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Effect of light curing units on Degree of Conversion, microhardness and Mechanical properties of a bulk fill composite

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Trabalho de conclusão de curso apresentado a Faculdade de Odontologia da UFU, como requisito parcial para obtenção do título de Graduado em Odontologia

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ATA DA COMISSÃO JULGADORA DA DEFESA DE TRABALHO DE CONCLUSÃO DE CURSO DO (A) DISCENTE **Júlia Freitas Gonçalves** DA FACULDADE DE ODONTOLOGIA DA UNIVERSIDADE FEDERAL DE UBERLÂNDIA.

No dia **28 de maio de 2019**, reuniu-se a Comissão Julgadora aprovada pelo Colegiado de Graduação da Faculdade de Odontologia da Universidade Federal de Uberlândia, para o julgamento do Trabalho de Conclusão de Curso apresentado pelo(a) aluno(a) **Júlia Freitas Gonçalves**, COM O TÍTULO: "**EFFECT OF LIGHT CURING UNITS ON DEGREE OF CONVERSION, MICROHARDNESS AND MECHANICAL PROPERTIES OF A BULK FILL COMPOSITE**". O julgamento do trabalho foi realizado em sessão pública compreendendo a exposição, seguida de arguição pelos examinadores. Encerrada a arguição, cada examinador, em sessão secreta, exarou o seu parecer. A Comissão Julgadora, após análise do Trabalho, verificou que o mesmo se encontra em condições de ser incorporado ao banco de Trabalhos de Conclusão de Curso desta Faculdade. O competente diploma será expedido após cumprimento dos demais requisitos, conforme as normas da Graduação, legislação e regulamentação da UFU. Nada mais havendo a tratar foram encerrados os trabalhos e lavrada a presente ata, que após lida e achada conforme, foi assinada pela Banca Examinadora.

Uberlândia, 28 de maio de 2019.

Prof. Dr. Murilo de Souza Menezes *Universidade Federal de Uberlândia – UFU*	Aprovado Aprovado/Reprovado
Prof. Dr. João César Guimarães Henriques *Universidade Federal de Uberlândia – UFU*	Aprovada Aprovado/Reprovado
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SUMÁRIO

Title: Effect of light curing units on Degree of Conversion, microhardness and mechanical properties of a bulk fill composite

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Abstract

Objectives: Evaluated mechanical and physical properties of bulk-fill composites (RBC) polymerized with different light-curing units (LCUs).

Materials and Methods: Composite discs (2x4 mm thickness) were produced with Tetric EvoCeram Bulk-Fill (TNB), Filtek Bulk-Fill (FBF) and Opus Bulk-Fill (OBF), divided into 5 groups (n=5) according to LCUs: polywave LED Bluephase, polywave LED VALO, monowave LED Radiical, monowave LED Emitter C and quartz–tungsten– halogen Optilux. Degree of conversion (DC) was determined using Fourier transform infrared spectrometer, microhardness (KHN) was measured with a Knoop indenter and mechanical properties with tensile strength diametral (DTS). Data were analyzed using two-way ANOVA and Tukey's post hoc test (α=0.05).

Results: The DC showed OBF and TNB was signficant difference than FBF, regardless of LCUs. The Bluephase and Optilux present highest values. Radicall and Emitter present lowest values, regardless composite resin. The KHN showed no statistical diference between RBCs for Bluephase and Optilux. For Valo, Radicall and Emitter, the TNB presented lowest values. Regarding the type of composite resin, the OBF showed no statistical diference between LCUs. The Bluphase and Optilux presented highest values for TNB. The Emitter showed lowest value for FBF, while Bluephase showed highest value. For DTS, regardless composite resin, Bluephase, Valo and Optilux showed highest values. The RBCs presented no statistic diference regardless LCUs.

Conclusions: The LCUs influenced in DC, KNH and DTS between RBCs.

Keywords: composite resins, polymerization, Fourier-transform infrared spectroscopy, hardness, Curing Lights Dental;

CLINICAL SIGNIFICANCE:

Bulk-fill resin-based composites are not a homogeneous group of materials. Their degree of conversion, microhardness and mechanical properties are affected by differents light-curing units used in dental practice.

Introduction

The clinical use of resin-based composite (RBC) has increased substantially in recent years due to aesthetic demands of patients, improvements the formulation and simplification of bonding procedures (1). In an attempt to accelerate the restoration process in posterior teeth, bulk fill composites were introduced into the dental market. This category currently gains popularity due to the simplification and reduction of time, making the clinical procedure more simple (2). Unlike conventional RBC, that are layered to a maximum thickness of 2 mm (3), bulk fill RBC can be placed up to 4 mm thick with suitable polymerization and low polymerization shrinkage (4, 5).

Polymerization effectiveness of light-activated materials is related by absorbed by photoinitiators and free radicals formed in the presence of activators. Free radicals subsequently trigger the polymerization reaction. Ideally, during the polymerization reaction all the monomer in the resin composite material would have been converted to polymer (6). The adequate conversion of RBC materials is essential in determining their physical and mechanical performance (7, 8).

To improve the depth of polymerization and the decrease of the polymerization shrinkage stress, variety of monomer and photoinitiators modifications are introduced. Moreover, increased translucency of these resins allows greater light transmission and adequate cure depth (9). Addition to it stress relieving monomers, specific polymerization modulators, more reactive photoinitiators and prepolymerized particles, results in less polymerization contraction (10, 11).

The main photoinitiators used in bulk fill is camphorquinone (CQ), phenylpropanedione (PPD), acyl phosphine oxide (APO) and germanium-based compounds such as bis-(4-ethoxybenzoyl) diethyl-germane (Ivocerin), which the polymerization process is usually activated by applying visible light. The activation of these photoinitiators is dependent upon the appropriate LCU and wave-length (12).

Two common types of dental LCUs are quartz-halogen-tungsten (QHT) and high-intensity light-emitting diode (LED). QTH curing unit has been extensively used for a long time. Their wide range of wavelengths (370 nm and 550 nm) allows for the curing of composites employing CQ, PPD and APO as photoinitiators (12, 13). However, light-emitting diode (LED) is becoming popular in dental practice, due to lower degradation over the time, not require cooling, consume less energy, have extended times without significant loss of light intensit and have blue light emission without requiring filter. The output from conventional single peak LED units, is designed to activate the CQ photoinitiator (12, 14). Both first- and second-generation LED lights used only one type of LED (monowave [single-peak] technology) and were unable to cure composites with PPD and APO initiator systems. Some third generation broad-spectrum LED units include additional LED emitters that produce light at these lower wavelengths, which are in the 'violet' range, to make these LCUs compatible with a wider range of photoinitiators and avoid wavelength-compatibility issues by deploying polywave (dual/multipeak) technology (14). The clinicians also need to know the emission spectrum of the light emitted from the LCU, so that they can match the light to the resin composite they are using (15).

Therefore, the objective of this study was to compare the effectiveness of cure of three bulk-fill composites with polywave LEDs, monowave LEDs, and conventional halogen curing lights using degree of conversion (DC), knoop microhardness (KHN) and diametral tensile strength (DTS) methodologies. The null hypothesis that no difference in the effectiveness of cure existed between the polywave LED, monowave LED, and QHT curing lights for different bulk-fill composites if the total light energy was kept constant.

Material and Methods

Three high-viscosity bulk-fill resin-based composites were investigated: Tetric EvoCeram Bulk Fill (TNB) containing the initiators CQ and Ivocerin; Filtek Bulk Fill (FBF) containing the initiators CQ and Opus Bulk Fill (OBF) containing the initiators CQ and APS (advanced polymerizarion system). The compositions and manufacturer information for these products are presented in Table 1.

Composite discs (n=5) of each material were prepared and polymerized with five different LUCs. The composites were placed in a single increment into cylindrical silicone mold (Contrast, VOCO, Cuxhaven, Germany); with 2-mm height and 4-mm diameter. A transparent mylar strip was placed on the top of the mold, and a glass slide were used to held down the excess of the materials. The composites were then irradiated through the mylar strip using either a polywave LED (Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein) or (VALO Cordless Ultradent, South Jordan, UT, United States), a monowave LED (Radii cal, SDI, Basywater, Victoria, Australia) or (Emitter C, Schuster, Santa Maria, RS, Brazil) and QHT (Optilux 501, Kerr, Orange, CA, USA). Table 2 provides details of the LCUs used in the study. Each specimes was irradiated once time according the LCUs manufacturer. Light emittance was measured daily using a radiometer (Kondortech Equipamentos Odontológicos, São Carlos, SP, Brasil) to ensure consistent irradiance.

Degree of conversion

The DC was monitored 24 hours after specimens preparation using Fourier Transformed Infrared Spectroscopy (FTIR, Vertex 70, Bruker Optik GmbH, Ettlingen, Germany). Specimens were stored in a dry and dark container at 37ºC. The DC was assessed using FTIR with attenuated total reflectance (ATR crystal) sampling, mid-infrared (MIR) and deuterated triglycine sulfate (DTGS) detector elements (Bruker Optic), with a 4 cm^{-1} resolution and co-addition of 32 scans. All analyses were performed under controlled temperature (25º ± 1ºC) and humidity (60±5%). The DC was calculated from the equivalent aliphatic (1638 cm-1) and aromatic (1608 cm-1) molar ratios of cured (C) and uncured (U) composite resin specimens according to the following equation $DC = (1 – C/U) x 100$.

Knoop microhardness

After storage for 24 hours, at 37ºC in distilled water, the same specimen locations analyzed using Fourier transform infrared spectroscopy were also used to determine KHN values. A microhardness tester (Microhardness tester Future-Tech FM-700, Instytut Metalurgii Żelaza, Gliwice, Poland) with a Knoop diamond indenter was used to apply a static load of 100 g (0.98 N) for 10 s to each composite surface. For each specimen, the averages of three indentations were used in the statistical analysis.

Diametral Tensile Strength

A diametral tensile strength test was performed in the specimens after used for obtaining DC and KHN (n=5) using a mechanical testing machine (DL 2000, EMIC, São José dos Pinhais, Brazil). Specimens were positioned vertically on the testing machine between a stainless-steel flat tip and base; a compressive load was applied vertically on the lateral portion of the cylinder at a crosshead speed of 0.5 mm/min, producing tensile stresses perpendicular to the vertical plane passing through the center of the specimen until failure. After each compressive test, the fracture load (F) was recorded in Newtons (N), and the diametral tensile strength (σt) was calculated (MPa) as follows: σt = 2F/ πdh where, d is the diameter (4 mm), h the height (2 mm) of specimens, and the constant π is 3.1416.

Statistical analysis

The statistical analysis was performed using the SigmaPlot (Sigma plot Verson 12.0, Systat Software Inc., San Jose, CA, USA). The data were normal and homoskedastic for all experiments. Two-way ANOVA/Tukey's test was performed to evaluate degree of conversion, Knoop microhardness and diametral tensile strength data (factors: Bulk-fill Resin Composites and dental light curing unit). A 95% level of significance was adopted ($\alpha = 0.05$) for all tests.

Results

Three bulk fill RBCs and five dental LCU were evaluated to determine the effect on the DC, DTS, KHN. Means and standard deviations for DC are presented in Table 3. In terms of LCUs polymerization ($P = <0.001$), Bluephase showed higher DC, with no signifcant difference for Optilux. Valo resulted in intermediate values that were not different from ther Optilux. Radicall and Emitter showed lower DC. Regarding the restorative materials tested, the OBF and TNB groups had the highest DC values and the FBF group had the lowest ($P = <0.001$). There is not a statistically significant interaction between bulk fill RBC and LCU ($P = 0.070$).

The KHN results are presented in Table 4. ANOVA showed significant difference for factors ($p < 0.05$), as well as for interactions of factors ($p = 0.011$). The LCUs showed no difference for the OPUS composite. TNB showed higher KHN for Bluephase and Optilux compared to Radicall, Valo and Emitter. FBF showed higher KHN for Bluephase, with signifcant difference only for Radicall. Bulk-fill composite resins not present significant difference when polymerized with Bluephase and Optilux. The TNB composite showed lower KHN for Valo than other RBCs. The TNB presented lower KHN and FBF showed no diference statistically between OBF and TNB for Radicall and Emitter.

The DTS results are presented in Table 5. For DTS analysys, Bluephase showed higher DTS, with no signifcant difference for Valo and Optilux. Radicall and Emitter showed lower DTS, with no signifcant difference for Optilux and Valo ($P = 0.023$). There is not a statistically significant difference among the different levels of bulk fill RBC ($P = 0.146$). There is not a statistically significant interaction between bulk fill RBC and LCU ($P = 0.301$).

Discussion

Since their introduction into the dental market, LCUs have been regularly improved by manufacturers to provide better polymerization. The effectiveness of cure of bulk-fill composites with polywave LED, monowave LED, and conventional halogen curing lights was evaluated. According to the results of the present study, the null hypothesis was rejected as significant differences in effectiveness of cure existed between the different lights and bulk fill resin composites.

Adequate photopolymerization of resin-based composites is a crucial factor for optimization of mechanical properties, biocompatibility and clinical longevity of composite restorations (16, 17). Photopolymerized resins initiate the polymerization process through light absorption by a photoinitiator, which, once activated, reacts as a reducing agent to produce free radicals. From that point on, there is the polymerization of the methacrylic monomers that form a polymeric matrix with cross links (6). Curing efficacy can be assessed by several techniques. FTIR spectroscopy is a direct method used to analyze the chemical bonds of polymers. This technique allows detection of the amount of unreacted C=C in the resin matrix (18), but this property alone is not enough to characterize all the composite material structure (18, 19). Hardness measurement is an indirect method that has proven to be the best indicator of the extent of polymerization and has been classified as a high level of evidence (20).

Several composite factors can affect the degree of polymerization of bulk fill RBCs, such as, photoinitiators, type of resin-matrix, filler type, size and loading, viscosity of material, thickness and opacity (21). In the present study, composites are regular materials with modified monomers and present relatively similar loading % by volume of filler content. The thickness controlled during the experiment, since all specimen have the same dimension (2 x 4 mm). All LCU have the hability to give superior degree of conversion and hardness of OBF. This result can be explained by their photoinitiators APS (APS: Advanced Polymerization System). According to the manufacturer, APS has a small concentration of canphorquinone and an addition of other initiators / co-initiators. This system can potentiate the energy coming from the LCU, because the photoinitiators interact with each other and release more free radicals by increasing the polymerization capacity. The bulk-fill composite TNB also had a high degree of conversion. This RBC has the photoinitiator Ivocerin, a derivative of dibenzoyl germanium, in addition to the camphorquinone/amine initiator system. Ivocerin is excited by short wave visible light (380–450 nm) and is a more efficient free-radical generator than camphorquinone, leading to polymerization and monomer conversion. For microhardness, Bluephase and Optilux showed more efficient for excited Ivocerin than the others LCU. FBF containing the initiator CQ, that have good hardness for Bluephase, Valo, Optilux and Emitter. Hardness testing was done 24 hours after photopolymerization to allow for composite postcure.

Curing efficacy can be affect also by light-curing constraints, include light type, total light energy, intensity, spectral wavelengths, exposure time, curing distance and shape of the different curing tips. This study used laboratory conditions for light-curing, in which no distance remained between the tip of the light source and the restorative material. So, this variable including curing distance and exposure time were controlled during the experiment. The results can be attributed to light type. The results showed that DC of Bluephase and Optilux was higher than the others LCUs, regardless of bulk fill RBC. The harder surface associated with halogen light could be contributed in part to a thermal effect. Studys reported the heating of composites from halogen lights has been increase hardness (22, 23). Uniform distribution of emitted energy in all layers of restorative material has been reported as crucially important to produce suffcient numbers of free radicals for adequate polymerization (24).

Time-saving procedures are an on going demand for restorative applications. A third generation LEDs curing units were marketed that claims to reach high irradiances and allow for shorter clinical application times. In this study, Bluephase and Valo were used in hight polywave mode. The results of Valo were significantly lower than Bluephase for conversion degree. (26).

Acoording to Pereira AG *et al* 2016, the battery level of the cordless LED unit affected the battery voltage and light intensity of the equipment in addition to the degree of conversion and mechanical properties of resin composite. Low battery levels affect the battery voltage and consequently influence the light intensity of cordless LED units, also changing some properties of composite resins. In this study, Valo, Radicall and Emitter are cordless LED unit, but before specim preparations, the maximum number of cycles that could be completed with the fully charged batteries (100%) was determined (27).

This study used laboratory conditions for light-curing. The situation may be different and worse in clinical situations, when the distance between the light source and the polymeric restorative material is increased by limiting factors, such as in the restoration of deep cavities, teeth position and morphology of fissures and cusps, which decreases irradiance and may impair polymerization efficacy. The bulk fill RBCs, usually applied in deep posterior cavities. A recent study suggest that, the operator visibility and access was the worst in the posterior region, this fact affected negatively the irradiance for light curing units (28). The intensity and DC decrease with increasing distance, between the curing tip and the material surface to be irradiated (29) .

Bulk-fill composite materials are likely to fulfil some requirements, low polymerization shrinkage, ease of use, improved depth of cure and enhanced physical characteristics. The latter is particularly important since bulk-fill composites will represent all of the restoration. According to the present work significant differences ($p < 0.05$) also were observed for considered mechanical properties within the bulk-fill composite category as different LCU. The mechanical properties of the bulk-fill composites are mostly lower when monowave LEDs were used. This results can be explain because these LCUs were unable to cure some photoinitiator like PPD and APO initiator systems present in RBCs (14).

Conclusions

Within the limitations of this study, the following conclusions can be drawn: Degree of conversion and microhardness values are affected by the different LCU and type of bulk fill resin composite. Photopolymerization with polywave LED (Bluephase G2 and VALO) and QHT (Optilux 501) may be more effective for mechanical properties of bulk-fill composites evaluated.

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Appendice

Table 1: Technical Profiles of Bulk-Fill Composites Evaluated

Material	Abbreviation	Shade	Composition	Filler Loading (% by Volume)	Recommended Curing Time and Light Intensity	Manufacturer

Tetric N-Ceram Bulk Fill	TNB	A2	Monomer: Dimethacrylates Filler: barium glass, ytterbium trifluoride, mixed oxide and copolymers	80%	20 s for ≥ 500 mW/cm2 or 10 s for ≥ 1000 mW/cm2	Ivoclar Vivadent, Schaan, Liechtenstein
Filtek Bulk Fill Posterior	FBF	A2	Bis-GMA, Bis EMA, UDMA, TEGDMA, Procrylat resins Filler: Zirconia/silica, ytterbium trifluoride	76.5%	40 s	3M ESPE, St Paul, MN, USA
Opus Bulk Fill	OBF	A2	Urethane-dimetacrylic monomers Filler: silicon dioxide	79%	40 s for ≤ 1000 mW/cm2 or 30 s ≥ 1000 mW/cm2	FGM, Joinvile, SC, Brazil

Table 2: Technical Profile of Curing Lights and Modes Evaluated			
LCU	Type	Irradiance*/Recommended Curing time*	Manufacturer
Bluephase	LED 3rd generation Polywave	High mode: ~1200 mW/cm2 10%, 20 s	Ivoclar Vivadent, Schaan, Liechtenstein

Valo	LED 3rd generation Polywave	High-power mode: ~1400 mW/cm2 10%, 12 s	Ultradent Products Inc, South Jordan, UT, USA
Optilux 501	QTH	: ~600 mW/cm2	Kerr, Orange, CA, USA
Radii cal	LED/monowave	: ~600 mW/cm2	SDI, Basywater, Victoria, Australia
Emitter C	LED/monowave	: ~600 mW/cm2	Schuster, Santa Maria

Table 3: Means (standard deviation) degree of conversion of composites at different LCU

Material	LCU				
	Bluephase	Valo	Optilux	Radicall	Emitter
OBF	70.9 (4.59)Aa	68.84 (6.18) Ba	68.03 (4.80) ABa	60.42 (2.14) Ca	61.14 (2.50) Ca
TNB	70.49 (4.28) Aa	62.61 (2.67) Ba	67.49 (4.78) ABa	59.82 (2.20) Ca	60.90 (4.34) Ca
FBF	65.96 (3.51) Ab	63.32 (2.23) Bb	62.19 (4.53)ABb	58.35 (6.63) Cb	54.22 (8.76) Cb

Mean and standard deviation of the DC (%) according to type of LCUs and RBCs. Capital letters indicates difference signficant of LCUs and lowercase indicates difference significant of RBCs, determinate by ANOVA and Tukey tests.

Table 4: Means (standard deviation) microhardness (KHN) of composites at different LCU

Material	LCU				
	Bluephase	Valo	Optilux	Radicall	Emitter
OBF	53.2 (2.19) Aa	51.9 (2.14)	51.9 (2.16)	51.0 (2.86)	51.2 (2.22)

		Aa	Aa	Aa	Aa
TNB	54.0 (1.91) Aa	47.5 (0.58) Bb	51.4 (1.91) Aa	47.5 (0.84) Bb	47.5 (1.14) Bb
FBF	52.1 (1.84) Aa	51.6 (0.71) ABa	51.3 (1.18) ABa	48.7 (0.56) Bab	49.5 (1.78) ABab

Mean and standard deviation of the KHN (Mpa) according to type of LCUs and RBCs. Capital letters indicates difference signficant of LCUs and lowercase indicates difference significant of RBCs, determinate by ANOVA and Tukey tests.

Table 5: Means (standard deviation) diametral tensile Strength (DTS), of composites at different LCU					
Material	LCU				
	Bluephase	Valo	Optilux	Radicall	Emitter
OBF	38.35 (4.33) Aa	36.87 (5.74) ABa	32.49 (2.61) ABa	35.82 (5.44) Ba	33.3 (4.35) Ba
TNB	39.70 (6.49) Aa	29.4 (6.6) ABa	34.5 (3.61) ABa	28.78 (5.59) Ba	28.2 (4.92) Ba
FBF	37.67 (2.79) Aa	38.9 (7.49) ABa	32.9 (6.95) ABa	30.25 (4.55) Ba	34.87 (4.64) Ba

Mean and standard deviation of the DTS (Mpa) according to type of LCUs and RBCs. Capital letters indicates difference signficant of LCUs and lowercase indicates difference significant of RBCs, determinate by ANOVA and Tukey tests.