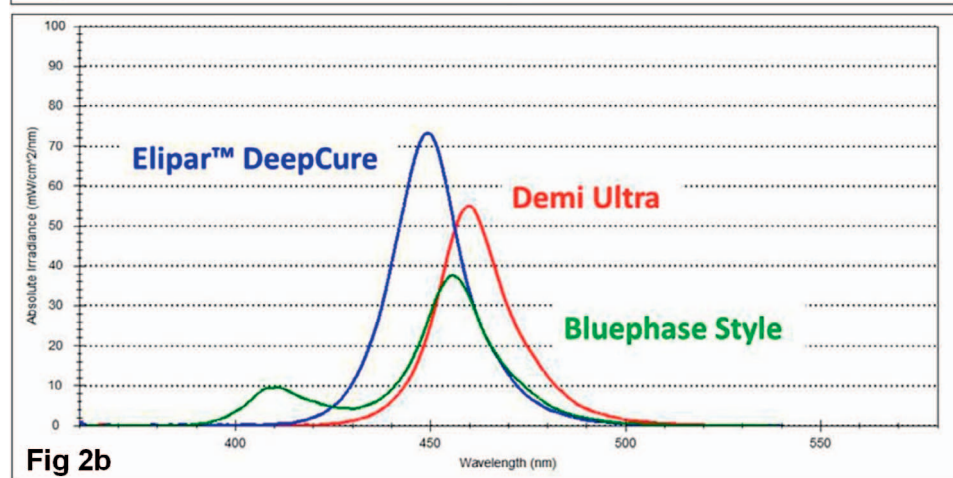


Figure 2. Characteristics of the LCUs collected at 3-mm distance from the spectrophotometer sensor. (a): LCU irradiance. (b): Emission light spectrum.



FBF reached the significantly highest DOC of 5.4 ± 0.2 mm ($p < 0.001$), while the DOC in TEVOBF (4.1 ± 0.7 mm) and SF (3.9 ± 0.6 mm) was statistically similar ($p = 0.168$). As for the LCUs, a statistically significant difference in the induced DOC was recorded only between Elipar DeepCure (4.6 ± 0.8 mm) and Demi Ultra (4.3 ± 0.9 mm; $p < 0.001$), while Bluephase Style induced a DOC of 4.5 ± 0.89 mm that was statistically similar to Elipar DeepCure ($p = 0.202$) and Demi Ultra ($p = 0.089$).

DISCUSSION

The fundamental approach of this study was to identify the efficacy of modern blue and violet-blue LED LCUs in curing adequately peripheral zones in large BF-RBC specimens. This attempt was founded in the notably heterogeneous light distribution of contemporary LCUs²⁵ that might produce irregular polymerization and inhomogeneity in mechanical properties within a RBC filling.²¹ Consequently, much effort has been currently invested to develop LCUs displaying homogeneous light beam profiles.

A recently launched LED LCU, Elipar Deep Cure-S, evidencing homogeneous light beam profile along a light guide²⁵ with a diameter of 10 mm, was used as a positive reference in the present study. The emission spectrum of Elipar Deep Cure-S indicates that the peak maximum in the blue wavelength range is shifted to a lower wavelength (449 nm) when compared with the other analyzed LCUs (460 nm and 456 nm; Figure 2b). The particularity of the blue-LED LCU Demi Ultra is seen in positioning the LEDs at the tip. This allows avoiding energy loss vs LCUs with a bent light guide and also to reduce light collimation. Two alternating levels of irradiances were identified in Demi Ultra: a base level, set at 1300 mW/cm² and maintained for 0.75 seconds followed by a higher irradiance level (1550 mW/cm²) for the subsequent 0.25 seconds (Figure 2b). This switch in irradiance is repeated for each second of the curing cycle and is intended to reduce heat buildup. Apart from blue LED LCUs, a violet-blue LCU, Bluephase Style, has also been analyzed. This LCU is characterized by a lower irradiance com-

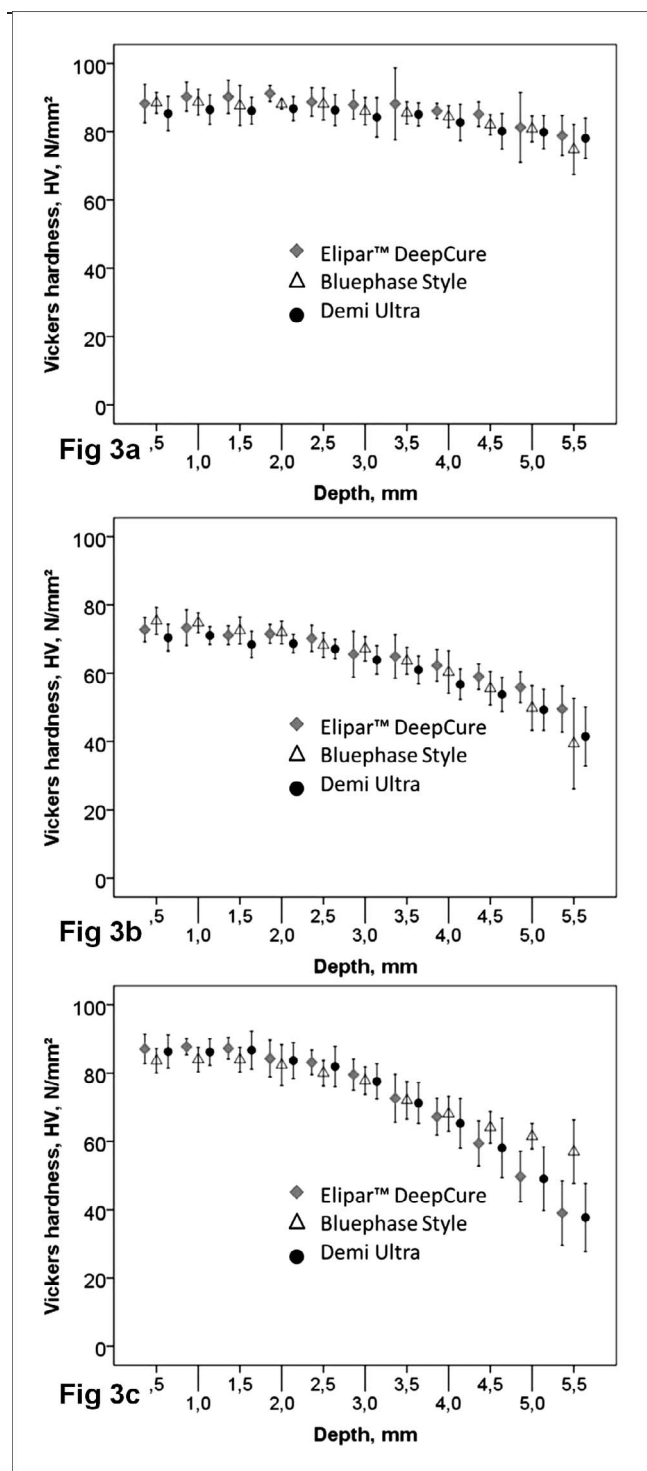


Figure 3. Mean and standard deviation of the Vickers hardness as function of depth and LCU for the bulk-fill composites. (a): FBF. (b): TEVOBF. (c): SF. Compared with values measured at a depth of 0.5 mm, one-way ANOVA ($p < 0.05$) identified in FBF a significant decrease in HV starting with a depth of 4.5 mm (Bluephase Style) or 5 mm (Elipar DeepCure and Demi Ultra). The corresponding depths were 2.5 mm (Bluephase Style) and 3 mm (Demi Ultra and Elipar DeepCure) in TEVOBF and 3 mm in SF (all LCUs). FBF: A significant difference among LCUs was identified only at 1-, 1.5-, 4-, and 4.5-mm

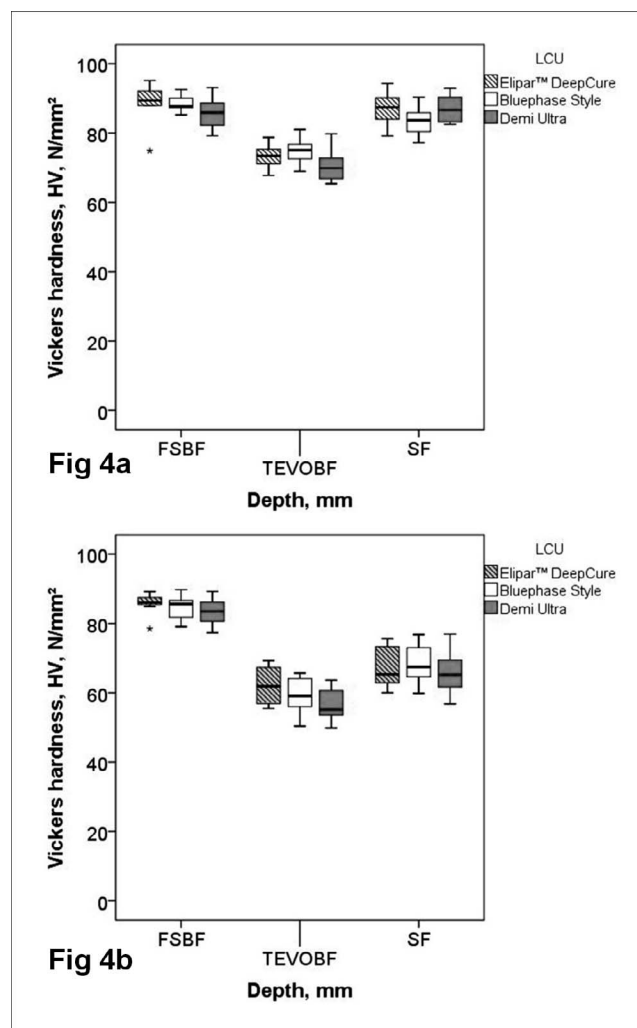


Figure 4. Vickers hardness as a function of material and LCU at depths of (a) 0.5 mm and (b) 4 mm.

depths (Elipar DeepCure induced significantly higher HV values compared with Demi Ultra, while Bluephase Style performed comparably to both LCUs). TEVOBF: Bluephase Style induced significantly higher HV values than Demi Ultra up to a depth of 2 mm, while Elipar DeepCure performed comparably to both LCUs. Subsequently, up to a depth of 3.5 mm, all three LCUs had statistically similar performance. Following that, Elipar DeepCure performed significantly better compared with Demi Ultra at 4 and 4.5 mm, while Bluephase Style performed comparably to both LCUs. At depths up to 5.5 mm, Elipar DeepCure performed significantly better compared with both other LCUs. SF: Elipar DeepCure induced significantly higher HV values than Bluephase Style up to a depth of 1 mm, while Demi Ultra performed comparably to both LCUs. Subsequently, up to a depth of 4 mm, all three LCUs had a statistically similar performance. At 4.5 mm, Bluephase Style performed significantly better than Demi Ultra, while Elipar DeepCure performed comparably to both LCUs. Bluephase Style then performed significantly better compared with the other LCUs.

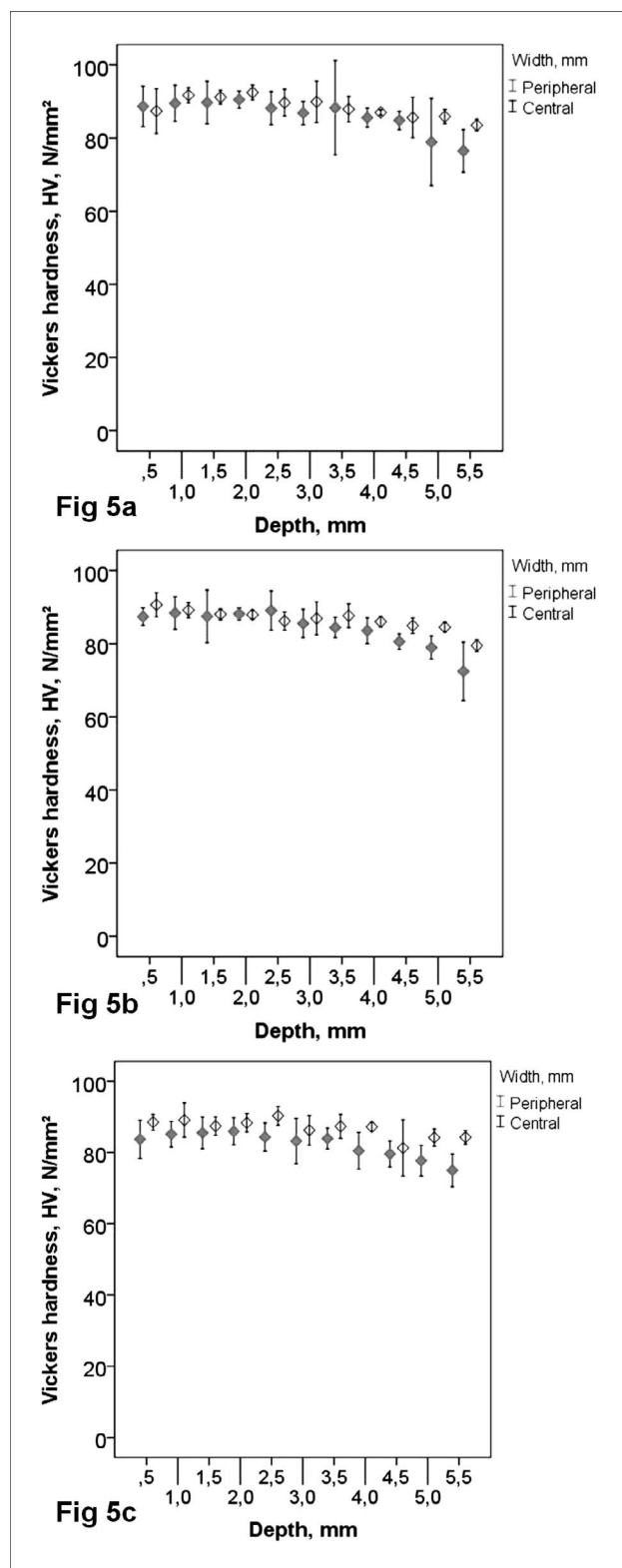


Figure 5. Mean and standard deviation of the Vickers hardness as a function of depth and width (center and peripheral position) after curing with the LCUs. (a): Elipar DeepCure. (b): Bluephase Style. (c): Demi Ultra, exemplified for the bulk-fill composite FBF.

pared with the above described LCUs, but the emission spectrum covers a wider wavelength range (390-500 nm) that allows initiating camphorquinone (CQ) and also alternative photo-initiators. This is particularly important when curing TEVOBF, for it contains, apart from a CQ/amine system that is present in FBF and SF, also two Norrish Type 1 initiators, namely, a dibenzoyl germanium derivative, bis-(4-methoxybenzoyl)diethylgermane), and an acyl phosphine oxide (APO).

Germanium-based photo-initiators for dental materials are characterized by lower absorption maxima ($\lambda_{\max} = 411$ nm or $\lambda_{\max} = 418$ nm) when compared with CQ ($\lambda_{\max} = 468$ nm).²⁶ They match accordingly well the emission spectrum (Figure 2b) of the violet-blue LCU Bluephase Style. The absorption maxima of germanium-based photo-initiators (411 nm, 418 nm) lie outside the emission spectra of blue LED LCUs (Figure 2b). However, their absorption spectra start in the violet wavelength range and extend up to 455 nm in the blue wavelength range, matching in large parts the emission spectra of blue LED LCUs.

It seems that the wider emission spectrum of Bluephase Style that initiates CQ, APO, and the germanium-based photo-initiator compensates for its lower irradiance, since it induced in TEVOBF statistically similar DOC values as Elipar DeepCure and Demi Ultra. Comparing both analyzed blue LED LCUs, it become evident that the emission spectrum of Elipar DeepCure starts at lower wavelength (410 nm) compared with Demi Ultra (425 nm), thus providing a better matching with the absorption spectrum of the germanium-based photo-initiator. Both effects—a slightly higher irradiance (Figure 2a) and a better spectra match with the germanium-based photo-initiator—may contribute to a slight but significant improvement in mechanical properties in TEVOBF when using Elipar DeepCure vs Demi Ultra.

Apart from spectra-matching considerations, the reactivity of the germanium-based photo-initiator was identified to be much higher compared with CQ, a fact attributed to the larger molar extinction coefficient ($\epsilon\lambda$).²⁶ A large $\epsilon\lambda$ indicates a high probability of light absorption at a certain wavelength, leading to large quantum yields of the initiating species and improved degree of conversion. For germanium-based initiators, a significantly stronger absorption at their maximum was identified ($\epsilon = 1460$ dm²mol⁻¹ and $\epsilon = 5470$ dm²mol⁻¹) in comparison to CQ ($\epsilon = 380$ dm²mol⁻¹).²⁶

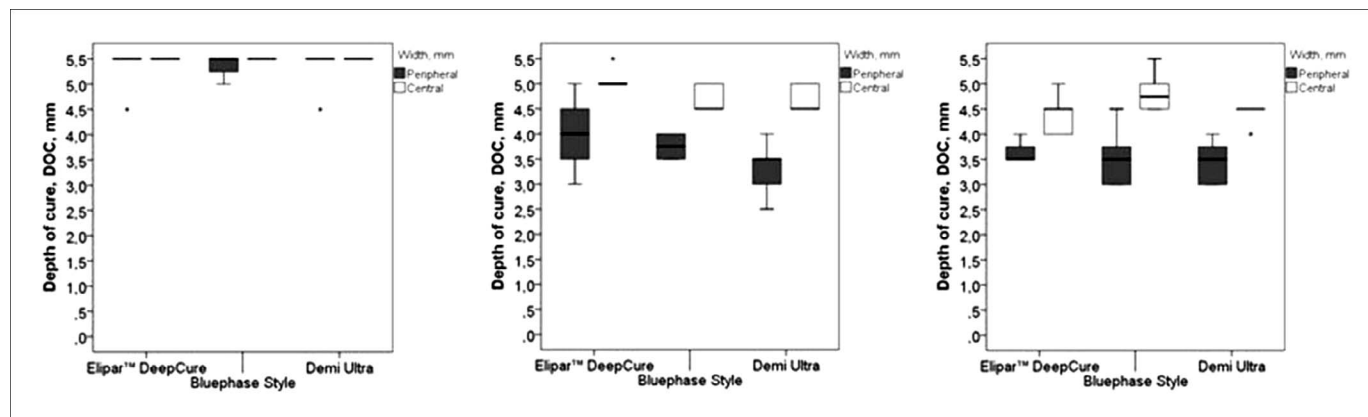


Figure 6. Depth of cure as a function of material and LCU. (a): FBF. (b): TEVOBF. (c): SF.

As for the APO initiator that is also contained in TEVOBF, it can be activated only by the violet-blue LCU Bluephase Style.²⁷ Similar to the germanium-based photo-initiators, APO is more efficient than CQ, since it undergoes homolytic α -cleavage of the C-P bond and generates two free radical species, that is, $[-(O=C)\bullet]$ and $[\bullet P(=O)<]$, both capable of initiating polymerization.²⁷ Albeit very efficient, APO is activated at shorter wavelengths compared with CQ, while the attenuation of light in depth is known to follow an exponential decay (Lambert's Law)²⁸ and is wavelength dependent. Hence, the violet light that is characterized by shorter wavelengths will be attenuated faster in depth than the blue light.²² The contribution of APO to the polymerization process of TEVOBF in deeper layers must therefore be considered as very low.

When comparing the quality of curing in center vs peripheral areas, differences in HV are low and started to be perceptible only at depths larger than 2.5 mm. It was also found in the present study that this difference is material dependent. Interestingly, the DOC measured for FBF was neither dependent from the LCU nor from the width (center-peripheral), attesting to a sufficiency of curing in peripheral areas under all analyzed curing conditions. As for TEVOBF, both LCU ($\eta_p^2=0.28$) and width ($\eta_p^2=0.672$), but not their interaction product, exerted an effect of DOC. Related to Elipar DeepCure, the DOC induced by Bluephase Style in TEVOBF was about 0.338 ± 0.141 mm lower, while the DOC induced by Demi Ultra was about 0.583 ± 0.137 mm lower. The peripheral areas cured with Demi Ultra reached in TEVOBF a mean DOC value that was lower than the 4-mm limit stipulated as a minimum for adequate curing in bulk-fill resin

composites. It must, however, be emphasized that TEVOBF specimens were cured for only 10 seconds compared with 20 seconds as done in all other materials and thus received half of the radiant exposure applied on the other materials. It becomes obvious that exposure duration of 10 seconds cannot be generalized for curing this material, since one of three analyzed LCUs induced peripheral DOC values lower than 4 mm. An exposure time of 10 seconds might become insufficient at peripheral regions of large fillings. As for SF, only the width showed an influence ($\eta_p^2=0.658$) on DOC, but not the LCU, while all measured peripheral DOC values were lower than 4 mm. In contrast, values measured in the center region exceeded this limit. This fact must be related to the higher filler content in SF, which generates a less translucent material in which light is more strongly attenuated. For more homogeneous properties, it is suggested to cure this material longer as recommended by the manufacturer, thus as long as 20 seconds.

The DOC was defined in the present study as the depth at which HV equals 80% of the HV value measured at a subsurface of 0.5 mm. While this method is well established, there might be reticence in accepting a 20% decrease in mechanical properties as an adequate curing. Besides, the used LCUs were applied in this study design perpendicular to the specimen's surface, at an exposure distance of 3 mm that simulates clinically relevant curing conditions for curing BF-RBCs fillings. All deviations from these curing conditions owing to a particular clinical situation, such as increased exposure distance or variances from perpendicularity when applying the LCU on the filling, must result in an enhanced exposure time.

CONCLUSIONS

The analyzed LCUs showed less to no difference in their effect on the measured properties within one BF-RBC. The placement of the LCU by rotation in 120° steps as well as the peripheral positions measured 4-mm left and right apart from the center showed no effect on the analyzed properties in any material.

Whether a BF-RBC filling is cured peripherally as well as in the center depends more on the material than on the curing unit used.

Acknowledgements

The work was supported by the Department of Operative/ Restorative Dentistry, Periodontology and Pedodontics, Ludwig-Maximilians University of Munich, to 75% and by the company 3M-ESPE to 25%.

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.

(Accepted 22 July 2017)

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