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Effects of delivering the same radiant exposures at 730, 1450, and 2920 mW/cm² to two resin-based composites

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ABSTRACT

Objective: To evaluate the effects of curing two resin-based composites (RBC) with the same radiant exposures at 730, 1450, and 2920 mW/cm². **Materials and Methods:** Two types of RBC, Filtek Supreme Ultra and Tetric-EvoCeram-Bulk Fill, were light-cured to deliver the same radiant exposures for 5, 10, or 20 s by means of a modified Valo light emitted diode light-curing unit with the light tip placed directly over each specimen. The RBC was expressed into metal rings that were 2.0 and 4.0 mm in thickness, directly on an attenuated total reflectance Fourier transform infrared plate heated to 33°C, and the degree of conversion (DC) of the RBC was recorded. The specimens were then removed and the Knoop microhardness (KHN) was tested at both the bottom and the top of each specimen. The KHN was tested again after 24 h and 7 days of storage in the dark at 37°C and 100% humidity. The DC and KHN results were analyzed with Fisher's protected least significant difference at $\alpha = 0.05$. **Results:** The DC values for the specimens cured at the three different irradiance levels were similar. However, at different depths, there were differences in the DC values. In general, there were no clear differences among the samples cured in the three different groups, and the KHN was always greater 24 h and 7 days later ($P < 0.05$). **Conclusions:** Despite the curing time, and as long as the samples were cured with the same radiant exposures, there were no significant effects on the DC and KHN of both RBCs.

Key words: Bulk Fill, degree of conversion, Knoop microhardness, radiant exposures

INTRODUCTION

In 2013 the Minamata Agreement to ban the use of mercury was ratified and has now been signed by over 100 countries.[1] As part of this agreement, there will be a phase-down in the use of dental amalgam. In Scandinavia, resin-based composites (RBC) already predominate as the restorative material of choice.[2] Most of these RBC restorations are light-cured in the tooth, since this feature provides the dentist with a flexible working time and "on demand" curing when the light-curing unit (LCU) is activated.

Fourier transform infrared (FT-IR) spectroscopic analysis is a commonly used method to determine the degree of conversion (DC) of RBCs. The literature reports that a good correlation exists between the DC and the Knoop hardness values and its relationship with the mechanical properties of RBCs.[3,4] The DC is a measure of the percentage of carbon-carbon double bonds that have been converted to single bonds or could represent the fraction of reactive groups converted to form a polymeric resin. The DC of RBCs has a significant and important effect on the material's

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mechanical properties, volumetric shrinkage, wear resistance, and monomer elution.[5] The higher the DC reached for a RBC, the better are its mechanical properties, and this will very likely increase its clinical success.[6,7] Many factors – such as the irradiance level from LCU, exposure time, radiant exposure received, wavelength, temperature, thickness of the RBC increment, and the distance between the LCU curing tip and the RBC increment – can affect the final DC.[7-9]

Initially, the majority of LCUs used a Quartz-Tungsten Halogen (QTH) light source. These units delivered a broad spectrum of light (from 380 to 520 nm) that successfully activated the camphorquinone (CQ) and the alternative photoinitiators used in dental resins. However, the QTH light source has some drawbacks: The bulb gets very hot when in use and degrades over time, the unit requires regular maintenance, a cooling fan is required (increasing both the noise and the clinical time while the unit cools), and most units require a main power supply.[10]

The development of light emitted diode (LED) units was supposed to overcome many of the drawbacks found in QTH units. LED units are cordless and often do not contain a cooling fan. However, the initial units delivered only a lower power output and low irradiance levels at a limited range of wavelengths.[10] Contemporary LCUs can now deliver irradiance levels above 5000 mW/cm^2, and some units, such as Valo (Ultradent, South Jordan, UT, USA) and Bluephase G2 (Ivoclar Vivadent, Schaan, Liechtenstein), deliver a broader spectral emission that covers and activates newer photoinitiators.[7,11,12] These alternative photoinitiators, such as monoacrylphosphine oxide (TPO) and derivatives of dibenzoyl germanium, have peak absorbance values below 420 nm.[13]

In general, RBCs would be light-cured in 2-mm-thick increments of material, but there exists a demand to minimize clinical procedure times. Christensen has calculated that the average dentist could potentially save $28,110 per annum using a "5-s curing light" and thus increasing dental productivity. There has been a trend toward the promotion of powerful curing lights and short exposure times. Some manufacturers now claim that just 3 s of light exposure is sufficient to cure the RBC adequately.[14,15] However, a minimum amount of energy (approximately 12–24 J/cm^2) must still be delivered for adequate polymerization of the RBC.[16] This minimum radiant exposure is calculated from two parameters, the irradiance and the exposure time. The exposure reciprocity law assumes that, provided that the same radiant exposure is delivered, the DC will be the same no matter what the exposure time or irradiance level.[17,18] Thus, if manufacturers can double the irradiance value from their LCUs, the clinician can still deliver the same amount of energy to the resin in half the time.[19] The question is, will the RBC achieve the same DC?

Several studies have investigated the validity of the concept of exposure reciprocity. Halvorson *et al*., using a QTH unit, exposed different commercial RBCs to similar radiant exposures (but different irradiance values and times). They found that the "conversion profile can be equivalent if the same high radiant exposures (in their study, 11.6 J/cm^2) is applied."[20] However, low radiant exposure (0.97 J/cm^2) resulted in a lower significant difference. Later, in 2006, Schneider *et al*. tested the effects of three different LCU systems delivering the same radiant exposure (13.38 J/cm^2) to cure the RBCs. They concluded that it "produced DC values that were statistically similar." However, the different irradiance levels produced different values of DC at 24 h and 1 month later.[21]

Leprince *et al.* tested the stability and validity of exposure reciprocity using different experimental resins with different photoinitiators. They used three curing modes to deliver 18 J/cm^2 using a QTH unit. They reported that the law was photoinitiator-dependent.[22]

In a room-temperature measurement (23°C), Hadis *et al.* also tested the effects of fillers using different commercial resins. They used 5 different irradiance levels to deliver a radiant exposure of 18 J/cm^2. The irradiance levels ranged from 400 to 3000 mW/cm^2 for a curing time between 6 and 45 s. In general, they found no significant difference in DC values calculated at their different irradiance levels.[19]

In a recent study, Palin *et al.* tested two different experimental resins that contained different photoinitiators. They delivered 18 J/cm^2 using three irradiance levels from one QTH unit. They found that their samples that used CQ had a higher DC when the exposure time was increased and the irradiance was decreased. They also reported that samples that used the monoacylphosphine oxide photoinitiator had high DC at low exposure times. They also concluded that exposure reciprocity was photoinitiator-dependent.[23]

The use of RBC in 2.0-mm increments is considered the norm in the delivery of a clinically accepted polymerized RBC restoration. However, bulk-fill

RBCs claim that they can be successfully polymerized in 4-mm-thick increments.

Many studies have tested the microhardness of different RBCs light-cured under different exposure conditions. Ilie and Stark found that, although hardness was higher when the RBC was light-cured with the high-power mode on the upper surface, the standard mode produced better results at the 4- and 6-mm depths.[24] Tarle *et al*. found that curing for a longer exposure time (30 s) achieved acceptable hardening in some of their tested RBCs in 4-mm increments.[25]

The aim of this study was to evaluate the effects of different irradiance values and different exposure times on the DC and Knoop microhardness (KHN) of two types of RBCs.

When the RBC samples were cured at different irradiance levels and times, and as long as they received the same radiant exposure, the null hypotheses were:

- There would be no difference in DC and KHN values
- There would be no difference in DC and KHN between thicknesses of different increments
- There would be no difference in the percentages of increase between immediate and postcuring readings (24 h and 7 days) for KHN values in the RBCs tested.

MATERIALS AND METHODS

This study evaluated two RBC materials [Table 1]:

1. Bulk-fill RBC, Tetric EvoCeram Bulk Fill (TBF), shade IVA (Ivoclar-Vivadent, Amherst, NY, USA)
2. Conventional nanocomposite RBC, Filtek Supreme Ultra (FS), shade A2B (3M ESPE, St. Paul, MN, USA).

The manufacturer of TBF claims that this bulk-fill RBC can be cured in up to 4-mm increments, whereas the manufacturer of FS recommends only 2 mm of thickness. This investigation tested TBF at 2- and 4-mm thicknesses and at a 2-mm thickness for the FS. All specimens were light-cured by means of a high-power LED, Valo (Ultradent, UT, USA), in three curing powers [Figure 1 and Table 2].

Light measurements

The spectral radiant power from the Valo LCU was measured with the use of a 6-inch integrating sphere (Labsphere, North Sutton, NH, USA) connected to a fiber-optic spectrometer (USB 4000, Ocean Optics, Dunedin, FL, USA). Since all specimens had a diameter of 4 mm, the output was also measured through a 4-mm-diameter aperture placed over the entrance to the integrating sphere, to ensure accurate measurements of the spectral radiant power and irradiance received by the specimens and not the total power output emitted from the LCU. Spectrasuite v2.0.162 software (Ocean Optics Dunedin, FL, USA) was used to collect and analyze the data. All curing modes were performed with the same curing unit, to standardize the spectral emission for all tested samples.

Degree of conversion and Knoop microhardness measurements

FS Ultra RBC was expressed and packed into a 2.0-mm-thick metal ring, whereas the TBF was packed into 2- and 4.0-mm-thick metal rings, with 4.0-mm internal diameter for the rings involved. All specimens were centered on the diamond element of a horizontal attenuated total reflectance attachment (Golden

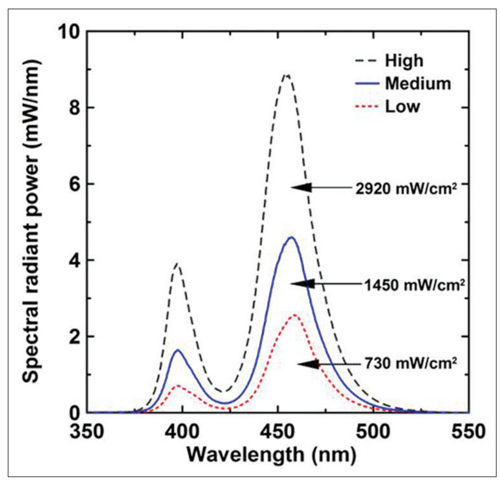

Figure 1: Spectral emission from the Valo measured through a 4-mm aperture into an integrating sphere, showing two distinct emission peaks at low-(730 mW/cm^2), medium-(1450 mW/cm^2), and high-power (2920 mW/cm^2) curing modes, all delivering ~14.6 J/cm^2

Table 1: The resin-based composite resins and light-curing unit used in the study

Material (shade)	Abbreviation	Type	Manufacturer	Lot number
Filtek supreme ultra (2AB)	FS	Conventional	3M ESPE, St. Paul, MN, USA	N553090
Tetric EvoCeram Bulk Fill (IVA)	TBF 2 or 4 mm	Bulk-fill	Ivoclar Vivadent, Amherst, NY, USA	R08233
Valo		LED	Ultradent, South Lake, UT, USA	V37330

LED: Light emitted diode

Gate, SPECAC, Inc., Smyrna, GA, USA) on a FT-IR spectrophotometer (Tensor 27, Bruker Optics Inc., Billerica, MA, USA) at 33°C. The surfaces of the uncured samples were covered by a Mylar strip to avoid immediate contact between the RBCs and the curing tip. Each sample was cured to the assigned curing mode for 5, 10, or 20 s. DC was collected in real time for 120 s. The sample was then removed and placed on an automated Knoop hardness-testing device (Mitutoyo Canada Inc., Mississauga, ON, Canada), with a 50-g load for 10 s. After the hardness measurements were made (at nine mapping points on the sample surface), first at the bottom and then at the top surface, the specimens were stored in distilled water in complete darkness at 37°C and 100% humidity. Both DC and Knoop hardness were retested at 24 h and 7 days, respectively. In total, 10 samples were fabricated for each tested group. The effects of the different curing powers and times that delivered the same radiant exposure, across two types of RBCs, on the DC and KHN values and the effects of 24-h and 7-day storage on KHN were compared by ANOVA and Fisher's protected least significant difference ($P < 0.05$).

RESULTS

The DC values for the specimens cured at the three radiant levels were similar [Figure 2]. However, at different depths, there were differences in the DC values.

Table 2: Different irradiance levels delivering similar radiant exposures

Curing power	Irradiance value (mW/cm²)	Exposure time (s)	Radiant exposure (J/cm²)
High	2920	5	14.6
Medium	1450	10	14.5
Low	730	20	14.6

In general, there were no clear differences among samples cured at the three different irradiance levels. Table 3 shows the KHN values for all samples in immediate and delayed (24 h and 7 days) stages for the bottom surfaces. For TBF 2, there were statistically significant differences among all groups in the samples collected immediately ($P < 0.05$). However, for samples collected at 24 h, statistically significant differences occurred only between the L and H irradiance levels, and there were no statistically significant differences among samples collected at 7 days. There were no statistically significant differences for all groups at different irradiance levels and collected times in the TBF 4 material. For the FS, there were statistically significant differences at immediate and at 7 days between samples cured at M and H irradiance levels and samples cured at L and H irradiance levels and collected at 24 h ($P < 0.05$). As for the top-surface values of tested samples [Table 4], there were no statistically significant differences for all groups, except between M- and H-cured samples in the FS material group at 7 days' collecting time ($P < 0.05$).

When the depth of cure was evaluated, all FS samples (at all irradiance levels) had a higher percentage of

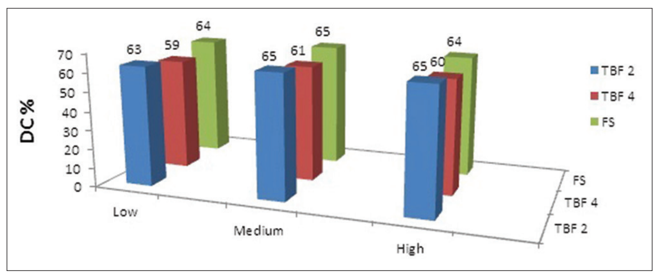

Figure 2: Degree of conversion of resin-based composites at low- (730 mW/cm²), medium-(1450 mW/cm²), and high-power (2920 mW/cm²) curing modes, all delivering ~14.6 J/cm²

Table 3: Means±standard deviations (bottom surface) for immediate and delayed (24 h and 7 days) Knoop microhardness for Tetric EvoCeram Bulk Fill (2 and 4 mm thick) and Filtek Supreme Ultra, 2 mm thick at 730, 1450, and 2920 mW/cm²

RBC type and thickness (mm)	Irradiance (mW/cm²)	Mean±SD		
		Immediate KHN	24-h KHN	7-day KHN
TBF (2)	730	17.9±0.39	20.3±1.03	20.6±0.39
	1450	17.4±0.95	19.5±0.74	19.7±1.0
	2920	17.7±1.57	19.3±1.0	20.1±0.81
TBF (4)	730	13.4±1.5	14.6±3.2	14.8±1.58
	1450	13.5±0.94	15.0±0.72	15.4±1.29
	2920	13.0±0.89	13.7±1.4	14.8±1.36
FS (2)	730	28.5±0.75	30.4±2.6	31.8±1.28
	1450	28.8±1.36	30.9±1.1	32.0±1.0
	2920	27.9±1.53	29.1±1.12	29.7±1.2

KHN: Knoop microhardness, TBF: Tetric EvoCeram Bulk Fill, FS: Filtek Supreme, RBC: Resin-based composites, SD: Standard deviation cure in reference to that of other specimens. At a low irradiance level, TBF 2 had 74.3%, FS had 81.0%, and TBF 4 showed only 53.2% depth of cure. For the medium irradiance level, samples for TBF 2, FS, and TBF 4 had 72.7, 79.6, and 51.3% depth of cure, respectively. When curing at a high irradiance level, we found the highest percentage of depth of cure for the FS sample with 82.8%, whereas the lowest percentage of depth of cure was recorded for the TBF 4 sample, with only 51.4%.

In reference to the effect of time on investigated samples, all had an increased KHN after 24 h and 7 days, compared with their immediate values, especially on the bottom surfaces [Table 3]. The highest percentage of increase was found in TBF 2 samples when cured at low irradiance levels (13.4% at 24 h and 15.1% at 7 days). The lowest percentage of increase was found when the FS was cured at the high irradiance level, ranging between 4.3% (24 h) and 6.5% (7 days).

DISCUSSION

Previous studies have reported mixed findings regarding the influence on the polymerization of dental restorative composites when cured in the same radiant exposure but with different irradiance levels.[7,17-19,21-24,26-31] This study showed that curing with different irradiance levels that delivered the same radiant exposure (14.6 J/cm^2) had no effect on the DC and KHN values of both conventional and bulk-fill RBCs. Thus, the first hypothesis was accepted. There was a significant difference between the results from the 2- and 4-mm-thick specimens of RBCs. Thus, the second hypothesis was rejected. This effect could not be seen in the immediate, 24-h, and 7-day samples; thus, the third hypothesis was accepted.

One of the major reasons for the use of an LED LCU in this study was its power stability, which guarantees constant power during all curing durations. This is not the case with QTH or photo-activated composite (PAC) LCUs. Figure 3 shows that even with modifications, the Valo LED unit continued to deliver stable and constant power at all testing parameters compared with the temporal instability of QTH and PAC units, whose output was unstable throughout exposure times over 20 s.

It has been stated that curing an RBC at high irradiance could reduce its DC.[32] This has been claimed to be due to a more rapid initiation rate at high irradiance, which produces more radical concentrations, leading to the premature termination of polymerization. In contrast, curing with low irradiance levels will delay the termination of polymerization and lead to a lower quantity of trapped monomer and hence to a higher DC.[32] In this study, highly viscous RBCs were investigated. With either conventional or bulk-fill RBCs, the findings showed that the law of reciprocity stands. This is in agreement with the results of other studies reported in the literature.[21,33-36] Schneider *et al*. found no statistically significant differences in the DC among tested RBCs when cured in QTH, LED, and PAC LCUs. Analysis of their raw data confirms our results. Furthermore, Gonzalez *et al*.

Table 4: Means±standard deviations (top surface) for immediate and delayed (24 h and 7 days) Knoop microhardness for Tetric EvoCeram Bulk Fill (2 and 4 mm thick) and Filtek Supreme Ultra, 2 mm thick at 730, 1450, and 2920 mW/cm^2

RBC type and thickness (mm)	Irradiance (mW/cm^2)	Mean±SD		
		Immediate KHN	24-h KHN	7-day KHN
TBF (2)	730	24.12±0.31	25.8±0.27	27.5±1.8
	1450	23.94±0.77	26.8±0.59	26.4±0.55
	2920	25.44±0.72	27.5±1.0	27.7±1.0
TBF (4)	730	25.2±0.72	27.1±1.5	27.1±0.70
	1450	26.3±1.42	28.4±1.2	27.8±1.75
	2920	25.3±1.57	26.9±0.72	27.1±0.50
FS (2)	730	35.2±0.89	37.4±0.68	38.9±1.5
	1450	36.2±1.66	38.2±1.0	38.4±0.65
	2920	33.7±0.47	36.4±1.1	36.8±1.2

KHN: Knoop microhardness, TBF: Tetric EvoCeram Bulk Fill, FS: Filtek Supreme, RBC: Resin-based composites, SD: Standard deviation

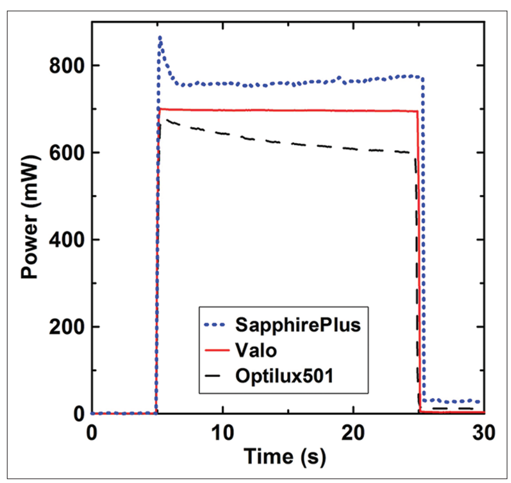

Figure 3: Power (mW) delivered over the 20-s exposure time, measured with a 4-mm-diameter aperture at the entrance to the integrating sphere. Note the stable radiant power output from the first to the final exposure (over the 20 s) for the Valo (red), compared with unstable outputs for both SapphirePlus (blue-dot) and Optilux 501 (black) light-curing units

reached the same results when curing their RBCs with an LED delivering 400, 700, and 950 mW/cm^2 at different times to deliver 24 J/cm^2. Conversely, some reports in the literature are not in agreement with our findings.[8,18,19,22,26,37,38] Leprince *et al*. concluded that the law is photoinitiator-dependent. Although this assertion stands in CQ RBCs, it does not with TPO photoinitiator RBCs.[22] Their findings were also confirmed by Palin *et al*.[23] Hadis *et al*. found the law to be viscous-dependent when testing commercial RBCs.[19]

When bulk-fill RBCs were introduced to the market, they were claimed to address certain requirements, including ease of clinical use, by increasing depth of cure without affecting mechanical properties.[39,40] The manufacturers of the bulk-fill RBCs continued to develop their product and increased the depth of cure by applying more efficient initiator systems and high translucency of fillers and composite contents.[41,42] Although analysis of some data in the literature supports equivalent or even improved values between conventional and bulk-fill materials in creep resistance or polymerization shrinkage, other factors show bulk-fill RBCs to be inferior to conventional RBCs.[39] The current study shows an effect of increment thickness (2 and 4 mm). Despite the curing protocol, when cured with radiant exposure of 14.6 J, 2-mm-thick samples showed significantly higher values in both DC and KHN when compared with 4-mm-thick samples. A major reason for that is related to less light restriction (attenuation) with such depth of the tested RBCs.

A possible reason for the higher KHN values for FS could be due to different filler particle size, resin matrix composition, and different photoinitiators and their ratios.[39] A recent study confirmed that spectral radiant power under 425 nm is ineffective at a depth of 4 mm, leading to potentially more polymerization complications of RBCs activated by photoinitiators at that spectral range.[43]

This study evaluated the possible effect of the exposure reciprocity law on hardness when tested immediately, at 24 h, and at 7 days. As an RBC sample is exposed to light polymerization, monomer conversion is initiated and continues in the so-called postpolymerization phase. This occurs because unreacted free radicals generated during composite polymerization are trapped within cured monomer and then continue to generate crosslinks, leading to a higher DC in cured samples, especially over the first 24 h.[21,44,45] Our findings showed a significant increase in KHN in the 24-h-tested samples compared with those tested immediately. However, there was no significant increase after a storage time of 7 days. This could be related to maximum conversion reached by the cured resin samples, leaving very few radicals to interact. In this study, we stored our tested samples for 24 h and 7 days in a humid environment (to simulate clinical conditions, since RBCs will be in the oral cavity environment). Theoretically, polymers tend to absorb water and dissolve, causing them to swell, leading to their expansion and increased molecular mobility. This results in fewer free radicals and a possible continuous rates of conversion post the photocuring phase.[46-48] When our storage conditions were compared with others reported in the literature, there were similar stable hardness values after 24 h or, sometimes, even longer storage periods (7 days).[44] Stability in hardness values was similar between 24 h and longer periods (7 and 30 days) when samples were stored in dry conditions.[21,46]

CONCLUSIONS

Within the limitations of this study, we conclude the following:

- Despite differences in the exposure times, provided that the RBCs received the same radiant exposures, irradiance differences between 730 and 2920 mW/cm^2 had no significant effect on the DC and KHN of both RBCs tested. However, this may not necessarily to be valid for other properties of the tested materials
- Provided that 14.6 J/cm^2 of energy is delivered, light-curing RBC restorations in short durations (i.e., as short as 5 s) can be successful
- Physical properties of the RBC should be tested both immediately and at least 24 h after light-curing because the properties continue to improve with time (up to a 15.1% increase).

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Conflicts of interest
There are no conflicts of interest.

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