



Effect of high irradiance and short exposure curing time on the fracture toughness of bulk-fill resin-based composite: an *in vitro* study

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ABSTRACT

Objectives: This study aimed to determine the effect of high irradiance and short exposure time on the fracture toughness of bulk-fill resin-based composites (RBCs).

Methods: Three RBCs were tested: Tetric PowerFill (TPF; Ivoclar Vivadent), Opus Bulk Fill APS (OBF; FGM Dental Group), and Filtek One Bulk Fill (FOB; Solventum). Sixty single-edge-notched disc specimens were prepared using a fracture toughness mold. Each group consisted of 20 samples, divided into two subgroups ($n = 10$). The RBCs were light-cured either for 3 seconds in high-irradiance mode ('3s cure') or for the manufacturer-recommended times (TPF, 10 seconds; OBF, 30 seconds; FOB, 20 seconds) in 'high power' mode using the Bluephase PowerCure (Ivoclar Vivadent). The peak spectral wavelength was measured using a spectrophotometer. Specimens were tested on a universal testing machine, and data were analyzed by two-way analysis of variance and Bonferroni test ($\alpha = 0.05$).

Results: Radiant exposure values (J/cm^2) were 9.5 for the 3-second mode and 12.4, 24.8, and 37.1 for 10, 20, and 30 seconds (high power mode), respectively. FOB (4.22 and 3.79 $MPa \cdot m^{0.5}$ for 20 and 3 seconds) had the highest mean fracture toughness, while OBF showed the lowest (2.01 and 2.10 $MPa \cdot m^{0.5}$ for 30 and 3 seconds). TPF produced intermediate results (2.72 and 2.70 $MPa \cdot m^{0.5}$ for 10 and 3 seconds). Exposure time did not affect TPF and OBF, while the 3-second exposure significantly reduced the fracture toughness for FOB.

Conclusions: The RBCs tested had different fracture toughness values regardless of exposure time. High irradiance and short exposure can reduce fracture toughness depending on the RBC tested.

Keywords: Composite resins; Polymerization; Stress fractures

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INTRODUCTION

Some bulk-fill resin-based composites (RBCs) can be light-cured in increments up to 5 mm thick [1,2]. However, not all light-curing units (LCUs) and their exposure modes can be used to cure such composite thickness, as they do not promote adequate photocuring at the deepest layers of bulk-fill composite restorations. Scientific articles have addressed this subject and shown the consequences of the deficient monomeric conversion that can affect some properties of the bulk-fill restorative materials [3,4].

In general, polymerization in the deeper areas of the RBC restoration occurs due to better light transmission through the bulk-fill RBC when compared to incrementally layered conventional composites. Lower light dispersion at the filler-resin matrix interface, reduced filler load, and changes in filler particle type contribute to higher light penetration. Also, bulk-fill RBC includes highly reactive photoinitiators, aromatic dimethacrylate, high-molecular-weight monomers, and fragmentation monomer technology to modulate polymerization shrinkage stress [5,6].

The development of RBCs has been carried out in parallel with high-power light-emitting diode (LED) LCUs that deliver a broad spectrum of blue and violet light, capable of exciting all photoinitiators used in contemporary RBCs. Additionally, many LCUs offer light exposure modes with varying exposure times and power outputs, delivering high irradiance of at least 1,000 mW/cm². This high irradiance from LED LCUs has allowed the light exposure time to be reduced from 40 to 10 seconds, or even 3 seconds [7,8].

Some LED LCUs, such as Bluephase PowerCure (Ivoclar Vivadent, Schaan, Liechtenstein) and Valo X-LED Curing Light (Ultradent Products Inc., South Jordan, UT, USA), are equipped with high power and short exposure modes ('3 seconds Cure' and '5 seconds-Xtra Power' modes, respectively) [9–11]. However, studies have shown that short curing times may generate fewer free radicals, compromising the polymerization reaction [12,13], which, in turn, reduces the depth of cure [10,14] and the physical-mechanical properties of bulk-fill RBCs [11,15].

The objective of this study was to investigate the in-

fluence of high irradiance and short exposure time from the LED LCU on the fracture toughness of three bulk-fill RBCs. The research hypotheses were: (1) the light exposure mode influences the fracture toughness of bulk-fill RBCs, and (2) the fracture toughness differs among bulk-fill RBCs regardless of the light exposure mode.

METHODS

Three high-viscosity bulk-fill RBCs were tested: Tetric PowerFill (TPF; Ivoclar Vivadent); Opus Bulk Fill APS (OBF; FGM Dental Group), and Filtek One Bulk Fill (FOB; Solventum, St Paul, MN, USA). The compositions and the lot number of bulk-fill RBCs used in this study, as well as their respective recommended light exposure time, are reported in Table 1.

The bulk-fill RBCs were light-polymerized with two light exposure modes ('3s cure' and 'high power') using the Bluephase PowerCure LED LCU. The light output from both light exposure modes of the LCU was measured using a spectrophotometer (MSC15W, SN 37560; Gigahertz-Optik, Amesbury, MA, USA) and the MSC15 measurement software ver. 2019.1.0 (Gigahertz-Optik). The peak spectral wavelength, power output, incident irradiance, and radiant exposure on RBC surfaces from the LED LCUs were determined.

Sixty single-edge-notched disc composite samples (6.5 mm diameter, 2.5 mm thick, and 3.5 mm notch) (Figure 1A) were prepared using a commercial fracture toughness metal mold (Odeme Dental Research, Luzerna, SC, Brazil) that is according to the ASTM Standard E-399-83 [16]. Each group consisted of 20 samples, which were divided into two subgroups ($n = 10$) according to the three bulk-fill RBCs and two different light exposure modes investigated in this study.

The bulk-fill RBC was inserted into the metal mold in a single increment using a stainless-steel spatula, ensuring complete filling and avoiding air entrapment. After filling, the mold was covered with a polyester strip (K Dent, Joinville, SC, Brazil), and the LCU was clamped and positioned perpendicular to the mold. The LCU tip was placed in contact with the polyester strip covering the composite surface, and bulk-fill RBCs were light-cured either using the 3s cure mode or according to the times recommended by the respective manufacturers

Table 1. Bulk-fill resin-based composites used, their compositions, and recommended light exposure times according to the manufacturers

Bulk-fill composite (lot number)	Composition	Recommended exposure time
Tetric PowerFill (Z04192)	Bisphenol A glycidyl methacrylate, ethoxylated bisphenol A dimethacrylate, 2,2-bis-(4-(3-methacryloxypropoxy)phenyl)propane, urethane dimethacrylate, tricyclodecane dimethanol dimethacrylate, propoxylated bisphenol A dimethacrylate, addition fragmentation chain transfer, β -allyl sulfone, Ba-Al-Si glass, ytterbium trifluoride, Si-Zr mixed oxide and copolymers (79% by weight)	3 sec or 10 sec ^{a)}
Opus Bulk Fill APS (171023)	Urethane-dimethacrylate monomers, stabilizers, photoinitiator composition (APS), co-initiators, stabilizers, pigments, and silanized silicon dioxide (79% by weight)	30 sec
Filtek One Bulk Fill (NC88136)	Aromatic urethane dimethacrylate, diurethane dimethacrylate, 1,12-dodecane dimethacrylate, ethyl 4-dimethyl aminobenzoate, silane-treated ceramic, silane-treated silica, ytterbium fluoride and silane-treated zirconia (76.5% by weight)	20 sec

^{a)}Three seconds or 10 seconds when using the light-curing units in '3s cure' mode and 'high power mode,' respectively.

Tetric PowerFill: Ivoclar Vivadent, Schaan, Liechtenstein; Opus Bulk Fill APS: FGM Dental Group, Joinville, SC, Brazil; Filtek One Bulk Fill: Solventum, St Paul, MN, USA.

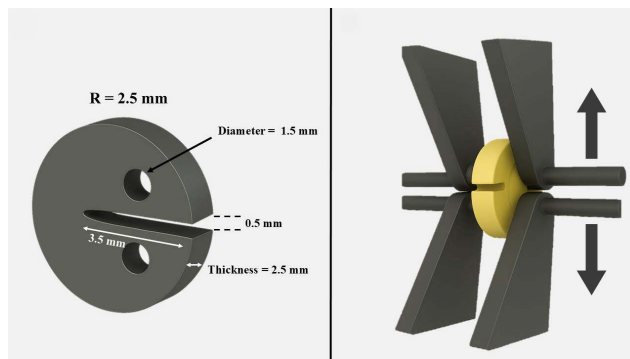


Figure 1. Dimensions and shape of the composite specimen and the fracture toughness testing apparatus.

(TPF, 10 seconds; OBF, 30 seconds; FOB, 20 seconds) in the high power mode, which represented the control groups.

Polymerized specimens were removed from the mold, stored in distilled water at 37°C for 24 hours, and then tested in a universal test machine (Instron 4411; Instron Corp., Norwood, MA, USA) at a 1.0 mm/min crosshead speed (Figure 1B). The fracture load values were acquired and applied to the formula below to calculate the fracture toughness (in MPa·m^{0.5}):

$$\text{Fracture toughness (KIC)} = (PL/BW1.5) Y$$

where: *P*, load fracture; *L*, distance between holes; *B*, specimen thickness; *W*, specimen width; *Y*, geometrical function dependent on *a/W*, and '*a*' is the notch length.

$$Y = [2.9(a/w)^{1/2} - 4.6(a/w)^{3/2} + 21.8(a/w)^{5/2} - 37.6(a/w)^{7/2} + 38.7(a/w)^{9/2}]$$

Fracture toughness data were analyzed for homoscedasticity with Levene test and normality with the Shap-

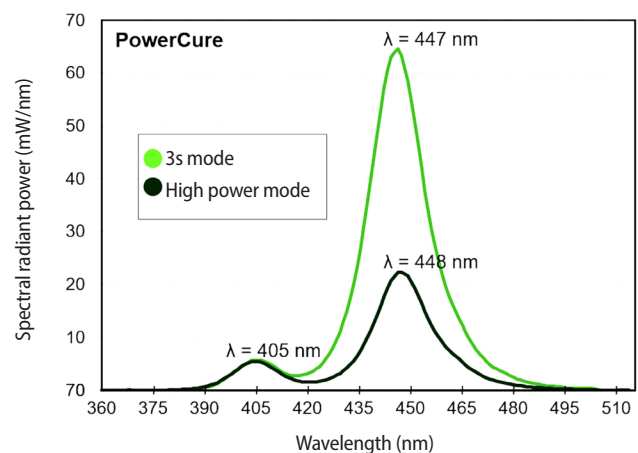


Figure 2. Spectral emissions from the Bluephase PowerCure (Ivoclar Vivadent, Schaan, Liechtenstein) light-curing unit, according to the curing mode.

iro-Wilks test. Data were analyzed by two-way analysis of variance (ANOVA; factors: 'type of bulk-fill RBC' and 'exposure mode') and Bonferroni test ($\alpha = 0.05$). The SAS software ver. 9.4 (SAS Institute Inc, Cary, NC, USA) was used for all the statistical analyses.

RESULTS

Figure 2 illustrates the emission spectra and the real-time irradiances delivered by the Bluephase PowerCure LCU for each light exposure mode. The emission peaks in the 3s cure mode were at 405 nm (violet) and 447 nm (blue), respectively. In the high power mode, the violet light emission peak remained the same at 405

nm and delivered the same amount of violet light, but the amount of blue light increased (448 nm). This 1-nm change was within the measurement uncertainty of the equipment used in the study. Figure 3 illustrates that 3s cure mode reached irradiance value of 3,178 mW/cm² that was maintained for only 3 seconds, while the high power mode delivered a constant irradiance of approximately 1,238 mW/cm² for 20 seconds in high power mode.

Table 2 reports the power (mW), irradiance (mW/cm²), and the radiant exposure (J/cm²) values from both light exposure modes of Bluephase PowerCure LCU. For the 3s cure mode, the LCU delivered 1,597 mW power output, 3,178 mW/cm² irradiance, and 9.5 J/cm² radiant exposure. The high power mode delivered a power output of 622 mW and an irradiance of 1,238 mW/cm². The exposure times of 10, 20, and 30 seconds delivered radiant exposures of 12.4, 24.8, and 37.1 J/cm², respectively.

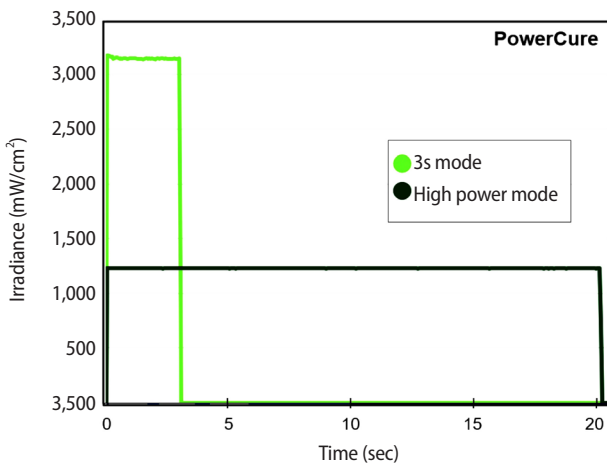


Figure 3. Real-time irradiances delivered from the Bluephase PowerCure (Ivoclar Vivadent, Schaan, Liechtenstein) light-curing unit, according to the light exposure mode.

Table 3 reports the fracture toughness means (MPa·m^{0.5}) for bulk-fill RBCs and exposure modes. The two-way ANOVA demonstrated that both ‘type of bulk-fill RBC’ ($p < 0.001$) and ‘exposure mode’ ($p < 0.001$) significantly influenced fracture toughness, with no significant interaction between the factors ($p = 0.100$).

The curing modes did not affect the fracture toughness results of TPF ($p = 0.974$) and OBF ($p = 0.632$), while the 3s cure mode significantly reduced the fracture toughness for FOB ($p = 0.020$). On the other hand, the highest fracture toughness means were found for FOB (4.22 and 3.79 MPa·m^{0.5} for 20 and 3 seconds, respectively), while the lowest one was for OBF (2.01 and 2.10 MPa·m^{0.5} for 30 and 3 seconds, respectively), regardless of the light exposure mode. TPF produced intermediate results (2.72 and 2.70 MPa·m^{0.5} for 10 and 3 seconds, respectively).

DISCUSSION

The first research hypothesis that the light exposure mode influences the fracture toughness of bulk-fill RBCs was accepted only for FOB bulk-fill RBC, because the fracture toughness of OBF and TPF cured using the 3s cure or high power modes did not differ statistically. According to the FOB RBC manufacturer, for LCUs that

Table 2. Power output (mW), incident irradiance (mW/cm²), and radiant exposure (J/cm²) values from the ‘3s cure’ and ‘high power’ modes

Variable	3s cure mode	High power mode
Power output (mW)	1,597	622
Incident irradiance (mW/cm ²)	3,178	1,238
Radiant exposure (J/cm ²)	9.5 ^{a)}	12.4, 24.8, 37.1 ^{b)}

Exposure time: ^{a)}3 sec; ^{b)}10, 20, and 30 sec.

Table 3. Fracture toughness (MPa·m^{0.5}) of the bulk-fill RBCs tested according to the exposure modes used

Bulk-fill RBC (recommended exposure time)	3 sec (‘3s cure’ mode)	Recommended exposure (‘high power’ mode)
Tetric PowerFill (10 sec)	2.70 ± 0.60 ^{bA}	2.72 ± 0.37 ^{bA}
Opus Bulk Fill APS (30 sec)	2.10 ± 0.28 ^{cA}	2.01 ± 0.19 ^{cA}
Filtek One Bulk Fill (20 sec)	3.79 ± 0.29 ^{bB}	4.22 ± 0.53 ^{aA}

Values are presented as mean ± standard deviation.

Different lowercase letters indicate significant differences among bulk-fill RBCs within the same curing mode ($p < 0.05$).

Different uppercase letters indicate significant differences between curing modes for the same bulk-fill RBC ($p < 0.05$).

Tetric PowerFill: Ivoclar Vivadent, Schaan, Liechtenstein; Opus Bulk Fill APS: FGM Dental Group, Joinville, SC, Brazil; Filtek One Bulk Fill: Solventum, St Paul, MN, USA.

deliver an irradiance of 1,000 mW/cm² or greater, the light exposure time must be 20 seconds, while for LCUs with lower irradiance (550–1,000 mW/cm²), the light exposure time must be 40 seconds. Thus, this study used the recommended exposure time (20 seconds), and the FOB composite achieved higher fracture toughness than when exposed for only 3 seconds. The 20-second exposure time in the high power mode delivered a radiant exposure of 24.8 J/cm² and irradiance around 1,200 mW/cm². In comparison, the '3s cure' mode delivered only 9.5 J/cm² radiant exposure and 3,178 mW/cm² irradiance. The difference in radiant exposure might explain the difference in fracture toughness between the cure modes used to polymerize the FOB. This contrast highlights two distinct energy-delivery strategies: an ultrashort, high-intensity exposure versus a lower, sustained output (Figure 3).

The 3-second exposure time is the shortest exposure time recommended by any RBC manufacturer. The ability of TPF to be photocured in a short light exposure time is most likely due to the types and concentration of photoinitiators (combination of camphorquinone and Ivocerin) used in this bulk-fill RBC [14,15]. The photoinitiator system in OBF bulk-fill RBC is a proprietary advanced polymerization system (APS), which yielded similar fracture toughness regardless of the light exposure mode used or the radiant exposure delivered to the RBC. The same result was obtained for the TPF bulk-fill RBC; however, this bulk-fill RBC is recommended to be cured in the 3s cure mode on the Bluephase PowerCure LCU. According to its manufacturer, the TPF can also be cured for 10 seconds using the high power mode from the same LCU.

The second research hypotheses that fracture toughness differs among bulk-fill RBCs, regardless of the light exposure mode was accepted because the RBCs showed distinct fracture toughness values and the light exposure mode did not change the order from the highest fracture toughness (FOB: for 20 seconds, 4.22 MPa·m^{0.5} and for 3 seconds, 3.79 MPa·m^{0.5}) to the lowest one (OBF: for 20 seconds, 2.01 MPa·m^{0.5} and for 3 seconds, 2.10 MPa·m^{0.5}). The TPF composite produced intermediate results (for 20 seconds, 2.70 MPa·m^{0.5} and for 3 seconds, 2.72 MPa·m^{0.5}).

The filler content of FOB is a combination of silica,

non-agglomerated and cluster fillers, and ytterbium trifluoride particles. The resin matrix contains two methacrylate monomers (aromatic urethane dimethacrylate [AUDMA] and addition-fragmentation methacrylate [AFM] monomers) that help reduce polymerization shrinkage stress. AUDMA is a high-molecular-weight AUDMA that decreases the number of reactive groups in the resin, while the AFM contains a reactive site that cleaves through a fragmentation process during polymerization. The cleaving process tends to reduce the polymerization shrinkage stress, and the monomer fragments can react with each other or with other reactive monomer sites, thereby maintaining the physical properties of FOB bulk-fill RBC due to increased monomer conversion. A study that used single-edge bend bar-shaped specimens and loaded them in a three-point bending test showed that the fracture toughness of FOB was among the highest of the bulk-fill RBCs investigated [17], corroborating this study.

The TPF contains a combination of monomers (bisphenol A glycidyl methacrylate, ethoxylated bisphenol A dimethacrylate, 2,2-bis[4-(3-methacryloxypropoxy)phenyl]propane, urethane dimethacrylate [UDMA], tricyclodecane dimethanol dimethacrylate, propoxylated bisphenol A dimethacrylate) and four types of particles (barium glass, ytterbium trifluoride, mixed oxide, and prepolymers). The polymerization shrinkage stress is controlled by the 'addition fragmentation chain transfer' and the β -allyl sulfone-based dimethacrylate networks. Both are also responsible for improving the polymer's physical properties and enhancing the homogeneity of the network architecture [18], particularly under high irradiance and fast photocuring [19]. However, some studies have recommended longer exposure time and lower irradiance to improve the degree of conversion and the depth of cure of RBCs. Also, the recommendation was to reduce the monomer elution, polymerization shrinkage, and the porosity of TPF [7,8]. On the other hand, a study that compared the fast high irradiance (3s cure mode from Bluephase PowerCure LCU) to conventional curing mode for 20 seconds (irradiance: 1,600 mW/cm² from an Elipar LED LCU, TM S10; 3M ESPE, St Paul, MN, USA) showed that the light exposure mode did not affect the mechanical properties (flexural strength, modulus, and fracture toughness) of two RBCs

(TPF and Essentia U, GC Corp., Tokyo, Japan) [20].

The OBF contains UDMA monomer, silanized silicon dioxide as the filler particles and the APS photoinitiator system. This study showed the lowest fracture toughness among the bulk-fill RBCs. A study showed that the flexural strength of OBF was lower than that obtained for X-tra fil (VOCO, Cuxhaven, Germany) [21]. However, another study showed that the OBF light-cured for 40 seconds presented better results for the evaluated mechanical properties than Tetric EvoCeram Bulk Fill (Ivoclar) light-cured for 10 seconds [22]. An *in vitro* study showed that OBF had mechanical performance and reliability similar to those of conventional and incremental RBCs [23].

Thanoon *et al.* [24] showed that the response of bulk-fill composites to a high-irradiation protocol varied with composition and viscosity, with light transmission and degree of conversion occurring more rapidly in low-viscosity materials. Another study indicated that high irradiance and short exposure times can affect the depth of cure of bulk-fill RBCs, and fast curing with high irradiance should be used only for some bulk-fill composites [4]. Ribeiro *et al.* [13] demonstrated that short exposure times (1 to 3 seconds) produced inferior physical-mechanical properties (fracture toughness, energy absorption, and Vickers hardness) in some composites compared to a 10-second exposure at a lower irradiance [13]. On the other hand, the TPF showed superior thermal and monomer conversion results when exposed to high irradiance for a short time (1–5 seconds) compared with the Tetric EvoCeram conventional bulk-fill composite [24].

Most studies have used bar-shaped specimens [25–28], unlike this study, which used disc-shaped ones [13]. The metal mold used in this study produced samples 6.5 mm in diameter and 2.5 mm thick (Figure 1A). Bulk-fill RBCs are recommended to be light-activated to a thickness of up to 4 or 5 mm, but in this study, the composite thickness was lower than their recommended values. The shape and thickness of the composite samples, in addition to the use of contemporary bulk-fill RBCs, may explain their superior fracture toughness values obtained in this study when compared to results (0.4 to 1.6 MPa·m^{0.5}) reported by previous studies that used conventional RBCs and others that are no longer available

[25–29].

The composites tested in this study are indicated for large restorations [30–33] and, when under-polymerized, may result in poor restoration longevity. These composites have been extensively studied regarding exposure time and degree of conversion, hardness, and flexural strength [34–37], but fracture toughness reflects a material's susceptibility to cracking; a higher fracture toughness value indicates that the material is less likely to fracture [38].

The results of this study suggest that the type of bulk-fill RBC and the light exposure mode have a significant influence on the fracture toughness of FOB bulk-fill RBC. This study did not standardize the radiant exposure delivered to all bulk-fill RBCs because different exposure times in high power mode were used to photo-cure the three bulk-fill RBCs. The exposure time was set to the light-activation time recommended by the manufacturers. The same exposure time for the tested bulk-fill RBCs might alter the fracture toughness results.

CONCLUSIONS

Light-curing with short exposure time and high irradiance did not reduce the fracture toughness of OBF and TPF bulk-fill restorative materials. However, this finding cannot be generalized to all bulk-fill composites, because the short high-irradiance protocol yielded lower fracture toughness for FOB.

CONFLICT OF INTEREST

No potential conflict of interest relevant to this article was reported.

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AUTHOR CONTRIBUTIONS

Conceptualization, Data curation: Sahadi BO, Giannini M. Formal analysis: Sahadi BO, Rifane TO, André CB, Araújo-Neto VG, Price RTB. Funding acquisition, Project administration,

Visualization: Giannini M. Giannini M. Investigation: Rifane TO, Araújo-Neto VG, Price RTB. Methodology: Sahadi BO, Rifane TO, Araújo-Neto VG, Price RTB. Supervision: Price RTB, Giannini M. Writing - original draft: Sahadi BO, Rifane TO, Giannini M. Writing - review & editing: Price RTB, Giannini M. All authors read and approved the final manuscript.

DATA SHARING STATEMENT

The datasets are not publicly available but are available from the corresponding author upon reasonable request.

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