REVIEW

Bulk Fill Resin Composite Materials Cured with Single-Peak versus Dual-Peak LED LCUs

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Introduction: Manufactures claim that recently introduced bulk fill materials (BFM) can be cured adequately in 4 mm increments. This requires adequate light energy to be transmitted through the material to ensure adequate polymerization at the bottom of the increment.
Aim: To compare the total light energy transmission through three BFMs and bottom/top (B/T) surface Vickers hardness (VH) when cured with single-peak versus dual-peak LED LCUs.
Methods and Materials: Samples (n=5) of two viscous BFMs, Tetric EvoCeram® Bulk Fill X-tra fil® [XF] flowable SureFil, were prepared. A conventional RBC, Tetric EvoCeram® was used as a control. Using MARC® RC, the irradiance delivered to top surface of samples was adjusted to 1200 mW/cm^2. Samples were cured with singlepeak EliparTM S10 or dual-peakBluephase® G2 for 10 seconds and irradiance transmitted to the bottom surface measured. Samples were stored for 24 hours, prior to VH measurements B/T VH ratios were calculated. Statistically analysed used oneway ANOVA (α=0.05).
Results: There was no statistically significant difference for B/T total energy transmission between materials except XF with EliparTM S10 (P<0.001). Total energy transmission ranged from 0.7 J/cm2 to 1.5 J/cm2. There was no statistically significant difference for B/T VH ratios between materials (P>0.05) when materials were cured with single-peak versus dual-peak LCU's, XF>SDR>TEC>TBF. TBF alone, did not reach the generally accepted B/T VH of 80%.
Conclusions: Both single-peak and dual-peak LCU's were equally effective for curing the studied bulk fill materials. Manufacture's recommended total energy delivered to the top surface may not always be sufficient for effective curing.

Keywords: Bulk-Fill Composite, Single-peak LED, Dual-peak LED

Introduction

Resin-Based Composite (RBC) is widely used in Dentistry to restore anterior and posterior teeth. There have been many advances in RBCs development to improve their clinical success rate and overcome the many disadvantages of the first materials [1]. These materials cure with visible light, and the light curing units have also undergone development to match the need of composite materials [2]. Recently, bulk fill composite materials were introduced to be placed in the cavity in bulk, claiming replacement of the traditional layering technique.

Resin-Based Composites became available to Dentistry in the 1960's following the development of the first methacrylate-based polymer system by Bowen in 1962 [3].

The early RBCs were, in the main, used in the anterior region, where the colour of amalgam was not desired. At that time, all materials were chemically cured. With the advent of photo-polymerization, Ultra-violet (UV) light curing systems superseded these and, in the late 1970's, the first report about a dental filling material that was cured with blue light in the visible range was published [4]. Effective dentine bonding agents became available at the beginning of the 1990's, and since then, composites have found increasingly broad use as a universal restora-

tive material [5]. Furthermore, there has been a growing demand for aesthetic restorations, not only in the anterior region, but increasingly also in posterior teeth. This, in turn, has resulted in the quest for composite materials that offer improved physical, aesthetic, and handling properties. The advent of bleaching has also resulted in the need for whiter shades of RBCs [6].

The total energy (J/cm^2) is the result of irradiance intensity (mW/cm^2) and exposure time (second). There is almost full agreement in the literature about the total energy concept which states that the result of multiplying intensity by curing time should always result in the same total energy, thus increasing intensity and reducing time, or decreasing intensity and increasing time can result in the same total energy delivered to the restoration surface. However, there is some question about the validity of reciprocity for combinations of irradiance and time particularly high irradiance and short time [7-9].

The total energy required to achieve sufficient polymerization is widely controversial in the literature and recent findings of literature are summarised in Table I.

When the cavity is large, incremental placement of 2 mm thickness composite was recommended to reduce the polymerization shrinkage stress [10] and to avoid the reduction in light transmission through the material to the bottom surface [11,12]. This was used as the gold standard [13-15].

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Table I. Recommended total energy required for sufficient curing in the literature

Author	Total energy recommended	Thickness of material
Lee and Greene, 1994	>12 J/cm²	2 mm
Rueggeberg et al. 1994	21-24 J/cm²	2 mm
Sobrinho et al. 2000	21-24 J/cm²	2 mm
Yap and Seneviratne, 2001	>12 J/cm²	2 mm
Yoon et al., 2002	8-16 J/cm² 16 J/cm²	2 mm > 3 mm
Fan et al. 2002	6-12 J/cm² up to 18 J/cm²	1.5 mm
Emami and Soderholm, 2003	> 5-15 J/cm² > 30 J/cm²	2 mm 4-6 mm
Da Silva et al., 2008	> 17 J/cm²	2 mm
Calheiros et al,. 2008	> 24 J/cm²	2 mm
Howard et al. 2010	> 8 J/cm²	2 mm

However, curing a RBC incrementally carries the risk that voids and/or contamination may be incorporated between the layers. Bond failure between increments has also been reported and the technique can be time consuming, thus increasing the potential for further contamination [16].

More recently, in an attempt to overcome some of the disadvantages of 2 mm incremental packing, bulk fill materials (BFMs) have been introduced to the market claiming that they would allow a 4 mm bulk placement in one layer while allowing sufficient polymerization.

Two types of BFMs are available in the market, viscous and flowable. The flowable BFMs have been recommended for use in low load bearing areas and was aimed to place in one layer of 4 mm thickness to reduce polymerization stress being mandatory covered by a 2 mm layer of conventional RBC, [17,18] because the modulus elasticity and hardness were reduced [19]. The manufacturers of these materials stated some changes in composition in order to achieve the adequacy of bulk curing. It seems that manufacturers have followed different strategies to achieve this.

Ivoclar Vivadent introduced Tetric EvoCeram® Bulk Fill as a viscous composite produced mainly for use in posterior teeth. They claimed two reasons why 4 mm increments could to be cured sufficiently. Firstly, by using two types of fillers with different mean particle sizes in order to match the refractive index of fillers to that of the monomer matrix for the ease of light transmission. Secondly, by using a patented photo-initiator (Ivocerin®), in addition to the standard systems, as a booster to make the polymerization faster and reliable in 10 seconds of curing by a dual-peak LED LCU. In addition, they claimed the use of special shrinkage stress relievers to reduce the polymerization shrinkage. Some manufacturers reduced the filler volume and increased the filler size, such as in X-tra base, X-tra fil® and SureFil SDR™. Consequently, the specific surface between fillers and organic matrix was lowered, thus reducing light scattering.

One example of the flowable materials is Smart dentin replacement/ Shrinkage decreased resin (SDR) or SDR™ technology with modification to monomer (high weight modulator embedded in the centre of monomer) to be less shrinkage.

Because of the decrease in the intensity of the light transmitting through the material the main concern with these BFMs is to ensure sufficient polymerization at the deeper portions and the bottom by receiving enough light energy. Also, concerns regarding increased stress and deformation as a result of curing in large increments.

The potential disadvantages of BFMs can include possibility of more voids as placed in the mass of material, difficulty of making adequate contact areas, more stress as the entire mass polymerize at one time, and inadequate curing in the deepest part of the material [20].

In the literature, there are few studies evaluating some of the properties of the BFMs such as polymerization shrinkage, hardness, microleakage, marginal integrity, and creep deformation [21,22].

A recent in vitro study by [23] used two BFMs, Tetric EvoCeram® Bulk Fill and X-tra base, to investigate the effect of curing time and light tip distance using a single peak LCU, and measured the light transmission by MARC® and degree of conversion (DC) using FTIR. They concluded the validity of curing BFMs in 4 mm thick increments.

In an in vitro study, [24] X-tra fil® (VOCO) was investigated when placed in bulk and incrementally and cured with two LED LCUs. They found no difference in Vickers Hardness between the two techniques. However, in their study, it was apparent the use of high total energy delivered to the top surface ranged from 24 J/cm² to 52 J/cm² in addition to the limited thickness of samples to 3.5 mm.

In an in vitro study, [25] the mechanical properties, VH and DC of SureFil SDR™ and another flowable composites, were compared when cured for 10, 20 and 40 seconds respectively. Variation between the materials with different curing parameters were shown to be statistically different. However, the authors recommended that 4 mm bulk specimens be cured for 20 seconds when total energy of 24 J/cm² was delivered to the top surface, which was higher than the manufacturers' recommendations.

Another study [26] used five BFMs including Tetric EvoCeram® Bulk Fill and X-tra base. Elipar™ S10 (1200 mW/cm²x20 sec) delivering 24 J/cm² was used and this is higher than what recommended by the manufacturers. In agreement with the manufacturer claims, it was concluded that the materials could be cured to an acceptable depth with increments ranging from 4 to 5 mm.

Another recent study by [27] investigated the light transmission and micromechanical properties of seven BFMs (three viscous and four flowable) using MARC® compared to seven conventional composites (five viscous and two flowable). It was found that most of the BFMs tested were more translucent and the flowable BFMs showed the lowest mechanical properties including VH.

Alshali et al. [28] used eight materials, four flowable bulk and four flowable conventional, including SDR and

X-tra base and investigating the DC using FTIR after curing directly or after 24 hours. Although their BFMs' results were comparable to the conventional materials, there was variation between materials. A major criticism is that a QTH LCU was used which delivered only 12 J/cm² which was less than the manufacturer' recommendation for some of the materials tested.

It was apparent that most of these studies were not following manufacturers recommendations regarding the least recommended total energy required. In addition, a variation in protocol, LCUs and techniques were used.

Many factors affect the adequate polymerization of RBCs. Some of these factors are related to the materials themselves, for example, light transmission, material composition, shade, opacity and material thickness. Other factors depend on the LCU type, irradiance, curing time and the tip-to-material surface distance.

The new BFMs were introduced recently to overcome the disadvantages of the traditional 2 mm layering technique saving clinical time when restoring larger cavity preparations and thus reducing the potential for contamination.

There is little evidence indicating that sufficient polymerization takes place in the new BFMs when used in 4 mm bulk placement in one layer. Also, previous workers had not investigated the effect of different types of LCUs on the polymerization and did not match the delivered top total energy to the recommended total energy required by the manufacturers.

Manufactures claim that recently introduced bulk fill materials can be cured adequately in 4 mm increments. This requires adequate light energy to be transmitted through the material to ensure adequate polymerization at the bottom of the increment.

The aim of the present study is to compare the total light energy transmission through three BFMs and bottom/top (B/T) surface Vickers hardness (VH) when cured with single-peak versus dual-peak LED LCUs.

Null Hypotheses

1. There was no difference in the light energy transmitted through BFMs when cured with a single peak or a dual peak LED LCU.
2. There was no difference in the bottom to top surface microhardness ratio of BFMs when cured with a single peak or a dual peak LED LCU.

Methods & Materials

The materials used in the study are given in Table II.

Samples (n=5) of two viscous BFMs, Tetric EvoCeram® Bulk Fill [TBF], (Ivoclar Vivadent), X-tra fil® [XF], (Voco), and one flowable BFM, SureFil SDR™ [SDR], (Dentsply), were prepared in 4 mm deep Delrin® rings (DuPont, Mississauga, Ontario, Canada). A conventional RBC, Tetric EvoCeram® [TEC], (Ivoclar Vivadent) was used as a control. Using MARC® RC, the irradiance delivered to the top surface of the samples was adjusted to 1200 mW/cm². Samples were then cured with single-peak Elipar™ S10 (3M ESPE) or dual-peak; Bluephase® G2 (Ivoclar Vivadent) LED LCU's for 10 seconds and the irradiance transmitted to the bottom surface measured. Additionally, MARC® internal soft-ware calculated the total energy in all cases. Samples were stored post-irradiation in a dry, sealed, light-proof container at room temperature (21+/-1⁰C) for 24 hours, prior to taking VH measurements (X3) from the top and bottom surfaces of each sample using a micro-hardness machine (HM-200 Series, Mitutoyo Corporation, Tokyo, Japan) with an applied 200g load for 10 seconds. B/T VH ratios were calculated. Data were statistically analysed using one-way ANOVA (α=0.05).

The following LCUs were used.

1. Single Peak LCU: Elipar™ S10, 3M ESPE, Spectral output (wavelength) 430-480 nm, Light Intensity 1200 mW -10%/+20%, Tip 10 mm
2. Dual Peak LCU: Bluephase® G2, Ivoclar Vivadent AG, Schaan, Liechtenstein, Spectral output (wavelength) 380-515 nm Second peak at approx. 410 nm, Light Intensity 1200 mW/cm²+/-10% (high power mode), Tip 10 mm

Table II. RBCs used in this study

Material	Organic Matrix	Photoinitiators	Filler
Viscous Bulk-fill materials			
Tetric EvoCeram® BulkFill	Dimethacrylate	CQ TPO Patented light activator; Ivocerin®	Barium aluminium silicate glass Ytterbium trifluoride (YbF3) Mixed oxide and Prepolymers. 80% by weight 60% by volume
X-tra fil®	Bis-GMA UDMA TEGDMA	Not specified	Inorganic fillers (not specified) 86% by weight 70% by volume
Flowable Bulk-fill materials			
SureFil SDRTM	Modified UDMA, EBPADMA TEGDMA	CQ	Ba-Al-F-B silicate glass, Sr-Al- F silicate glass 68% by weight 44% by volume
Viscous Conventional materials			
Tetric EvoCeram®	Bis-GMA UDMA Bis-EMA	Not specified	Barium glass Ytterbium trifluoride (YbF3), Mixed oxide and Prepolymers. 82.5% by weight

Measuring Instruments

The MARC®-Resin Calibrator (MARC®-RC), (BlueLight Analytics Inc., Halifax, Nova Scotia, Canada) was used to measure light energy data (Figure 1).

Micro Vickers Hardness Testing Machine (HM-200 Series, Mitutoyo Corporation, Tokyo, Japan)   used to record Vickers hardness data from the top and bottom surfaces of the cured specimens (Figure 2).

The temperature and humidity of the room were recorded every hour using the Diplex® thermometer. (Diplex® thermometer, London, UK)

Calibration and Standardisation of the LCU Intensity

Care was taken to be away from the direct light of the ceiling lights and the laptop screen to reduce any possible external effect on the MARC®-RC sensors throughout the experimental phase.

Prior to LCU calibration, a clear Mylar strip was placed over the top sensor and held in place with clear adhesive tape (Sellotape®), placed at both ends of the strip and away from the top sensor.

The Elipar™ S10 LCU was powered cordless with full charge, while the Bluephase® G2 was powered directly from the mains supply.

The Elipar™ S10 was mounted on the MARC®- RC fixing arm, fixed and centred perpendicularly over the top sensor. The Elipar™ S10 was activated, and moved up and down using the vertical adjustment, to reach irradiance (intensity) of 1200 mW/cm². The irradiance was shown in real-time on the laptop screen. The Elipar™ S10 calibration was reconfirmed by irradiating the top sensor of MARC®-RC and then positioned in the same horizontal plane by horizontal adjustment over the bottom sensor before sample preparation.

After curing all samples of the Elipar™ S10, the same procedure of calibration was repeated for the Bluephase® G2.

Sample Preparation and Measurement of Bottom Light Energy and Light Transmission

For all combinations of BFM (3), LCU (2) and total top energy of 12 J/cm², five 4 mm thick samples were prepared. This gave six groups with a total of thirty samples.

In order to compare the BFMs used in this study to the conventional composite Tetric EvoCeram®, as a control, a conventional composite from one of the manufacturers (Ivoclar Vivadent) of the BFMs (Tetric EvoCeram® Bulk Fill) was used. It was placed in 2 mm thick increment, and the rest of the methodology was the same using the combination of TetricEvoCeram® (1), LCU (2) and total top energy of 12 J/cm². This gave extra 2 control groups with a total of ten samples. This gave a total of 8 groups with a total of 40 samples.

The Delrin® ring was placed on a clear Mylar strip on the glass slab. In the case of the flowable BFMs, the material was injected directly in the ring placed over the bottom sensor of MARC®-RC with Mylar strip in place.
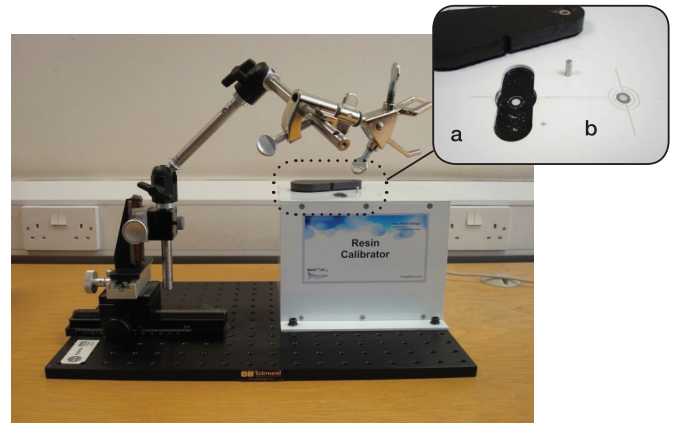

Fig. 1. MARC®-RC and arrangement of the sensors (inset) (a) bottom surface sensor, and (b) top surface sensor
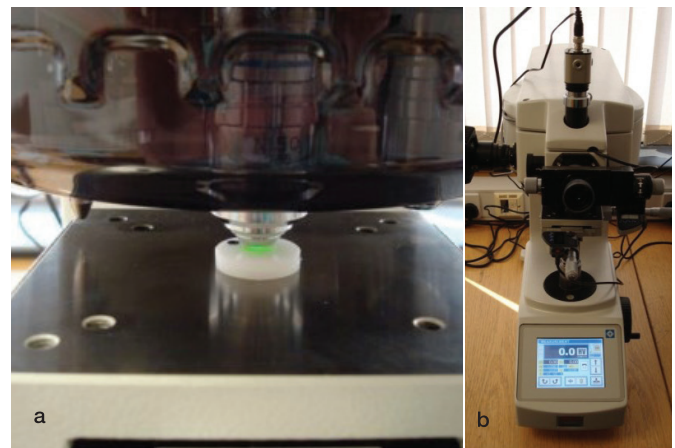

Fig. 2. Micro Vickers Hardness Testing Machine: (a) The VH tester; (b) Microscope over a sample on testing platform

The samples were prepared by placing a single increment of X-tra fil® viscous BFM in the lumen of the 4 mm thick Delrin® ring, using a clean and dry flat plastic instrument. The material was packed, flattened and excess material removed with the flat plastic instrument. Each sample was covered by another clear Mylar strip, and pressed by manual finger load to extrude excess material and create a flat surface. Once prepared, the Delrin® ring with the sample of RBC was placed over the bottom sensor.

The Elipar™ S10 was activated for 10 sec and the total light energy reaching the bottom surface of the sample, was recorded by the sensor. The data were shown on the laptop screen and saved on the computer related to the bottom surface. In this way, a known total light energy of 12 J/cm² was delivered to the top surface of the sample (1200 mW/cm²x10 sec). This was repeated for four further samples, giving five samples in total. Then, this was repeated for all combination of experimental groups.

To differentiate between the top and bottom surfaces, for later VH measurement, a black permanent mark was placed on the top surface of the ring using a permanent ink marker.

The light energy delivered to the bottom surface of each sample was shown on the laptop software of MARC®- RC. The light transmission (%) was calculated as follows:

Light transmission (%)=(Bottom light energy/ Top light energy)x100

The Mylar strips were removed and the cured samples were placed in their individual groups in clearly labelled light-proof paper envelopes, and the opening of the envelope stapled. All samples were stored in the envelopes in a dark at a room temperature of $(21+/-1^0C)$ for 24 hours.

Measurement of Vickers Microhardness

The standard to reflect the adequate curing of RBCs using bottom to top surface VH ratio (%) was set at 80% according to the literature [29].

After 24 hours storage post-irradiation, the VH was measured on both top and bottom surfaces of each sample using the Vickers Hardness tester (Figure 3). For each sample, the sample was placed on the tester platform and viewed under X50 magnification microscope (Figure 4). The magnified view was focused and the platform was moved in the horizontal plane to select a suitable area to make the indentation. The criteria for the selection of this were the smoothest surface possible, without microscopic voids, and close to the centre of the sample. Once located, the indentation button was pressed and the VH tester made the indentation with a previously configured 200g load applied for ten seconds.

When the indenter unloaded, the horizontal and vertical dimensions (D1 and D2) of the indentation were measured under microscopic vision, using the built-in measuring planes. This automatically gave the VH values directly on the LED screen as the tester automatically measures the depth of the indentation (D3). This method was repeated to take a total of three measurements close to the centre of the sample from the top surface and then repeated for the bottom surface three times.

For each sample, the data for top and bottom VH were entered into a Microsoft® Excel® spreadsheet (Microsoft® Office 2010, Microsoft® Corp., Redmond, WA,

USA), and the bottom to top VH ratio (%) was calculated using the following formula:

VH ratio (%)=mean bottom VH value /mean top VH value x 100

Using MARC® RC, the irradiance delivered to the top surface of the samples was adjusted to 1200 mW/cm² and delivered for 10 seconds (12 J/cm²). The irradiance (mW/cm²) transmitted to the bottom surface was measured with MARC® and the internal software converted this to J/cm². Three measurements were taken from both top and bottom surfaces of each sample using a Vickers Hardness Testing Machine (HM-200 Series, Mitutoyo Corporation, Tokyo, Japan) with 200g load applied for 10 seconds. Bottom to top (B/T) VH ratios were calculated.

Statistical Analyses

Data were statistically analysed using one-way ANOVA (α=0.05).

The mean values of bottom light energy, light transmission (%), and VH ratio (%) in addition to the standard deviations (SD) for each group were calculated using Minitab® 16 (Minitab Inc., State College, PA, USA). The dependent factors were bottom light energy, light transmission (%), and VH ratio (%). The independent factors were materials (4 levels), LCUs (2 levels) and total top light energy (1 level). The true power of the study was calculated using the same software. For each dependent factor, the greatest standard deviation of the means between groups was chosen to avoid over-estimating the power.

All statistical comparisons were conducted at the 95% significant level of confidence intervals (CIs), and alpha value of 0.05. If a confidence interval did not contain zero, there was a statistically significant difference between the corresponding means.

Results

The results are presented in Table III. There was no statistical significant difference for B/T total energy transmission between materials except XF with Elipar™ S10 (P<0.001).
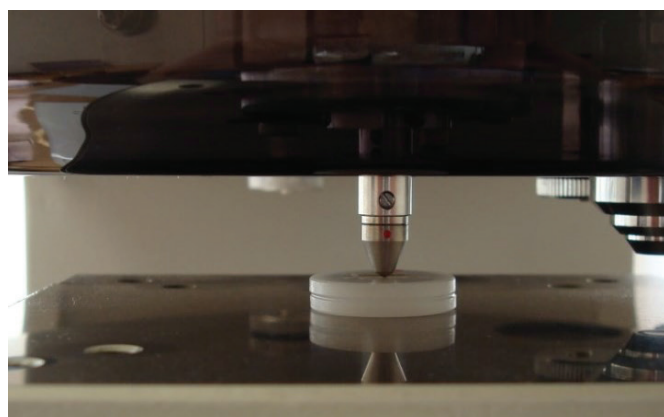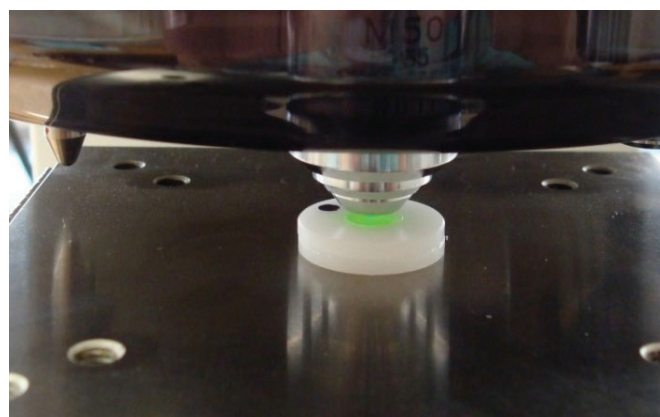

Fig. 3. VH indentation

Fig. 4. VH magnification X50

The room temperature and humidity average readings were 21.0+/-1.0º C and 57.0%+/-5.0% respectively throughout the duration of the experimental phase of the study.

Total energy transmission ranged from 0.7 J/cm² (6.2%) to 1.5 J/cm² (12.5%). Between materials, TEC>SDR>XF>TBF. The mean of the total energy delivered to the bottom surfaces of all groups is presented in Table III and gives a broad view of the performance of each material.

There was no statistically significant difference for B/T VH ratios (range, 63% to 90%) between materials (P>0.05) when materials were cured with single-peak versus dual-peak LCU's. Between materials, XF>SDR>TEC>TBF. TBF alone, did not reach the generally accepted B/T VH of 80%.

The emission spectra of the LED LCUs used in this study measured with MARC®- RC confirmed the single peak of Elipar^{TM} S10 and the dual peak nature of Bluephase® G2 LED LCUs. While only the other peak, at 409 nm, for the Bluephase® G2 LED LCU overlapped the absorption spectrum of TPO, both units' emission spectrum coincided with the absorption spectrum of CQ (figure 5-8).

Power Calculation

This study had an 80% power to detect a difference of 0.3 J/cm² in bottom energy values, 2.3% in light energy transmission, and 10.7% in VH ratio.
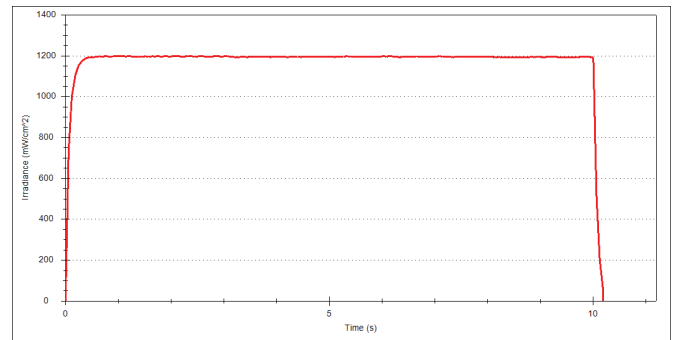

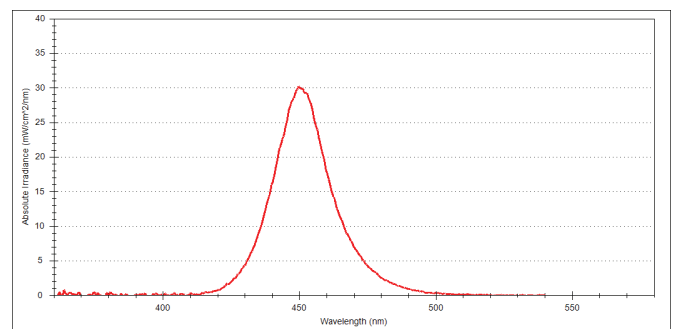Fig. 5. Graph produced during calibration of Elipar^{TM} S10 at 1200 mW/cm2

Fig. 6. Emission spectrum - Elipar^{TM} S10

Table III.

Material		Tetric EvoCeram BulkFil (viscous) (Ivoclar Vivadent) [TBF]			XtraFil (viscous) (Voco) [XF]			Surefil SDR (flowable) (Dentsply) [SDR]			Tetric EvoCeram Conventional (Ivoclar Vivadent) [TEC]		
Manufacturer's Total Energy Recommended		10-12 J/cm² 20 sec @ 500 mW/cm² 10 sec @ 1200 mW/cm²			8 J/cm² 10 sec @ 800 mW/cm²			11 J/cm² 20 sec @ 550 mW/cm²			10 J/cm² 20 sec @ 500 mW/cm² 10 sec @ 1000 mW/cm²		
		Bottom Total Energy (J/cm²) Mean(SD)	B/T Total Energy Transmission Mean(SD) %	B/T Ratio Mean(SD) VH %	Bottom Total Energy (J/cm²) Mean(SD)	B/T Total Energy Transmission Mean(SD) %	B/T Ratio Mean(SD) VH %	Bottom Total Energy (J/cm²) Mean(SD)	B/T Total Energy Transmission Mean(SD) %	B/T Ratio Mean(SD) VH %	Bottom Total Energy (J/cm²) Mean(SD)	B/T Total Energy Transmission Mean(SD) %	B/T Ratio Mean(SD) VH %
Elipar S10	1200X10 12 J/cm²	0.8(0.1)[1]	7.0(0.4)[2]	71.1(5.1)[3]	1.4(0.1)	11.9(0.4)	88.3(4.0)[4]	1.5(0.1)[5]	12.4(1.1)[6]	81.8(3.1)[7]	1.5(0.1)[8]	12.5(0.8)[9]	80.7(4.3)[10]
Bluephase G2	1200X10 12 J/cm²	0.7(0.1)[1]	6.2(0.5)[2]	63.0(4.8)[3]	1.0(0.1)	8.7(1.1)	90.0(3.5)[4]	1.3(0.1)[5]	11.0(0.9)[6]	82.4(2.5)[7]	1.3(0.1)[8]	11.2(1.0)[9]	78.9(5.3)[10]
CI		- 0.096 to 0.296	- 0.78 to 2.50	- 0.62 to 16.89	0.184 to 0.576	1.54 to 4.82	- 10.47 to 7.03	- 0.04 to 0.36	- 0.30 to 2.98	- 9.29 to 8.21	- 0.04 to 0.36	- 0.30 to 2.98	- 6.91 to 10.59

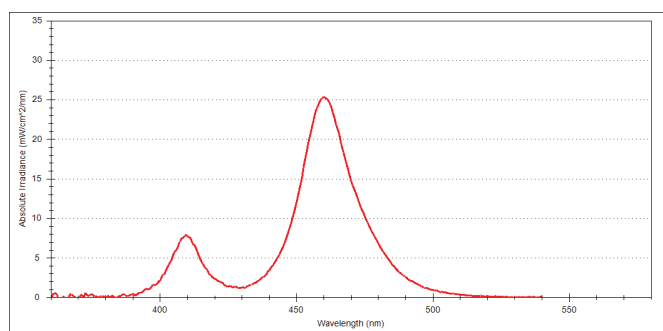Vertical: same superscript number = no significant difference.
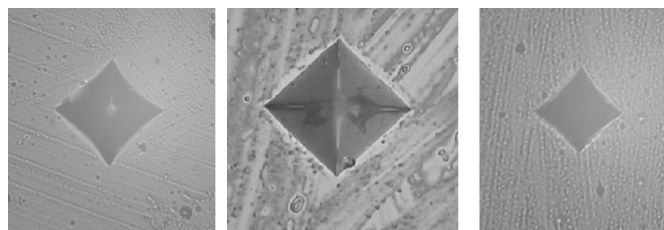
Fig. 7. Emission spectrum - Bluephase® G2

Fig. 8. Examples of VH indentations under X50 magnification

Discussion

Selection of materials

At the time of conducting this study, there were only ten brands of bulk-fill materials on the market worldwide. Five of them were viscous and five flowable. In this study, two viscous and one flowable bulk-fill materials were used from well-known manufacturers. These were felt to be representative of the two classes of RBC consistency.

The selection for the control composite, Tetric Evo-Ceram®, was made because it was sold by Ivoclar Vivadent, the same manufacturer producing the bulk-fill material, Tetric EvoCeram® Bulk Fill, and the dual peak LED LCU (Bluephase® G2), both of which were used in this study.

Consideration of the effect of shade of the RBCs was important since this may influence the results. There were different shade classifications between manufacturers and some materials were only available in one shade, a Universal shade. An effort was made to match the study shade to A2 whenever an appropriate material was available from the manufacturer to avoid extra-white or extra-dark shades and also because this was probably the most commonly used in clinical dental practice.

Selection of LCUs

Previous workers in their studies on BFMs used mainly a single peak LED LCU [23,26,27] or QTH [28]. Furthermore, most of the RBC manufacturers specify the irradiance and time required to cure their materials without specifying the type of LCU or the spectral wavelength to ensure compatibility. It is important that clinicians are made aware of the constituents of RBCs, especially the

type of photo-initiator, so that a compatible light source can be used [30]. Unfortunately, some manufacturers of these RBCs do not specify these details.

As the stated aim of this study was to investigate the effect of single peak and dual peak LED LCUs on curing bulk-fill materials, it was decided to compare one single peak (Elipar^{TM} S10) with one dual peak LED LCU (Bluephase® G2). Selection of the latter was made because it was sold by Ivoclar Vivadent, the same manufacturer producing the bulk-fill material, Tetric EvoCeram® Bulk Fill, and the conventional control RBC, Tetric EvoCeram®, both of which were used in this study. Elipar^{TM} S10 was selected from another company (3M ESPE) because it was well-known and had been used in several earlier studies [26].

Selection of Measuring Instruments

The use of MARC®-RC in this experiment provided an objective and reasonably straightforward method to standardise the LCU irradiance on the top surface and to measure the light transmission through the samples to the bottom surface. The availability of MARC®-RC for this study proved to be advantageous because measurements of the emission spectrum, irradiance, energy distribution and the total energy were easily obtained in a short period of time. Although, it would be optimum to measure DC directly to give a more direct result about a material's polymerization, the VH tester was easy to use and provided a means of indirect evaluating the degree of cure. Considering our lack of availability of devices like FTIR and micro-Raman, and reported good correlation between VH and DC, the VH tester was used in this study.

Selection of Total Energy value delivered to the top of the specimen

An important aspect, often ignored in previous studies, is where the light tip of the curing unit is placed in relation to the top surface of the material. When placed directly against the material, higher levels of energy would be delivered to top, and possible subsequently to the bottom surface. Alshali et al. [28] delivered a lower total energy to the top surface than was recommended by the manufacturers, placing the LCU at a distance from the surface. This would result in less total energy delivered to top surface which in turn may affect the curing of the material; thus not appropriate to judge the material as failed to be adequately cured.

In this study, the materials used varied in the total energy recommended by the manufacturers for curing their materials. The total energy recommended by the manufacturers of Tetric EvoCeram® Bulk Fill, X-tra fil®, SureFil SDR^{TM} and the control material, Tetric Evo-Ceram® ranged between 8 and 12 J/cm². Thus 12 J/cm² should have been more than sufficient to achieve adequate polymerization of the materials.

Calibration and Standardisation of LCUs

Prior to LCU calibration, a Mylar strip was placed over the top sensor. This was performed because the Mylar strip will be used on top and bottom surface of the samples, and thus, standardising the possible effect of its placement on the light scattering or absorption.

LCU calibration was repeated between experimental groups to ensure standardisation and not between each irradiation because it was not thought likely that significant deterioration of the LCUs through short continuous use would be expected.

Sample Thickness

A sample thickness of 4 mm was used in this study both because it was recommended by the manufacturers of the materials and to assess the non-incremental packing technique, the bulk packing technique. This was compared to 2 mm thick samples of the conventional composite which provided the standard.

Mylar strips were used by most of the earlier studies. However, Bucuta & Ilie [27] used a thin transparent plastic foil instead of the Mylar strip, because it demonstrated less light absorption in their primary tests than Mylar strip. The aims of using Mylar strips during sample preparation were to produce a flat and smooth surface and to minimise the formation of the oxygen-inhibition layer. However, this layer is not completely preventable during sample preparation and is unavoidable clinically. In this study, Mylar strips were used to make smooth surface for the ease of indentation measurement and to avoid the need for polishing. This was confirmed during the training on VH tester. Polishing of the sample surfaces has been done in several of the earlier studies [23,27]. However, in this study, the surfaces were not polished prior to testing. This was for several reasons. In the earlier studies, the aims of polishing were to produce a smooth surface and to remove the oxygen-inhibition layer. The latter has been shown to affect the reading of FTIR and micro-Raman when measuring the DC. As to the effect of polishing on VH measurements, it was reported that polishing sample surface will result in a higher VH value [31]. Conversely, Chung and Yap [32] concluded that surface hardness was independent of the surface finish provided because the indenter penetration was sufficiently deep. In addition, in this study, the bottom to top surface VH ratio (%) was recorded and not each surface value in isolation, thus this should not affect the ratio as neither surface was polished [31]. Polishing of the composite surfaces is not clinically relevant and does not simulate the clinical situation especially when polishing the bottom surface. Furthermore, when the surfaces were polished as a part of the VH test training, polishing resulted in a less clear surface when viewed under magnification as a result of dust creation. Therefore polishing was not carried out. Polishing also may result in heat generation that may affect polymerization [31]. Also, the use of Mylar strips in this study resulted in smooth and clear surfaces for the ease of VH indentation, which eliminated the need for surface polishing.

Sample Storage

In similarity to several earlier studies, in this study, after curing, the samples were stored for 24 hours in light-proof paper envelopes in the dark, thus allowing the dark phase of polymerization to take place. However, studies have varied in the use of a storage medium. Some studies stored their samples dry [26] while other stored them wet in distilled water [23,25,27]. In common, all of these studies kept the temperature at 37° C in an attempt to simulate the intra-oral condition. In contrast to this study, the samples were stored dry at room temperature. Given this was laboratory study, dry storage at room temperature was felt to be sufficient considering the standardisation methods used for all samples.

MARC®-RC and Measuring Light Transmission: Transmission measurements are the essential starting point to all other property testing of RBCs [33] thus the light transmission measurements, when combined with the VH ratio (%), would be beneficial and advantageous to this study. Prior to the calibration of the LCU in this study, a clear Mylar strip was placed in contact over the top sensor. This was performed because the Mylar strip was to be used on the top and bottom of the samples, thus, standardising the total energy delivered to the top surface of samples. Through the calibration process and measuring of the bottom light energy, it was shown that MARC®-RC had a good reproducibility, with the possible confounding factor of variety between manual sample preparations.

VH Indentation

In this study, the VH indentations were made using a 200 g load for ten seconds. The selection of these experimental conditions was based on the outcomes of many trials during training on the VH tester. The indentation should give the same VH value regardless of the load and duration used as the principle is measuring three-dimension indentation. It is all about how clear and easy to measure the indentation. If too small, it is likely to be hard to measure, or too big, the borders of the indentation may be out of focus.

The mean bottom light energy in the present investigation ranged from 0.7 to 1.5 J/cm^2, the light transmission (%) ranged from 6.2 to 12.5% and VH ratio (%) ranged from 63 to 90%. It is not always appropriate to compare the value reached in this study with previous works because the top energy delivered in the studies varied. Compared to the 12 J/cm^2 delivered in this study, Alshali et al. (2013) for example, delivered only 12 J/cm^2 to all samples despite some of the materials having higher energies recommended by their manufacturers. In contrast, Bucuta & Ilie [27] delivered 34.6 J/cm^2 top energy for all materials which was much higher than recom-

mended. It could be speculated that this difference arose because of variations in the distance between the LCU tip and the material surface factor considered in these studies for the LCU to be at 0 mm tip to top surface distance or at specific distance. Placing the tip of LCU in contact with the top surface of the restoration cannot be achieved clinically because of the tooth cusps, and hence the distance factor was not investigated in this study.

For this study, the standard selected to reflect the adequate curing of RBC using the bottom to top surface VH ratio (%) was 80% [26]. This was in agreement with all studies measuring VH ratio (%). In this study, with a VH ratio (%) of less than 80%, Tetric EvoCeram® Bulk Fill was not sufficiently cured in 4 mm thick increments when the manufacturers' recommendations were followed. This emphasized the care needed when following the manufacturers' instructions and the importance of carrying out such independent studies.

The findings of this study were in contrast to the results obtained by Bucuta & Ilie [2 7] and Alrahlah [26] when all BFMs used in their studies reached a VH ratio of 80% at 4 mm thickness. This might be explained by the fact that Bucuta and Ilie [26] used the LCU for 20 seconds in contact with the top surface of the samples (distance 0 mm) delivering 34.6 J/cm² top energy, and Alrahlah [26] used the LCU with an intensity of 1200 mW/cm² for 20 sec, delivering 24 J/cm² top energy, both of which were much higher than the manufacturers' recommendation.

The influence of the type of LCU used on the bottom energy values and light transmission was different from o n e material to another. The H0(1)=0 was accepted for Tetric EvoCeram® Bulk Fill, SureFil SDR™ and the conventional composite Tetric EvoCeram® where there was no significant difference in the bottom energy and light transmission (%) between the Elipar™ S10 and the Bluephase® G2 LCUs. In contrast, the bottom energy and light transmission (%) of X-tra fil® when cured with the Elipar™ S10 were significantly greater than when cured with the Bluephase® G2 LCUs. Therefore H0 (1) was partially rejected.

The influence of the type of LCU used on the VH ratio (%) was not significantly different for a l l materials when cured with the Elipar™ S10 compared to the Bluephase® G2. Therefore, H0 (2) was accepted.

The shade and opacity, and filler particle size and distribution can affect the transmission of light through a material [30,34,35]. and therefore the degree of cure [34,36,37]. The presence of unknown photo-initiators can also influence these results. In this study, the manufacturers failed to accurately state the photo- initiators contained in their materials and therefore it was assumed that the majority contain CQ [27].

As some materials also showed better VH ratio (%) at lower light energy transmission (%), it needs to be emphasized that the polymerization process in the bottom surface is not only dependent on the volume of light/pho-tons reaching this surface but also from the polymerization process already initiated in the upper layers. The results of better VH ratio (%) at lower light energy transmission were in agreement with the results obtained by Bucuta & Ilie [27].

One advantage of this study, over other studies, was to include the control material, Tetric EvoCeram®. This gave an over view of the BFMs compared to a conventional composite cured in a 2 mm thick increment.

In this study, the control material, Tetric EvoCeram®, when cured in a 2 mm thick increment, recorded a bottom energy of 1.5 J/cm², 1.3 J/cm² , a light transmission (%) of 12.5 %, 11.2% and a VH ratio (%) of 80.7%, 78.9%, when cured with Elipar™ S10 and the Bluephase® G2 LCUs, respectively.

Power of the Study

The power calculation based on the results of this study indicated that the difference detected at this power would be 0.3 J/cm² in bottom energy values, 2.3% in light energy transmission, and 10.7% in VH ratios. The greatest standard deviation of the means between the groups was chosen to avoid over-estimating the power. Therefore, the source of disparity in the detected difference may be related to the greater variance in these groups. Other groups in this study had SD ranges from 0.1 to 5.3 and therefore using a lower SD would result in lower defer-ence detection. This could be reduced in future work by increasing the sample size especially if the materials with a higher SD are used.

Conclusions

The RBC materials all showed different results when compared to each other.

Two of the BFMs did not reach a VH ratio of 80% when cured according to manufacturer's instructions.

Some materials will benefit from increasing curing time, a recommendation of 20 seconds curing over 10 seconds.

Both single peak and dual peak LCU's were equally effective for curing the studied bulk fill materials.

Manufacture's recommended total energy delivered to the top surface may not always be sufficient for effective curing.

It is important that manufacturers accurately identify the photoinitiators used in their materials, to ensure that a LCU with an appropriate emission spectrum is used.

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