

CURING EFFECTIVENESS OF SINGLE-PEAK AND MULTI-PEAK LED LIGHT CURING UNITS ON TPO-CONTAINING RESIN COMPOSITES WITH DIFFERENT CHROMATIC CHARACTERISTICS

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SUMMARY

This study evaluated the surface microhardness of Lucirin-TPO (TPO) containing resin based composite (RBC) cured with three light-emitting diode (LED) light curing units (LCUs) (two single-peak LED and one multi-peak LED), and two different energy density (ED) (8 J/cm² and 16 J/cm²). Ninety specimens (8 mm wide and 2 mm thick) (n=5), were prepared with three different shades: translucent (T), A2 dentin (A2d), and A4 dentin (A4d). Specimens were subjected to micro-hardness Vickers measurements (Vickers Hardness Number, VHN) on both top and bottom surfaces. Hardness ratio (rHV) was also calculated. Data were analyzed using multifactorial ANOVA and Bonferroni tests (≤ 0.05). Results indicated that higher ED performed better than lower ED. Multi-peak LED achieved higher VHNs and rHV than single-peak LED when curing a TPO-containing RBC. A4d invariably achieved lower rHV and VHN than T and A2d. Single-peak LED achieved comparable VHNs and rHVs with multi-peak LED only curing A2d and T shades with 16J/cm².

Key words: resin composite, LED, shade, lucirin TPO, microhardness.

Introduction

Degree of conversion (DC) is an important parameter to evaluate the final physical, mechanical and biological properties of photo-activated resin-based composites (RBCs) (1). It has been shown that RBCs properties improve as the DC obtained during photo-polymerization increases (2). An inadequate DC may results in reduced hardness, decreased dynamic elastic modulus, reduced flexural and fractural strengths, decreased biocompatibility due to the leach of un-

reacted monomers (3), and increased wear and breakdown at the margins of the resin restoration (4-6). Therefore, DC plays an important role in determining the ultimate success of restorations (4-6).

Several parameters influencing polymerization of RBCs should be considered: RBCs' chemical composition, amount and size of filler particles, type of photoinitiators, spectral output emitted by light curing units (LCUs), light intensity delivered by LCUs to the polymer (power density), exposure time, total amount of light transmitted by LCUs (energy density), and distance between

light guiding tip and RBCs (5-9). Shade and translucency of RBCs should also be considered because light passing through the material is absorbed and scattered, resulting in an attenuation of the intensity and subsequent reduction of light available for activate curing process (10-13).

It was extensively shown that energy density (ED) (i.e. power density*exposure time) delivered to RBCs is more strictly correlated to their DC than power density or exposure time by their self (14). To achieve an adequate DC, RBCs require a total energy ranging from 6 to 24 J/cm² for 2-mm increment of material depending on brand and shade (15, 16). On the other hand the same ED could be achieved using higher power density for few seconds as well as using a lower power density for longer exposure time.

A variety of LCUs has been developed ranging from quartz-tungsten-halogen (QTH), PAC and the argon LASER (14, 17). Light emitting diodes (LEDs) represent the alternative light source to traditional halogen units (18). Second generation single-peak LED LCUs produce a narrow spectrum of light that usually peaks in the 450 to 470 nm range of wavelength (19-21), which matches camphorquinone's (CQ) absorbance wavelength (19-21). On the other hand, alternative photoinitiators [e.g., 2,4,6-trimethylbenzoyldiphenylphosphine oxide (Lucirin TPO) and 1-phenyl-1,2-propanedione (PPD)], more sensitive to shorter wavelengths (< 420 nm), have been introduced in some RBCs (22-27). The use to mix diverse photoinitiators leads to an improvement of DC and better esthetic qualities (28) and reduces the rate of stress (29). Manufacturers do not state all photoinitiators used in their products; moreover various photoinitiators could be used in different shades within the same brand (24, 30). The presence of these photoinitiators may reduce photoactivation effectiveness when a single-peak LED lights (450-470 nm) is used (8). The introduction of "so called" third-generation multi-peak or polywave LED LCUs, provided with additional light output in the 400 to 415 nm range of wavelengths, is supposed to overcome that problem (8, 20, 21). Although single-peak and multi-peak lights

can produce similar overall power output (watts) and deliver the same power density (mW/cm²) to the restoration, differences in their spectral outputs may have a significant effect on the photoinitiating system (30).

The DC of RBCs can be measured using different testing techniques. Direct methods, such as infrared spectroscopy and LASER Raman spectroscopy, have not been accepted for routine use because complex, expensive and time consuming (31-34). Conversely, indirect methods, such as scraping, visual examination, dye uptake, Knoop and Vickers surface hardness tests are more widely used in the literature (35-38).

Although there are some studies evaluating the influence of chroma and translucency on the DC of CQ containing RBCs cured with QTH LCUs (39-43) or single-peak LED, few have been published on the influence of TPO-containing RBCs chromatic characteristics using single-peak or multi-peak LED LCUs (44). Therefore, the aim of the present study was threefold: to evaluate the influence on TPO-containing RBCs Vickers surface micro-hardness of (i) energy density (ED-8 J/cm² and 16 J/cm²), (ii) type of LCUs (second-generation single-peak LED, third-generation multi-peak LED) and (iii) material's shade.

Methods and materials

Materials and LCUs tested in the present study are reported in Table 1.

LCUs' emission spectra, irradiance and total energy were obtained using MARC™ (BlueLight Analytics Inc., Halifax, NS, Canada) which incorporates two spectroradiometers (USB 4000, Ocean Optics, Dunedin, FL).

All three RBCs incorporate in addition to CQ, the TPO as photoinitiator.

Ninety standardized specimens were assigned to 18 groups (n=5) using three variables: ED, LCU type, and RBCs' shade.

Cylindrical specimens were prepared using stainless steel molds (8 mm diameter and 2 mm

Table 1 - Materials used in the present study were listed in the present Table.

Material	Shade	Manufacture	Composition	
Tetric EvoCeram	A2 Lot N30152	Ivoclar Vivadent, Schaan, Liechtenstein	<i>bis</i> -GMA, urethan dimethacrylate, ethoxylated <i>bis</i> -EMA (16,8 wt%); barium glass filler, ytterbiumtrifluoride, mixed oxide (48,5 wt%); prepolymers (34%); additives, catalysts, stabilizers an pigments (<1 wt%)	
	A4 Lot M35813			
	T Lot L26216			
LCUs	Type	Manufacturer	Wavelength peak	Operating mode
Smartlite iQ (SQ)	Single-peak	Dentsply DeTrey, Konstanz, Germany	462 nm	-
Starlight Pro (SP)	Single-peak	Mectron S.p.A. Medical Technology, Carasco, Italy	450 nm	-
Valo (VA)	Multi-peak	Ultradent, South Jordan, UT, USA	465 nm; 445 nm; 405 nm	Standard

thick). A light brown sheet covered with a Mylar strip (KerrHawe SA, Bioggio, Switzerland) was placed on the bottom of the mold to resemble deep dentin and enhance a smooth surface. The mold was packed with RBC material in one single apposition and covered with another Mylar strip. Microscope slide was pressed by 1 Kg load against the top of the mold to extrude excesses and obtain a flat surface and then removed. All tested LCUs' light guiding tip was placed in direct contact with the upper Mylar strip of each sample. Specimens were cured with 8 J/cm² or 16 J/cm² ED values according to the group of appearance. Exposure times (ET) to deliver selected ED were calculated (ET=ED/power density) and approximated to the nearest second.

The surface exposed to direct light radiation was defined top, while the surface that received light through the material was named bottom. The surface of RBC specimens was not ground nor polished before testing because this would have generated heat, causing further polymerization within the material.

For microindentation hardness testing, a micro-durometer (Shimadzu Corporation, Tokyo, Japan) was used submitting all specimens to a 50-g load for 45 s.

Three indentation tests, with a minimum distance of 100 µm each other and near to the cen-

ter of the specimens, were carried out on the top and bottom surfaces. Micro-hardness measurements were calculated in Vickers pyramid number (VHN 5 · 10⁻²/45 following VHN). Hardness ratio (rHV) was also calculated by the formula rHV=bottom VHN/ top VHN.

All data analysis was performed using the Statistical Package for the Social Sciences Windows, version 15.0 (SPSS, Chicago, Illinois, USA). Descriptive statistics consisted of the mean ± SD for variables with Gaussian distributions (after confirmation with histograms and the Kolgomorov-Smirnov test) or median (min-max) for parameter categorical and non-parameters. Comparisons among data groups were carried out with multifactorial ANOVA followed by a Bonferroni test for multiple comparisons. Statistical significance was set at a *p* value <0.05.

Results

LCUs power density at the light guiding tip exit and ET to achieve ED respectively of 8 J/cm² and 16 J/cm² are reported in Table 2.

Spectral absorbance of TPO and CQ and emission's spectral distributions of LCUs tested are presented in Figure 1.

Table 2 - Exposure times to achieve 8 J/cm² and 16 J/cm² were calculated according to different LCUs power densities (ET=ED/PD) and are reported in the present Table.

LCU	Power Density	Exposure Time 8 J/cm ²	Exposure Time 16 J/cm ²
Smartlite iQ (SQ)	712 mW/cm ²	11 s	22 s
Starlight Pro (SP)	753 mW/cm ²	11 s	22 s
Valo (VA)	1224 mW/cm ²	7 s	14 s

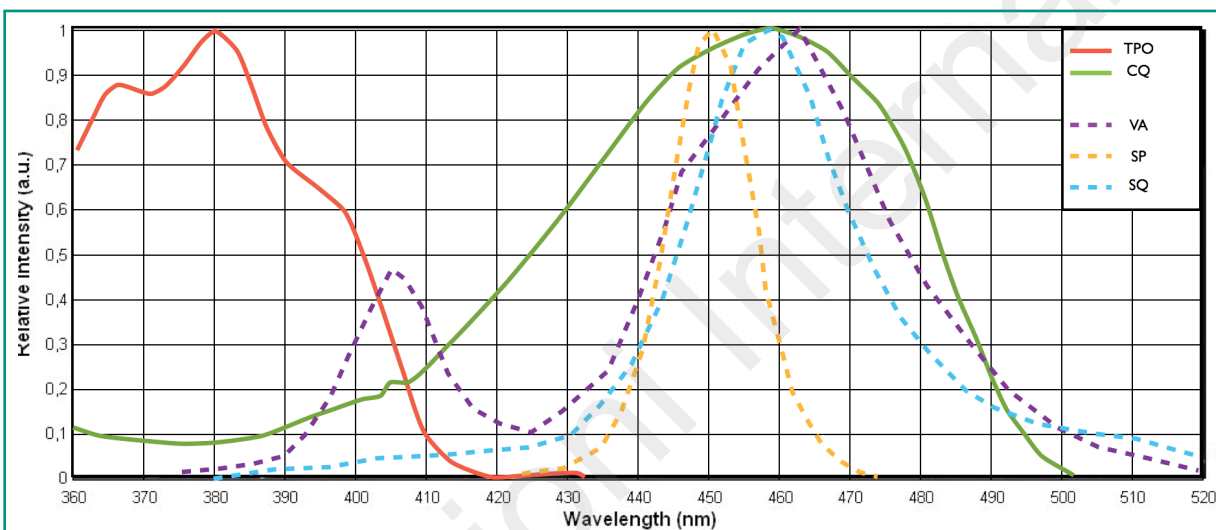


Figure 1
Normalized absorption spectra of Camphoroquinone (CQ) and Lucirin TPO (TPO) are reported in the present graph together with normalized emission spectra of Valo (VA) Smartlite iQ (SQ) and Starlite Pro (SP) LCUs.

The means top and bottom VHN of all specimens after curing with 8 J/cm² or 16 J/cm² are presented in Table 3.

Hardness ratios are shown in Figure 2.

Sample size allowed a statistical power of 1-beta=0,999. The multifactorial ANOVA showed that ED, LCU, and RBC's shade significantly affect composite micro-hardness ($p<0.001$) both on the top and bottom surfaces.

Multifactorial Anova showed that top VHN values were statistically higher ($p<0.0001$) than bottom VHN values for any shade, LCU and ED tested. Therefore rHVs were invariably <1.

Bonferroni test for multiple comparison showed that:

- **Inter-energy density analysis:** Specimens

cured with 8 J/cm² achieved top and bottom VHNs lower than those cured with 16 J/cm² ($p<0.05$). Conversely rHV of specimens cured with 8 J/cm² were generally higher than those of specimens cured with 16 J/cm² although differences were not statistically significant ($p>0.05$).

- **Inter-LCU analysis:** VA achieved higher bottom VHN values compared to SP and SQ. Differences between groups were all statistically significant ($p<0.05$). Regarding rHV, VA performed statistically better than other two LCUs ($p<0.05$) although differences between SP, SQ rHVs were not statistically significant ($p>0.05$).

- **Intra-LCU analysis:** Delivering 8 J/cm² VA

Table 3 - Means and SD Vickers Hardness Number (VHN) of A2d, A4d and T specimens cured with Valo (VA), Smartlite Q (SQ), Stralight Pro (SP) and QHL 75 (Q75) in continuous mode for 8 J/cm² and 16 J/cm².

		VA	SQ	SP
8J/cm ²	T	A2d 59,91 (0,97)	61,57 (2,82)	58,18 (2,31)
		A4d 50,39 (1,96)	44,80 (3,14)	68,36 (3,73)
		T 66,20 (1,43)	64,85 (3,44)	61,40 (1,35)
	B	A2d 55,15 (2,68)	57,51 (1,83)	46,77 (8,13)
		A4d 48 (3,22)	33,93 (2,49)	45,45 (3,59)
		T 55,37 (1,73)	56,81 (5,27)	57,55 (1,76)
16J/cm ²	T	A2d 76,45 (8,45)	72,48 (3,27)	64,33 (2,41)
		A4d 55 (2,98)	50,37 (2,20)	70,25 (3,55)
		T 72,03 (4,70)	67,01 (5,43)	69,10 (2,43)
	B	A2d 64,35 (1,47)	60,88 (2,50)	58 (2,78)
		A4d 52,10 (1,66)	38,22 (2,70)	46,2 (3,77)
		T 57,36 (3,05)	56,24 (1,82)	59,38 (1,28)

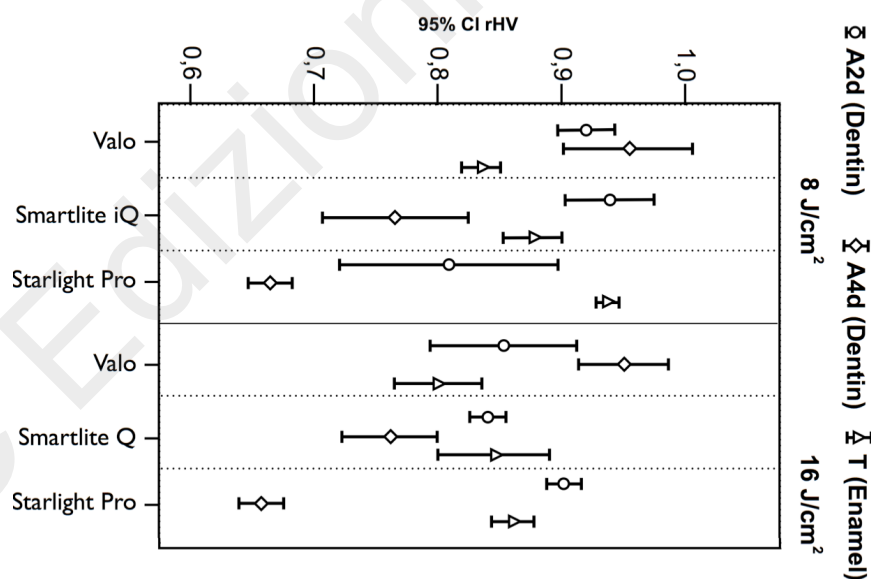


Figure 2

Mean Hardness ratio values (rHV) and standard deviations of different shade TPO containing RBC (A2d, A4d, T) after photocuring with multi-peak (VA) or single-peak LED LCUs (SQ, SP) with different ED (8J/cm² and 16 J/cm²) are summarized in the present graph.

and SQ achieved comparable bottom VHN values with A2d and T shades which were

significantly higher than those obtained with A4d shade. Similarly SP achieved higher

VHN values with T shade than with A2d and A4d while differences between A2d and A4d were not statistically significant.

Delivering 16 J/cm² all tested LCUs achieved significantly higher bottom VHN values with T than with A4d. A2d, in turn, obtained higher bottom VHN values than T with all LCUs although differences were statistically significant only for VA and SQ.

On the other hand, only VA achieved statistically significant higher rHV with A4d than with T shade both with 8 J/cm² and 16 J/cm² ED values. Regarding rHV VA performed better with A4d than with A2d although differences were statistically significant only with the higher ED tested. Single-peak LCUs achieved statistically lower rHV values with A4d than with A2d and T.

- **Inter-shade analysis:** A2d and T achieved higher bottom VHN and rHV than A4d ($p < 0.05$) while difference between A2d and T where not statistically significant ($p < 0.05$).
- **Intra-shade analysis:** Curing with 8 J/cm² A2d resulted in statistically significant higher bottom VHN when cured with VA and SQ than with SP ($p < 0.05$). Using 16 J/cm² ED, dentin shades, A2d and A4d, performed statistically better with VA than with other LCUs.

A4d shade achieved statistically significant higher bottom VHN when cured with VA and SP than with SQ both with 8 J/cm² and 16 J/cm² ED values ($p > 0.05$).

T shade achieved comparable bottom VHN values with all LCUs and both ED tested ($p > 0.05$). Analyzing rHV values, A2d cured with 8 J/cm² with VA and SQ achieved statistically better performance than with SP ($p < 0.05$). Using 16 J/cm² A2d achieved statically comparable results with all LCUs tested ($p > 0.05$). Regardless to ED used A4d achieved statistically higher rHVs with VA than others LCUs. On the other hand, T shade, when cured with 8 J/cm², achieved statistically higher rHV with SP rather than with other LCU tested ($p < 0.05$). With 16 J/cm² differences between LCUs curing the T shade were not statistically significant ($p > 0.05$).

Discussion

A high DC is necessary to obtain good physical properties and low cytotoxicity of RBCs (45).

CQ combined with tertiary amine still represents the most widespread photoinitiating system in RBCs and dental adhesives. However other initiators have been introduced such as trimethylbenzoyl-diphenylphosphine oxide (TPO) mainly to overcome problems derived from CQ's intensive yellow color and relative color instability. TPO-containing RBCs showed comparable or higher DC and greater color stability than those containing CQ and tertiary amines (8, 9). Moreover TPO has a higher value of light absorption than CQ: this indicate that the use of such a photoinitiator confers higher polymerization efficiency (8). On the other hand, beside a lower curing depth due to the shorter wavelength required, that scatters more than the common wavelength needed for CQ (46, 47), one of the major drawbacks of TPO is represented by the mismatch between its absorption spectra, shifted towards the UV range, and the emission spectra of commonly used single-peak LED LCUs (Figure 1). Despite this relevant issue some manufacturers have started to introduced TPO into their RBCs. Beside manufacturers statements, Santini et al. (48) reported the presence of TPO initiator system in Tetric EvoCeram A1 (Ivoclar Vivadent, Schaan, Liechtenstein) and Vit-l-essence OW (Ultradent Products Inc., South Jordan, UT, USA). Poly-wave LCUs, provided with additional LED chips delivering shorter wavelengths of emission spectra, were marketed with the aim of effectively cure alternative initiators-containing RBCs. Such types of LCUs are produced also by the two above mentioned manufacturers: Valo (Ultradent Products Inc., South Jordan, UT, USA) and Bluephase G2 (Ivoclar Vivadent, Schaan, Liechtenstein).

It is therefore important for the clinician to know whether the applied TPO-containing RBC will be optimally cured with different LCUs and curing regimens and whether the shade of the material could have any influence on the curing

process.

The purpose of the present study was to investigate the effect on surface Vickers micro-hardness of ED, LCU used and chromatic characteristics (shade and translucency) of a TPO-containing RBC. In the present study, with the aim of better compare LCUs for their emission spectra rather than for their power density, it was decided to use uniform ED for all LCUs by changing the exposure time as reported in Table 2.

RBC's cure can be evaluated with different methods as described by Yearn (31). Hardness is related to mechanical strength, rigidity, and resistance to intraoral softening (49). It has also been shown that hardness correlates with the DC (39). Although a low ED may adequately cure the closest surface of resins, number of photons reaching the bottom of thick and opaque composites is reduced exponentially (31, 50-53). Insufficient polymerization of bottom surfaces increases the risk of bulk and marginal fractures (4-6). Consequently, testing the hardness only at the top of the restoration seems to be a poor predictor of the hardness at the bottom (15, 52, 54). Thus, to ensure proper polymerization throughout the RBCs, as previously suggested by many Authors (15, 52, 54) Vickers hardness measurements were performed both at the irradiated (top) and at the non-irradiated (bottom) surfaces of the specimens. Moreover, Ferracane (55) stated that an absolute hardness number could not be used to predict DC when different composites were compared; thus, the hardness ratio (rHV) between the bottom and top surfaces have been introduced.

Differences in amount, size and type of filler particles and alternative photosensitizers used may exist for RBC of the same system with different shades and translucencies (24, 25, 30). Results of the present study confirmed differences in top VHN of different shade specimens of the same material cured with the same polymerization regimen. Thus, VHN top and bottom should be considered together with rHV when comparing curing effectiveness of different shade RBC (56).

The experimental results confirmed that shade,

LCU and ED may influence RBC's top and bottom surface micro-hardness. The results of the present study also confirmed findings of several previous studies where top VHN were invariably higher than bottom VHN (41, 57, 58). Therefore rHVs were constantly <1 demonstrating a decrease in the surface Vickers microhardness, and thus of the DC, proceeding from the top to the bottom surface even in 2 mm thick specimen. This is consistent with the reduction of light intensity and effectiveness as long as the light passes through the RBC.

As shown in the present study, specimens cured with 16 J/cm² achieved higher surface hardness. Indeed, differences were statistically significant ($p<0.05$) both at the top and the bottom surfaces, confirming, as many other studies, that higher ED improve curing effectiveness (59). Therefore, after 8 J/cm² direct light exposure, top VHN of TPO-containing RBC resulted still improvable. On the contrary ED do not significantly influenced rHVs ($p>0.05$).

Moreover, a statistically significant difference was revealed ($p<0.05$) between top VHN values of A2d, A4d, and T specimens cured with the same LCU and ED. This could be explained by manufacturing undeclared differences in filler and photosensitizer content in shades tested. Under the condition of the present study it is not possible to find out how much those results are correlated with differences in RBC chemical composition and how much with differences in the DC of diverse shade RBCs. Basing on this early finding, besides the importance of top VHN by itself, it is important to point out how looking only at the bottom VHN could be quite a weak way of interpreting results deriving from RBCs of the same brand but differing on shade. Indeed it is presumable that, aside from DC, material's composition influences the bottom surface as well as the top surface. On the other hand, as previously suggested by Nakfoor et al., (56) also rHV alone may be misleading, and should be examined together with the actual surface VHN to ensure a LCU's curing effectiveness on a RBC. For example, the above mentioned lack of significant differences between

rHV of specimens cured with higher and lower ED reveal the concurrent increase of top and bottom VHN rather than the absence of curing improvement between 8 J/cm² and 16 J/cm².

In agreement with several other Authors (30, 48, 60), the results of the present study confirm that, curing a TPO-containing RBC, the multi-peak LCU VA achieved statistically higher bottom VHN and rHV than other LCUs. Among single-peak LED SP achieved statistically higher bottom VHN than SQ, although rHV were not statistically significant. Once again this could be attributed more to a concurrent increase of top and bottom VHNs rather than to the absence of an actual curing improvement. Therefore, comparing different LCU performances with the same material, seems to be preferable to refer to bottom VHNs than to rHVs.

On the other hand present results showed that shade itself may influence curing effectiveness. As previously mentioned, in agreement with Ferracane (55), evaluating LCU's curing effectiveness on different materials rHV should be used as the reference value as the bottom VHN could be influenced by factors other than DC.

Generally translucent shade (T) and lower chroma dentin shade (A2d) achieved statistically significant higher rHV than higher chroma dentin shade (A4d). This is consistent with several previous studies carried out with monowave or QTH LCU investigating the influence of the shade on RBC cure (23, 61, 62). On the contrary differences between T and A2d shade rHV were not statistically significant. Particularly A2d and T shades cured with lower ED values showed better performance using the VA or SQ LCUs while increasing the ED differences between A2d and T rHVs were not statistically significant. Furthermore regardless to ED used, A4d achieved statistically higher rHVs with VA than with other LCUs. Therefore, under the condition of the present study, regardless to ED, multi-peak LED LCU showed better performances than single-peak LED devices curing both translucent and opaque TPO-containing RBC. These findings strengthen the importance of effectively activating all the initiator systems in

TPO containing materials. Second generation LED LCUs tested in the present study showed that drawbacks deriving from their narrower emission spectra may be overcome increasing ED but only when curing translucent or light shaded TPO-containing RBC. Nevertheless, curing dark shaded TPO-containing RBC, monowave LED does not achieve comparable results with polywave LED neither with the lower nor with the higher ED tested.

It has been suggested that an rHV of 0.8 can be used as a predictor for an effective and uniform cure of RBCs (31). In the present study, A2d and T groups obtained this threshold value with all LCUs tested either using a 8 J/cm² or 16 J/cm². The darkest shade (A4d) achieved an adequate polymerization (rHV>0.8) only with VA. This can be explained by the VA's wider spectral output which can more effectively activate TPO-containing RBC.

The influence of ED, LCU and shade on the polymerization of a TPO-containing RBC support the unheeded Suh's recommendation (63), renewed by Price & Felix (30), that all RBCs and bonding systems should carry a label stating both the ED and spectral bandwidth required to adequately polymerize the resin. Furthermore it would be advisable that those requirements would be specifically calibrated for any different shade.

Conclusions

Within the limits of the present study, the following conclusions were drawn:

1. Bottom surfaces VHN should be considered together with rHV during surface micro-hardness testing of different shade RBCs.
2. Curing a TPO-containing RBC multi-peak LED LCU achieved higher VHN (Vickers microhardness numbers) and rHV (bottom/top hardness ratio) than single-peak LED LCUs especially with darker shade materials.
3. Increasing the ED from 8 J/cm² to 16 J/cm², regardless to LCU or shade tested, invariably

led to better curing effectiveness.

4. If single-peak LED LCU has to be used to cure a TPO-containing RBC, clinician should increase the ED delivered to the material (i.e. increasing the exposure time), and prefer lighter or translucent materials.

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