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Orientador: Prof. Dr. Luís Henrique Araújo Raposo

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Prof. Dr. Luís Henrique Araújo Raposo
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Ata da defesa de DISSERTAÇÃO DE MESTRADO junto ao Programa de Pós-graduação em Odontologia, Faculdade de Odontologia da Universidade Federal de Uberlândia.

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As **nove** horas do dia **vinte e oito de fevereiro do ano de 2018** no Anfiteatro do Bloco 4L, Campus Umuarama da Universidade Federal de Uberlândia, reuniu-se a Banca Examinadora, designada pelo Colegiado do Programa de Pós-graduação em janeiro 2018 , assim composta: Professores Doutores: Paulo Vinícius Soares (UFU); Hugo Lemes Carlo (UFJF); e Luis Henrique Araújo Raposo (UFU) orientador(a) do(a) candidato(a) **Michele Ramos de Azevedo.** Ressalta-se que o Prof. Dr. Hugo participou da defesa por meio de Videoconferência na cidade de Governador Valadares-MG e os demais membros da banca e o aluno(a) participaram *in loco*.

Iniciando os trabalhos o(a) presidente da mesa Dr. Luis Henrique Araújo Raposo apresentou a Comissão Examinadora e o candidato(a), agradeceu a presença do público, e concedeu ao Discente a palavra para a exposição do seu trabalho. A duração da apresentação do Discente e o tempo de argüição e resposta foram conforme as normas do Programa.

A seguir o senhor (a) presidente concedeu a palavra, pela ordem sucessivamente, aos (às) examinadores (as), que passaram a argüir o (a) candidato (a). Após a argüição, que se desenvolveu dentro dos termos regimentais, a Banca, em sessão secreta, atribuiu os conceitos finais.

Em face do resultado obtido, a Banca Examinadora considerou o (a) candidato(a) ___provado(a).

Esta defesa de Dissertação de Mestrado Acadêmico é parte dos requisitos necessários à obtenção do título de Mestre. O competente diploma será expedido após cumprimento dos demais requisitos, conforme as normas do Programa, a legislação pertinente e a regulamentação interna da UFU.

Nada mais havendo a tratar foram encerrados os trabalhos às 12 horas e 02 minutos. Foi lavrada a presente ata que após lida e achada conforme foi assinada pela Banca Examinadora.

Participou por meio de Videoconferência

Prof. Dr. Paulo Vinícius Soares (UFU)

Prof. Dr. Hugo Lemes Carlo (UFJF)

Prof. Dr.Luis Henrique Araújo Raposo UFU

DEDICATÓRIA

*Dedico este trabalho à **minha família** que, com tanto esforço, me deu todas as condições necessárias para a conclusão de mais uma etapa em minha vida.*

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“ Quer ir mais rápido, vá sozinho. Quer ir mais longe, vá acompanhado”.

(Provérbio Africano)

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RESUMO

Objetivos: Este estudo objetivou avaliar a influência do nível de bateria de uma unidade de LED de amplo espectro na transmitância e grau de conversão de um cimento resinoso fotoativado, de fotoiniciadores alternativos, sob diferentes espessuras e translucidez cerâmicas, reforçadas por de dissilicato de lítio. Os comprimentos de onda fornecidos pela unidade de LED sob as diferentes condições também foram determinados. **Materiais e métodos:** foram produzidos quarenta discos cerâmicos de dissilicato de lítio de alta e baixa translucidez (IPS e.max Press HT e LT, cor A1), com 12 mm de diâmetro e espessuras variáveis (0,5 mm, 1,0 mm, 1,5 mm e 2,0 mm), divididos em grupos experimentais de acordo com a espessura e translucidez cerâmica (n = 5). Foram obtidas amostras de cimento resinoso (Variolink Esthetic LC, Neutral), com 3,9 mm de diâmetro e 1,0 mm de espessura para cada espessura e translucidez cerâmica em diferentes níveis de bateria (100%, 50%, 10%) da unidade de LED de amplo espectro (BluePhase G2). A análise da transmitância (T) e comprimentos de onda (C) entregues pela unidade LED nas diferentes condições foram determinadas usando um espectrômetro USB4000 referenciado pelo NIST, MARC (Bluelight Analytics Inc., Halifax, Canadá). Após armazenamento de 24 h, o grau de conversão (GC) dos espécimes de cimento resinoso fotoativados sob as diferentes condições foi avaliado em FTIR. Após verificada a homocedasticidade, os dados foram submetidos à análise de variância three-way, seguido do teste Tukey HSD ($\alpha = 0{,}05$). **Resultados:** Nenhuma interação significativa foi detectada para o nível de bateria x espessura nas amostras HT ($P = 0{,}265$), apenas nas LT ($P < 0{,}001$). Diferenças significantes para transmitância foram observadas nas amostras HT entre níveis de bateria 100% e 10% ($P < 0{,}001$), mas não entre os níveis 100% e 50% ($P = 0{,}085$) e 50% e 10% ($P = 0.170$). As diferentes espessuras cerâmicas produziram diferenças significantes para amostras HT ($P < 0{,}001$). Nos grupos LT foram observadas diferenças entre os níveis de bateria 100% e 10% ($P < 0{,}001$), 50% e 10% ($P < 0{,}001$), mas não para 100% e 50% ($P = 0{,}994$). A transmitância entre os níveis de bateria não apresentou diferenças nos grupos LT 2.0 mm ($P = 0{,}125$), 1,5 mm

(P = 0,326) e 1,0 mm (P = 0,007), mas os valores do grupo LT /0,5 mm foram significativamente diferentes (P <0,001). Foram detectadas diferenças para todas as espessuras de LT avaliadas nos diferentes níveis de bateria (P <0,001). Nos níveis de bateria de 10%, o grupo LT 0,5 mm apresentou transmitância semelhante ao grupo LT 1,0mm (P = 0,599). Os valores de GC foram significativamente diferentes entre os níveis de bateria (P = 0,002), mas não foram encontradas diferenças no GC entre as diferentes espessuras cerâmicas (P = 0,328) ou entre os níveis de translucidez (P = 0,650). **Conclusões:** níveis mais baixos de bateria influenciaram a transmitância de luz da unidade de LED de amplo espectro e, consequentemente, o grau de conversão do cimento resinoso nas diferentes espessuras/ translucidez cerâmicas. A quantidade de luz transmitida através de restaurações cerâmicas pode ser gradualmente reduzida com aumento de espessura / translucidez, independentemente dos níveis de bateria da unidade LED.

Palavras-chave: unidades de LED; amplo espectro; cerâmicas; cimento resinoso; transmitância; grau de conversão.

ABSTRACT

Objectives: This study aimed to evaluate the influence of battery level of a broad spectrum LED unit on transmittance and degree of conversion of a light-cure resin cement with alternative photoinitiators activated under different lithium disilicate ceramic thicknesses and translucency. The wavelenghts delivered by the LED curing unit under the different conditions were also determinated. **Methods and Materials:** Forty lithium disilicate ceramic discs of high and low translucency (IPS e.max Press HT and LT, shade A1), 12 mm in diameter with variable thickness (0.5 mm, 1.0 mm, 1.5mm, and 2.0 mm) were produced and divided into the experimental groups according to the ceramic thickness and translucency (n=5). Resin cement specimens (Variolink Esthetic LC, Neutral), with 3,9 mm of diameter and 1,0mm of thickness, were obtained for each ceramic thickness and translucency under the different battery levels (100%, 50%, 10%) of the broad spectrum LED curing unit (BluePhase G2). The analysis of the transmittance (T) and wavelengths (W) delivered by the LED unit at the different conditions were determined using a USB4000 spectrometer referenced by NIST, MARC (Bluelight Analytics Inc., Halifax, Canada). After storing specimens for 24 h, the degree of conversion (DC) of the resin cement photoactivated under the different conditions was evaluated using FTIR. After checking for homocedasticity, data were submitted to three-way analysis of variance, followed by Tukey HSD test (α=0.05). **Results:** No significant interaction was detected for battery level x thickness at HT translucency (P = 0.265), but at LT translucency it was significant (P <0.001). Significant differences were found for transmittance on HT specimens between 100% and 10% battery levels (P <0.001), but it was not significant between 100% and 50% (P = 0.085) and 50% and 10% (P = 0.170) battery levels. Significant differences among the different ceramic thickness were verified for HT specimens (P <0.001). Differences were observed on LT specimens between 100% and 10% (P <0,001), 50% and 10% (P <0,001), but not for 100% and 50% (P = 0,994). Transmittance values among battery levels was not significantly different within LT 2.0 mm (P = 0.125), 1.5 mm (P = 0.326), and 1.0 mm (P = 0.007) groups, but for the values of LT 0.5 mm group were significantly different

(P <0.001). Significant differences were detected for all LT thickness evaluated within 100% (P <0,001), 50% (P <0,001), 10% (P <0,001) battery levels. At 10% battery level, the LT 0,5 mm group showed similar transmittance to LT 1,0mm group (P= 0,599). The DC values were significantly different among battery levels (P = 0,002), but no significant differences were found for DC among the different ceramic thickness (P = 0,328) neither among the translucency (P = 0,650). **Conclusions:** Lower battery levels influenced the broad spectrum LED unit transmittance and consequently the degree of conversion of the resin cement in the different ceramic thicknesses and translucency. The amount of light transmitted through ceramic restorations can be gradually reduced by ceramic restorations with increased thickness/ translucency, irrespective of the LED unit battery levels.

Keywords: LED units; broad spectrum; ceramics; resin cement; transmittance; degree of conversion.

INTRODUÇÃO E REFERENCIAL TEÓRICO

As propriedades físicas e mecânicas dos compósitos restauradores são diretamente relacionadas ao espectro de luz e irradiância emitidos e transmitidos pelos aparelhos fotopolimerizadores utilizados e a quantidade máxima de energia absorvida pelos fotoiniciadores em determinado comprimento de onda, através das restaurações (Neumann et al., 2006; Schroeder & Vallo, 2007).

As cerâmicas reforçadas por dissilicato de lítio, possuem cerca de 60% de matriz cristalina o que possibilitou equilibrar as propriedades mecânicas e estéticas desse material, uma vez que o índice de refração dos cristais de dissilicato de lítio é similar ao da matriz vítrea. As cerâmicas reforçadas por dissilicato de lítio são indicadas na confecção de coroas totais anteriores e posteriores, além de próteses fixas de três elementos (Conrad et al., 2007; Perdigao, 2007).

Na prática clínica, a fotoativação dos cimentos resinosos utilizados na fixação de restaurações protéticas, é realizada indiretamente, através de materiais restauradores, sejam eles cerâmicos ou resinosos. O aumento na espessura desses materiais restauradores promove maior dispersão e absorção da luz no interior do mesmo, atenuando a energia incidente nos cimentos resinosos (Pacheco, 2013; Price & Felix, 2009). Cimentos resinosos fotoativados apresentam ótima estabilidade de cor a longo prazo (Sensi & Webley, 2007), porém sua indicação está limitada às restaurações que permitam a passagem de luz, como facetas de cerâmica e peças protéticas com menos de 2 mm de espessura e baixa opacidade (el-Mowafy et al., 1999).

As propriedades físicas e mecânicas dos cimentos resinosos são influenciadas pelo grau de conversão desses cimentos, que por sua vez, depende da quantidade de energia recebida durante o processo de fotoativação (Rueggeberg et al., 1994; Halvorson et al., 2003).Uma quantidade inadequada de radiação emitida pelas unidades fotoativadoras, pode influenciar negativamente o processo de polimerização dos cimentos resinosos fotoativados ou de dupla ativação, podendo prejudicar as propriedades mecânicas e biológicas desses materiais (Koch *et* al., 2007; Valentino *et* al., 2010).

Esses cimentos resinosos em sua maioria são a base de fotoiniciadores que dependem de aminas terciárias em sua reação de polimerização, como a canforoquinona. A canforoquinona é um fotoiniciador de conhecidos efeitos estéticos negativos nos procedimentos restauradores devido ao amarelamento das restaurações, em consequência da presença das aminas terciárias. Fotoiniciadores alternativos tem sido desenvolvidos e incorporados a diversos materiais fixadores de presa física, por exemplo, BAPO, Lucerina TPO, Ivocerina, entre outros (Albuquerque et al., 2008). Esses fotoiniciadores alternativos são sensíveis a baixos comprimentos de onda, como por exemplo a luz violeta, e não somente a luz azul como a canforoquinona. Para isso, foram desenvolvidas as unidades de LED fotoativadoras de amplo espectro que possibilitam a polimerização compósitos resinosos que contenham fotoiniciadores diferentes da canforoquinona (Mozner et al., 2008; Sampaio et al., 2017).

Esses LEDs têm em seu diferencial a inclusão de um chip que emite luz no comprimento de onda da luz violeta em disposição com outras luzes no comprimento de onda da luz azul (Price et al., 2010a; Price et al., 2010b). Também foram desenvolvidos aparelhos de LED onde pode ser incluídos dois tipos diferentes de LEDs de luz azul (diferentes comprimentos de onda) e um LED de luz violeta em um único aparelho para que o espectro de luz atinja a maioria dos fotoiniciadores utilizados atualmente na odontologia (Rueggeberg et al., 2010).

Por esse motivo, é possível que as propriedades dos cimentos resinosos também possam ser influenciadas pela menor intensidade de luz produzida por unidades fotoativadoras com baixo nível de bateria e pela interposição de restaurações cerâmicas indiretas com diferentes espessuras e opacidades. Dessa forma, o presente estudo procurou investigar a influência desses fatores na relação entre materiais restauradores e cimentantes.

PROPOSIÇÃO

O objetivo geral do presente estudo foi avaliar a influência do nível de bateria de uma unidade de LED de amplo espectro na transmitância e no grau de conversão de um cimento resinoso fotoativado com fotoiniciadores alternativos, ativados sob diferentes espessuras e translucidez de cerâmica reforçada por de dissilicato de lítio.

Os objetivos específicos foram:

Avaliar a transmitância de um aparelho de LED fotoativador de amplo espectro em diferentes níveis de bateria (100%, 50% e 10%), através de material restaurador cerâmico indireto, reforçado por dissilicato de lítio, em diferentes espessuras (0,5mm; 1,0mm; 1,5mm; 2,0mm) e translucidez (HT e LT) – ambos de cor A1;

Analisar os comprimentos de onda específicos (violeta e azul) produzidos pelos diferentes níveis de bateria do LED utilizado nesse estudo através de diferentes níveis de espessura e translucidez da cerâmica;

Determinar o grau de conversão do cimento resinoso utilizado, em diferentes níveis de bateria, espessuras e translucidez.

CAPÍTULO 1

Influence of broad spectrum LED unit battery level on transmittance and degree of conversion of photoactivated resin cement under different ceramic thicknesses / translucency

Influence of broad spectrum LED battery on light transmission and degree of conversion of resin cement in different ceramic restorations.

Authors: Azevedo MR, Borella PS, Bruno L, Soares PV, Soares CJ, Raposo LHA

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CLINICAL RELEVANCE

The light intensity delivered by LED units can be affected by several factors, influencing the mechanical and biological properties of polymeric materials as light-cure resin cements activated under ceramics, what may compromise the performance and longevity of indirect restorations. Light cure resin cements with alternative photoinitiators can be even more affected, since they depend on broad-spectrum LED units to be activated by the violet light spectrum, which can be potentially blocked by thicker/opaque indirect restorations.

SUMMARY

Objectives: This study aimed to evaluate the influence of battery level of a broad spectrum LED unit on transmittance and degree of conversion of a light-cure resin cement with alternative photoinitiators activated under different lithium disilicate ceramic thicknesses and translucency. The wavelenghts delivered by the LED curing unit under the different conditions were also determinated. **Methods and Materials:** Forty lithium disilicate ceramic discs of high and low translucency (IPS e.max Press HT and LT, shade A1), 12 mm in diameter with variable thickness (0.5 mm, 1.0 mm, 1.5mm, and 2.0 mm) were produced and divided into the experimental groups according to the ceramic thickness and translucency (n=5). Resin cement specimens (Variolink Esthetic LC, Neutral), with 3,9 mm of diameter and 1,0mm of thickness, were obtained for each ceramic thickness and translucency under the different battery levels (100%, 50%, 10%) of the broad spectrum LED curing unit (BluePhase G2). The analysis of the transmittance (T) and wavelengths (W) delivered by the LED unit at the different conditions were determined using a USB4000 spectrometer referenced by NIST, MARC (Bluelight Analytics Inc., Halifax, Canada). After storing specimens for 24 h, the degree of conversion (DC) of the resin cement photoactivated under the different conditions was evaluated using FTIR. After checking for homocedasticity, data were submitted to three-way analysis of variance, followed by Tukey HSD test (α=0.05). **Results:** No significant interaction was detected for battery level x thickness at HT translucency (P = 0.265), but at LT translucency it was significant (P <0.001). Significant differences were found for transmittance on HT specimens between 100% and 10% battery levels (P <0.001), but it was not significant between 100% and 50% (P = 0.085) and 50% and 10% (P = 0.170) battery levels. Significant differences among the different ceramic thickness were verified for HT specimens (P <0.001). Differences were observed on LT specimens between 100% and 10% (P <0,001), 50% and 10% (P <0,001), but not for 100% and 50% (P = 0,994). Transmittance values among battery levels was not significantly different within LT 2.0 mm (P = 0.125), 1.5 mm (P = 0.326), and 1.0 mm (P = 0.007) groups, but for the values of LT 0.5 mm group were significantly different (P <0.001). Significant differences were detected for all LT thickness evaluated within 100% (P <0,001), 50% (P <0,001), 10% (P <0,001) battery levels. At 10% battery level, the LT 0,5 mm group showed similar transmittance to LT 1,0mm group (P= 0,599). The DC values were significantly different among battery levels (P = 0,002), but no significant differences were found for DC among the different ceramic thickness (P = 0,328) neither among the translucency (P = 0,650). **Conclusions:** Lower battery levels influenced the broad spectrum LED unit transmittance and consequently the degree of conversion of the resin cement in the different ceramic thicknesses and translucency. The amount of light transmitted through ceramic restorations can be gradually reduced by ceramic restorations with increased thickness/ translucency, irrespective of the LED unit battery levels.

Keywords: LED units; broad spectrum; ceramics; resin cement; transmittance; degree of conversion.

INTRODUCTION

The bonding capacity of contemporary resin based cements allow to partially prevent the diffusion and propagation of cracks in the internal surfaces of ceramics, making these materials well accepted for luting ceramic restorations[1,2]. Besides presenting lower solubility, the resin cements have superior physical, mechanical and aesthetic properties compared to conventional cements, providing effective bonding at the tooth/restoration interface[1,2]. However, resin cements are dependent on adequate polymerization, and particularly for the light-cure cements, light intensity capable of generating sufficient energy at the appropriate wavelengths is required to activate and promote adequate conversion degree, so that optimal physical properties can be achieved in the restorative procedure[3,4].

Irradiance (mW / cm²) is one of the most important factor for the photoactivation process, even as the exposure time of the material to light. Irradiance can be defined as the useful power of a polymerization unit in watts (W) by the tip area of the unit (cm^2)[5,6]. An insufficient irradiance affect the properties of the material, such as the degree of conversion, allowing wears and fracture at the margins, reduction of hardness and modulus of elasticity, and increased toxicity[7,8]. The amount of the light and the emitted wavelength depend directly on the type of photoactivating unit used[9].

The values of light passing through a restoring material are the transmittance of the light curing unit, that is the amount of light passing through the restorative material, compared to the initial irradiance value provided. The amount of light that can not pass trough the material is the absorbance of the restorative material. The absorbance can be defined as the inverse of the of the transmittance, thus, that the higher the transmittance of a material at a certain wavelength, the lower the absorbance at the same wavelength[10].

In the case of photocatalytic resin cements, the adsorption of the cationic surfactants can be detrimental to the mechanical and biological properties of these materials[11]. The most common photoinitiator used in resin cements is camphorquinone, but, since this component is yellow colored and might lead to undesirable yellowing of thin ceramics restorations after cure[12], several manufacturers started to use alternative photoinitiators such as PPD, BAPO, Lucirin TPO, Ivocerin, and others[13]. This photoinitiators exclude the need for tertiary amines which interfere with the final staining of the restorations. These photoinitiators are more reactive at lower wavelength spectra (320nm - 410nm) and reduced penetration capacity compared to the reactive light spectrum of camphoroquinone (468nm).

Additionally, several other factors can influence the proper polymerization of light-cure resin cements, such as composition, filler particles and their refractive index, which can alter the light transmission[14,15], and the distance of the light output from the composite material. In addition, the curing unit used, as well as the shape of the tip have been suggested as a significant factors, which can influence the polymerization of resin composite materials, considering irradiance, total energy and the wavelengths[16]. The influence of these factors on light-cure resin cements can be intensified during indirect restorative procedures due to the differences in translucency, thickness and color of the restorative materials. The thicker, opaque and saturated the indirect restorations, the higher will be the hinder to light passage, what may affect the polymerization of the resin luting agent[17].

Currently, the most used light curing units are based on light-emitting diodes (LED), most of which are cordless, powered by lithium-ion (Li-Ion) or lithium-polymer (LiPo) batteries that must be periodically recharged[18]. However, little is known about the relationship between the battery charge and the light intensity emitted by these units[19]. Recently, a study has shown reduced degree of conversion and decreased mechanical properties for a nanofilled composite resin when using a cordless LED unit with low battery level, due to the reduced intensity of the light output[20]. Nowadays, monowave cordless LED units that emit blue light spectrum only are most commonly used in dental practice, but with the increased use of alternative photoinitiators in resin composite materials by the dental industry, polywave or broad-spectrum LED units, which emit light in blue and violet spectra, may be required for adequate activation of these products[21]. Given the indispensable role of light sources in the activation and polymerization process of composite-based materials, the battery level of broad-spectrum cordless LED units may also influence the final luting of indirect restorations when using light-cure resin cements with alternative photoinitiators, since the energy generated by the light curing units is one of the most important factors for the success of the restorative procedure[14,15]. For this reason, that the properties of light-cure resin cements with alternative photoinitiators can also be influenced by the lower intensity of light produced by light curing units with low battery level, mainly when thicker/opaque indirect restorations are used.

Consequently, it is necessary to investigate how the characteristics of ceramic restorations and different battery levels of cordless LED units can influence the properties of light-cure resin cements, in order to better understand the relationship between the restorative and luting materials under different light emitting conditions. Thus, the aim of the presented study was to evaluate how the influence of the battery level of a broad-spectrum cordless LED unit can influence on the transmittance and irradiance of the light and on the degree of conversion of a light cured cement under different lithium disilicate ceramic thicknesses and translucency. Two null hypotheses were tested: 1) the different ceramic thicknesses and translucency would not influence the transmittance of the LED unit or the degree of conversion of the resin cement; 2) the different battery levels of the LED unit would not influence the transmittance of the resin cement degree of conversion.

METHODS AND MATERIALS

Ceramic specimens

Initially, discs patterns 12 mm in diameter with variable thickness (0.5 mm, 1.0 mm, 1.5mm, 2.0mm) were made using autopolymerizing polymethylmethacrylate resin (Dencrilay, Dencril, Pirassununga, SP) poured in silicon matrices. The resulting discs were submitted to finishing procedures and then included in modified phosphate-based investment (IPS Empress Speed, Ivoclar Vivadent). After removing the resin patterns in a proper furnace (3000P, EDG, São Carlos, SP, Brazil) at 850 °C for 1 h, a ceramic ingot associated to a plunger were inserted in the investment cylinder and transferred to the pressing furnace (EP 3010, Ivoclar Vivadent) were pressing was performed according to manufacturer´s directions. Subsequently, the ceramic disks were disinvested using 100 μm glass microspheres at 4 bar pressure, followed by cleaning in running water and drying with air. After disinvestment, the discs were finished using #600 grit silicon carbide papers, followed by ultrasonic cleaning for 10 min.

Forty lithium disilicate ceramic discs of high and low translucency (IPS e.max Press HT and LT, A1, Ivoclar Vivadent, Schaan, Liechtenstein), 12 mm diameter with variable thickness were selected and divided into the experimental groups according to the ceramic thickness and translucency (n=5).

Transmittance (T) and Wavelengths (W)

Specimens from a light-cure resin cement with alternative photoinitiators (Variolink Esthetic LC, Neutral, Ivoclar Vivadent, Schaan, Liechtenstein) (n=5) were obtained using circular matrices (3.9mm diameter × 1.0mm thickness), under a neutral yellow light, for each ceramic thickness and translucency under the different battery levels (100%, 50%, 10%) of the broad-spectrum LED curing unit (BluePhase G2, Ivoclar Vivadent, Schaan, Liechtenstein), which was set in the high mode for 40 s. The analysis of the variation in the transmittance (T) delivered by the broad-spectrum LED unit was performed to a maximum distance of up to 1 mm using a USB4000 spectrometer referenced by NIST, MARC (Bluelight Analytics Inc., Halifax, Canada). The system uses a CC3-UV cosine corrector to collect the radiation in a 180 ° field of view, thus mitigating the effects of optical interference associated with sampling light collection. Irradiation was evaluated within the range of violet (360-420 nm) and blue wavelength (420-540 nm) and data was collected individually at a rate of 16 registers / s with sensor powered at 20 mW. The LED unit was centered above the circular sensor (Bottom) (Fig. 1) with a diameter of 3.9 mm, allowing obtaining the transmittance value considering this area of exposure.

Degree of Conversion (DC)

After storing the resin cement specimens in their respective matrices for 24 h at 37 °C in 100% relative humidity, the degree of conversion of the specimens photoactivated under the different conditions was evaluated using Fourier Transformed Infra-Red (FTIR) spectroscopic unit (Tensor 27, Bruker, Germany). The specimens were placed with the base directly over the diamond sensor at the center of the Golden Gate Attenuated Total Reflectance (ATR) platform. Unactivated portions of the resin cement were evaluated to determine the degree of conversion of the cement according to the following formula: % DC = 100 [1 - (Abs. (C=C)Abs (C=O) polymer / Abs(C=C)Ab(C=O) monomer)].

Statistical analysis

The data obtained were tabulated and submitted to normality and homogeneity analysis using Kolmogorov-Smirnov test. Three-way analysis of variance (ANOVA) was used to assess the effect of the battery levels of a broad-spectrum LED unit and different ceramic thicknesses/ translucency on the properties of the light-cure resin cement. Multiple comparisons were performed by the Tukey HSD test. All tests were conducted with a confidence level of 0.05% using a statistical software (SigmaPlot 12.0, 2011-2012).

RESULTS

Transmittance (T) and Wavelengths (W)

The Transmittance (T) values from each battery level X thickness across levels of translucency are reported in Table 1. Battery level x thickness analysis across levels of translucency showed the effect of the interaction depends on what level of translucency is present. No significant interaction was detected for battery level x thickness at HT translucency (P = 0.265), but at LT translucency it was significant (P <0.001). Significant differences were found for the transmittance on HT specimens between 100% and 10% battery levels (P <0.001), but it was not significant between 100% and 50% (P = 0.085) and 50% and 10% (P = 0.170) battery levels. Significant differences among the different ceramic thickness were verified for HT specimens (P <0.001). For 2,0mm HT specimens the transmittance values were lower ,than 1,5mm HT, 1,0mm HT and 0,5mm HT, the latter being the highest values of transmittance. Significant differences were observed for LT specimens between 100% and 10% (P <0,001), 50% and 10% (P <0,001), but not for 100% and 50% (P = 0,994). Transmittance values among battery levels was not significantly different within LT 2.0 mm (P = 0.125), 1.5 mm (P = 0.326), and 1.0 mm (P = 0.007) groups, but for the transmittance values of LT 0.5 mm group were significantly different (P <0.001). Significant differences were detected for all LT thickness evaluated within 100% (P <0,001), 50% (P <0,001), 10% (P <0,001) battery levels. At 10% battery level, the LT 0,5 mm group showed similar LED transmittance to LT 1,0mm group (P= 0,599).

Wavelengths spectra provided by MARC measurement of transmittance through the different ceramic thickness and translucency according the battery level are shown on Fig.2, Fig. 3 and Fig.4. Broad-spectrum LED unit used showed 16.6 J/cm2 radiant exposure in the 420–490 nm wavelength range with the remaining 3.4 J/cm2 radiant exposure being emitted over the wavelength range of 380–420 nm. The wavelength absorption peak to the light-cure resin cement used was between 440–490nm, and was reduced according to increase in thickness or translucency. This emission spectra match with the absorption spectra of the germanium based photoinitiator used on this study.

Degree of Conversion (DC)

The degree of conversion (DC) values from each battery levels X thickness across levels of translucency are reported in Table 2. The DC values were significantly different among battery levels (P = 0,002). For HT translucency 2,0mm specimens the mean DC numeric values were lower, than 1,5mm HT, 1,0mm HT and 0,5mm HT, respectively, with decrease of thickness. At LT translucency 2.0 mm also showed lowest numeric values of DC than 1.5 mm LT,1.0 mm LT, 0,5mm LT, after allowing for the effects of differences in thickness and translucency. But no significant differences were found for DC among the different ceramic thickness (P = 0,328) neither among the translucency (P = 0,650).

DISCUSSION

The null hypotheses evaluated in the present study were rejected. The different ceramic thicknesses and translucency as well as the different battery levels influenced the transmittance of the broad-spectrum LED unit and the conversion degree of the light-cure resin cement. This study analyzed how lithium disilicate restorations with different thickness (2.0 mm; 1.5 mm; 1.0 mm; 0.5 mm) and translucency (HT or LT) would affect the properties of a light-cure resin cement activated with a broad-spectrum LED unit with different battery levels (100%; 50%; 10%). As seen, the polymerization performance of the light-cure resin cement evaluated was influenced under these conditions, since the ceramic material absorbs a certain percentage of the emitted light, mainly in cases that restorations with increased thickness/ translucency are used. Thus, clinicians should guarantee an adequate activation of light-cure resin cements under ceramic restorations by using curing units capable to produce sufficient irradiance to allow light reaching the cement layer through indirect restorations, besides assuring that cordless LED units are charged with battery levels above 50%. The adequate polymerization of resin cements is an essential step for the success of indirect restorations[22,23].

The battery level x thickness showed no significant difference interaction at HT translucency level ($P = 0.265$), while for LT translucency there is a significant interaction correlation ($P < 0.001$). It probably, why LT translucency shows itself as a bulkhead greater than HT translucency, making light passing easier on all HT restorations despite increasing thicknesses. Furthermore, for HT level of translucency the battery level showed a considerably difference of irradiance when compared 100% and 10% ($P < 0.001$), but no statistic difference was found between 100% and 50% level ($P = 0.085$), or 50% and 10% ($P = 0.170$). Considering the high light intensity provided by the broad-spectrum LED unit used in this study, these results, shows that the difference in battery level of Bluephase G2 LED has to be considerable to make significant difference on transmittance. For LT level of translucency the battery levels only has influenced the transmittance throw the 0.5 mm thickness at 10% of battery level ($P<0.001$), despite this, his values, considering thickness level, were statically similar as 1,0mm. Then, no statistically, difference on transmittance was verified between 100%, 50% and 10% battery level, except for LT/10%/0.5mm group. These results show that low battery levels of the broad-spectrum LED unit evaluated, at LT groups, did not affected the properties of the light-cure resin cement used, once the amount of light delivered by the unit was not significantly compromised. This fact contradicts previous studies on light emission and restoration thickness results[19,24], but also corroborate with a studies where the thickness shows more intense effect than ceramic shades, also shades showed significantly differences for thicknesses above 2.0mm. In this study the highest thickness evaluated was 2.0mm[25 26].

It was also demonstrated that indirect restorations with increased thickness could significantly influence the amount of light passing throw the ceramic materials[10]. The irradiance emitted by the LED unit was significantly different among all different thickness evaluated for HT translucency ($P<0,001$), as well as for LT translucency ($P<0,001$), except between 0.5 and 1.0mm groups at 10% battery level ($P=0,599$). In addition, when the ceramic thickness is considered associated with different translucency and battery levels, it might produce a slight influence on the properties of the light-cure resin cements frequently used on clinical practice[20]. The difference on translucency of opaquer ceramics as LT can result in significant differences on transmittance between the translucency, once was proved that more translucent ceramics restorations has influence about the properties of resin cements due to the light transmittance[25,26].

The effect of ceramic interposition on the mechanical properties of resin cements has been previously investigated in other in vitro studies[27,28]. However, the results of the current study showed that not only the interposition of indirect materials, but also the thickness/ translucency of the ceramic restorations and the battery level of LED units can influence the irradiance emitted and consequently the degree of conversion of light-cure resin cements. The different battery levels caused differences on transmittance for both ceramic translucency (HT and LT), with significant differences between 100% and 50% levels for HT translucency, and between 50% and 10% level for LT translucency. The literature shows the degree of conversion of photoactivated resin cements is between 54-60%[29], corroborating with the results found in this study, where DC values remained between 53 - 60%. Despite these findings, no significant differences were found for the degree of conversion of the light-cure resin cement among the different ceramic thickness or translucency. This fact can be explained by the high light intensity provided by the broad-spectrum LED unit used. Besides that, a broad-spectrum LED curing light contain two or more different types of LED chips that produce different emission spectra[9,18,30], favoring the activation of light-cure resin cements with alternative photoinitiators as the one used in this study.

The resin-based materials that contain alternative photoinitiators have better performance when activated by broad spectrum LED lights, since this can significantly improve the polymerization and physical properties of these materials because blue and violet wavelengths are reached, exciting both the camphorquinone and other initiators [30]. The broad-spectrum LED unit used showed 420–490 nm wavelength range, being emitted over the wavelength range of 380–420 nm, the necessary wavelength range to activate alternative photoinitiators as present on the resin cement used this study. The present study showed that the wavelength absorption peak to the light-cure resin cement used was kept between 440–490nm. Despite the camphoroquinone (CQ) be the most common photoinitiator used at resin-based materials, different types of photoinitiators have been developed and used in luting resin cements to replace CQ/amine avoiding the yellowing effect. TPO and germanium-based initiator as Ivocerin (present on the resin cement used on this study), BTMGe and DBDEGe, are based on the photochemical cleavage of aldehydes and ketones into two or more free radical intermediates, which initiate polymerization without the need of an additional amine. Despite not affecting the color of the final restoration, these compounds are most reactive to lower light wavelengths (410 nm or 320 nm) as compared to camphorquinone (468 nm)[31]. Lower light wavelengths such as the violet spectrum (~320-440 nm) required for activation of some alternative photoinitiators are known to have reduced penetration capacity when comparing to blue spectrum (~440-540 nm) used for camphorquinone[21]. This fact may lead to poor deep of cure, mainly when trying to activate light-cure resin cements through indirect restorations[21], once the restorations presents a higher absorbance on this wavelengths, consequently, lower transmittance[10].

The emission spectra of the broad-spectrum LED unit used match with the absorption spectra of the germanium based photoinitiator as well as the absorption spectra of CQ. What means, the germanium-based photoinitiators are consequently absorbing light at a higher wavelength range than the acylphosphine oxides, and could be well activated by commercially available broad-spectrum LED units[32]. Beside this, many studies have shown that the distribution of the irradiance and emission spectrum throw the light tip of LED units can be highly inhomogeneous[19,33]. This fact, can result in some well cured areas, while other regions can be fairly cured, in the same restoration[33,34]. This fact shows how important is the beam profile at the tip of the LED light curing units[19,35].

The different restoration thickness evaluated in this study may represent an acceptable limit of light obstruction during clinical procedures. Significant differences at the properties of resin cements was demonstrated when ceramic thickness is superior to 3.0 mm, once exponential decrease on irradiance is observed with increased restoration thickness[32]. However, it also depends on the differences at the ceramic microstructure, for example, the lithium disilicate ceramic evaluated on this study, has elongated lithium disilicate ($Li_2Si_2O_5$) crystals and a lot of small interlocking needle-like crystals that are organized in a non-standardized way, characterizing a dense structure, maybe allowing more obstruction of light in lower thickness than leucite-reinforced ceramics[36]. A previous study verified that a ceramic with 1.0 mm thickness caused a reduction of approximately 50% in the emitted irradiance of a curing unit, but a corresponding reduction in the degree of conversion was not observed[26,32]. These findings are in accordance to the results of the present study, and a possible explanation is the base of the chain polymerization process, based on the methacrylate monomers present in dental composite resin.

Therefore, all the factors discussed on this study can affect the clinical success of ceramic restorations. Although the present study has its limitations once its an in vitro study. Other studies evaluating the hardness or modulus of elasticity of the materials nor its adhesive capacity over the proposed conditions should be developed. Therefore, further research is also needed to investigate other possible factors influencing light transmission through dental ceramic restorations.

CONCLUSION

- Low battery levels of a broad-spectrum LED unit can affect the transmittance and degree of conversion of a light cure resin cement activated through ceramic restorations.
- The amount of light passing the ceramic restorations can be impaired by increased thickness/ translucency.
- More translucent restorations allow higher light transmission through different ceramic thickness, compared to less translucent restorations.
- Clinicians should keep their LED light-curing units with the battery charge level above 50% in order to obtain efficient values of transmittance and sufficient energy in the polymerization process, ensuring a suitable degree of conversion to the resin materials.

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ANEXOS

Tables, ilustrations, graphs and figures.

	HT			LT		
TRANSMITTANCE (T)	***100%***	***50%***	***10%***	***100%***	***50%***	***10%***
2,0mm	**116,20 ±17,77Aa€**	**122,12 ±10,29ABa€**	**118,84 ±20,59Ba€**	**95,69 ±25,76Aa£**	**93,10 ±11,75Aa£**	**79,48 ±5,04Ba£**
1,5mm	**158,87 ±25,63Ab€**	**146,27 ±22,53ABb€**	**135,05 ±8,71Bb€**	**130,12 ±41,14Ab£**	**125,64 ±32,89Ab£**	**108,11 ±20,66Bb£**
1,0mm	**226,14 ±44,51Ac€**	**187,78 ±21,88ABc€**	**176,60 ±33,68Bc€**	**206,68 ±39,68Ac£**	**188,39 ±37,83Ac£**	**156,59 ±15,37Bc£**
0,5mm	**256,93 ±86,18Ad€**	**248,68 ±90,60ABd€**	**257,16 ±22,96Bd€**	**229,24 ±72,37Ad£**	**234,55 ±72,61Ad£**	**169,17 ±10,61Bc£**

* Different uppercase letters indicate significant differences among the battery levels for each translucency (horizontal); Different lowercase letters indicate significant differences among the ceramic thickness for each translucency (vertical); Different symbols indicate significant differences between the translucency; Tukey HDS test (α=.05%).

Table 1. Transmittance (mW/cm^2) and standard deviation (±) assessed under the different experimental conditions.

	HT			LT		
CONVERSION DEGREE (DC)	***100%***	***50%***	***10%***	***100%***	***50%***	***10%***
2,0mm	**54,71 ±1,91 Aa€**	**56,27 ±8,1 BCa€**	**55,95 ±2,37 Ca€**	**53,21 ±3,21 Aa€**	**53,77 ±2,87 BCa€**	**53,75 ±3,77 Ca€**
1,5mm	**57,27± 2,66 Aa€**	**53,83 ±8,32 BCa€**	**55,85 ±1,88 Ca€**	**56,65 ±1,59 Aa€**	**54,63 ±2,57 BCa€**	**55,59 ±3,73 Ca€**
1,0mm	**57,70± 1,79 Aa€**	**52,00 ±2,28 BCa€**	**55,78 ±1,94 Ca€**	**57,83 ±0,69 Aa€**	**52,37 ±2,41 BCa€**	**52,68 ±2,67 Ca€**
0,5mm	**57,03 ±4,46 Aa€**	**56,50 ±3,76 BCa€**	**50,20 ±2,51 Ca€**	**59,01 ± 7,79 Aa€**	**55,13 ±4,58 BCa€**	**50,13 ±3,46 Ca€**

* Different uppercase letters indicate significant differences among the battery levels for each translucency (horizontal); Different lowercase letters indicate significant differences among the ceramic thickness for each translucency (vertical); Different symbols indicate significant differences between the translucency; Tukey HDS test (α=.05%).

Table 2 - Degree of conversion (%) and standard deviation (±) assessed under the different experimental conditions.

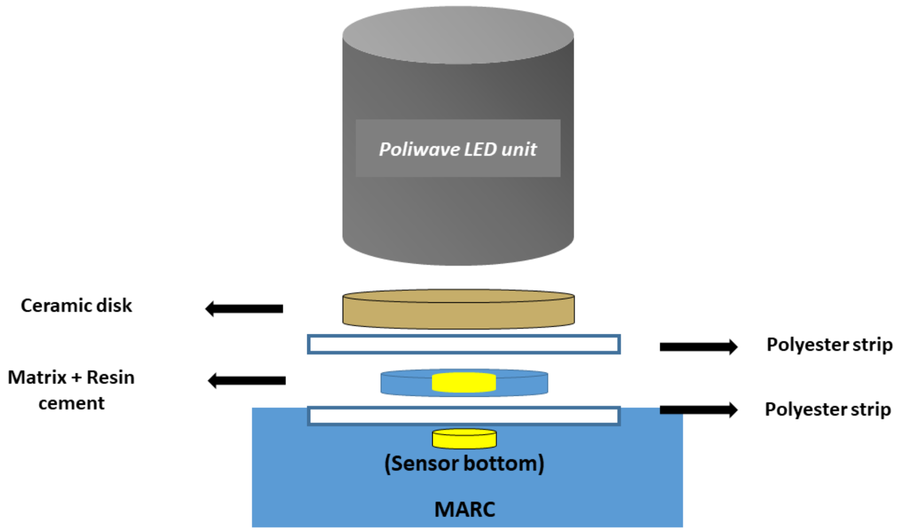


Figure. 1 - Schematic drawing of the photoactivation of the resin cement specimens during irradiance analysis.

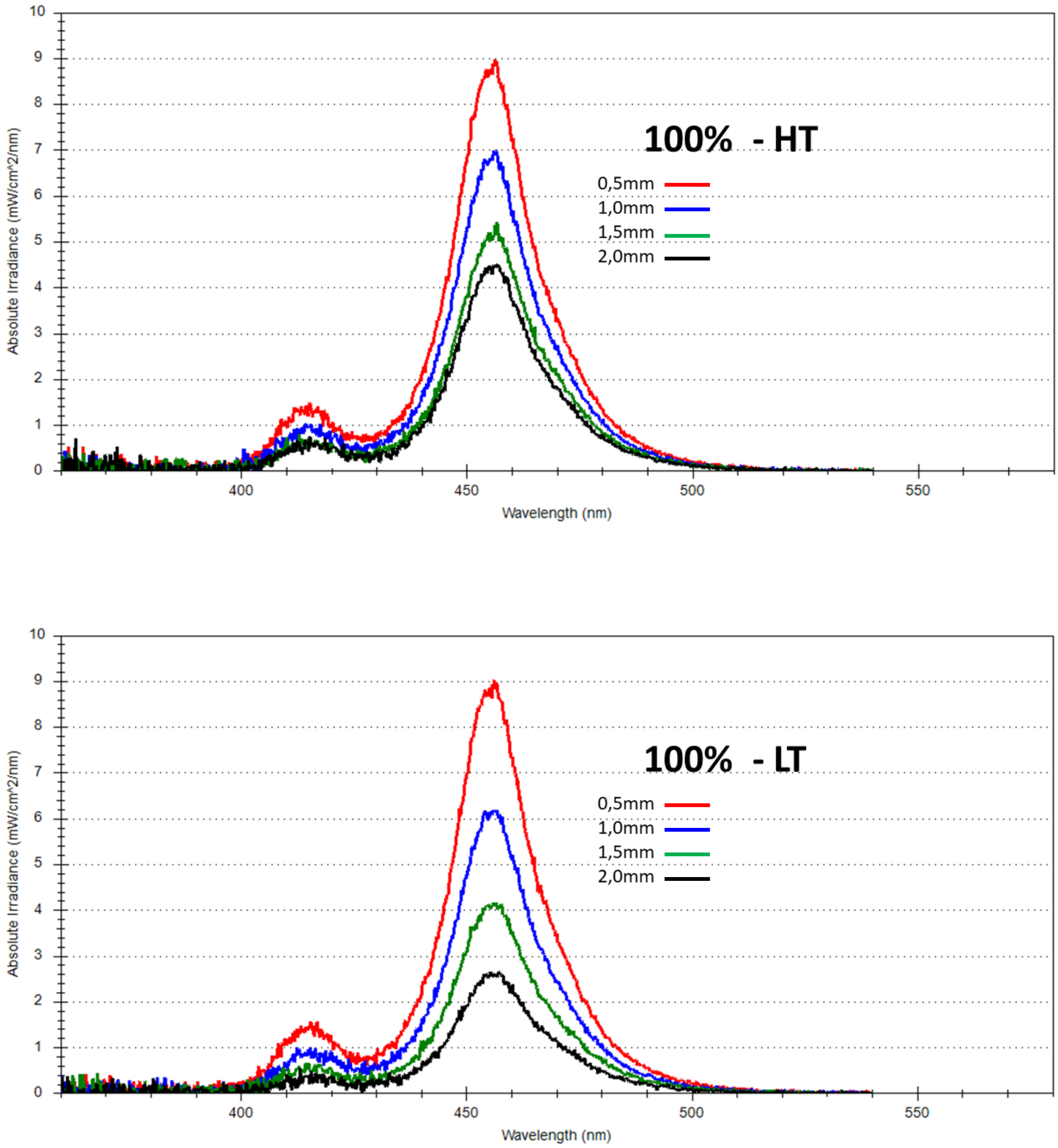
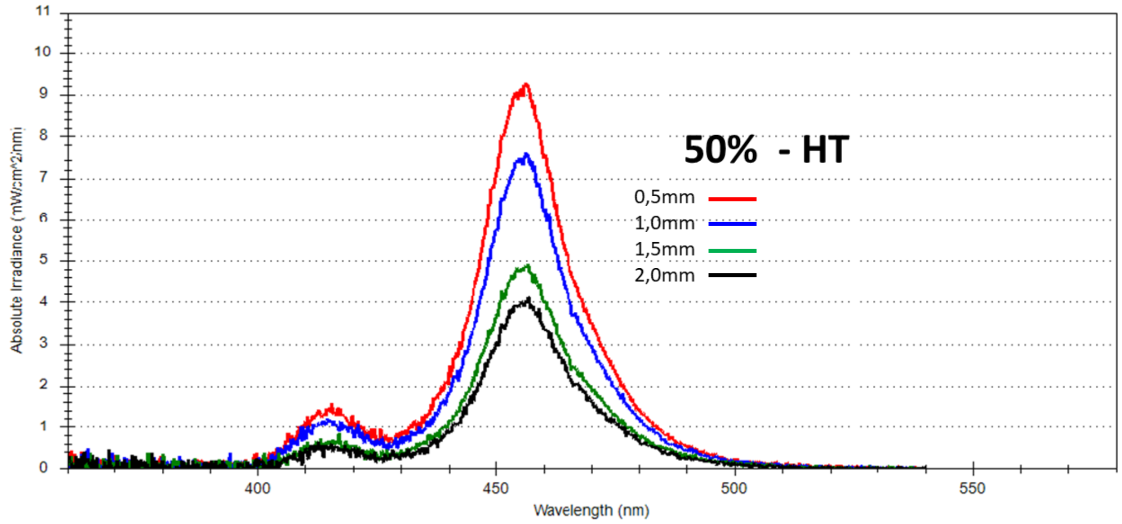


Figure. 2 - Absolute Irradiance ($mW/cm^2/nm$) x wavelength (nm): transmittance through the different ceramic thickness and translucency at 100% battery level.



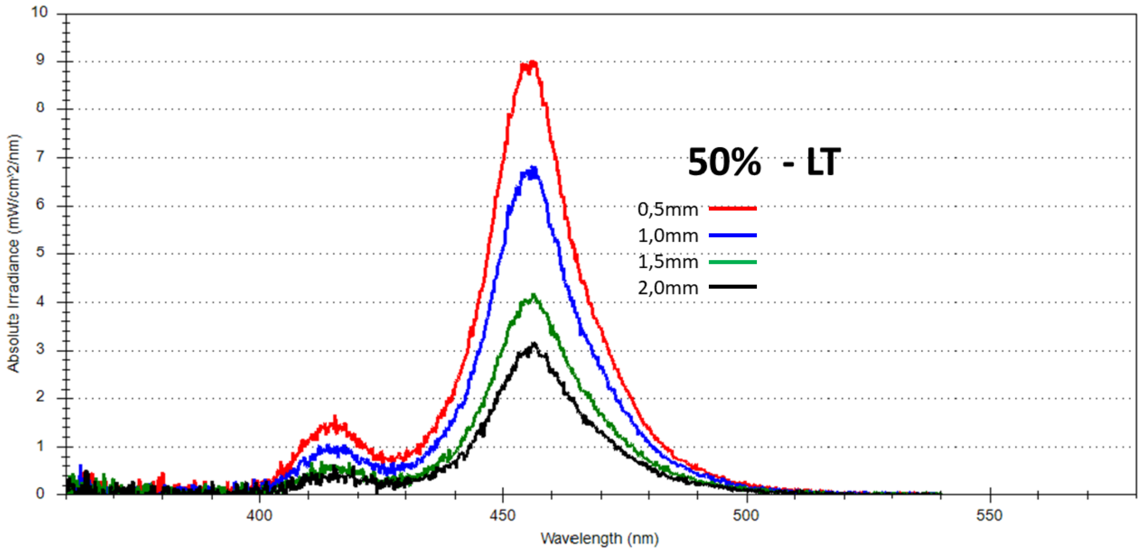
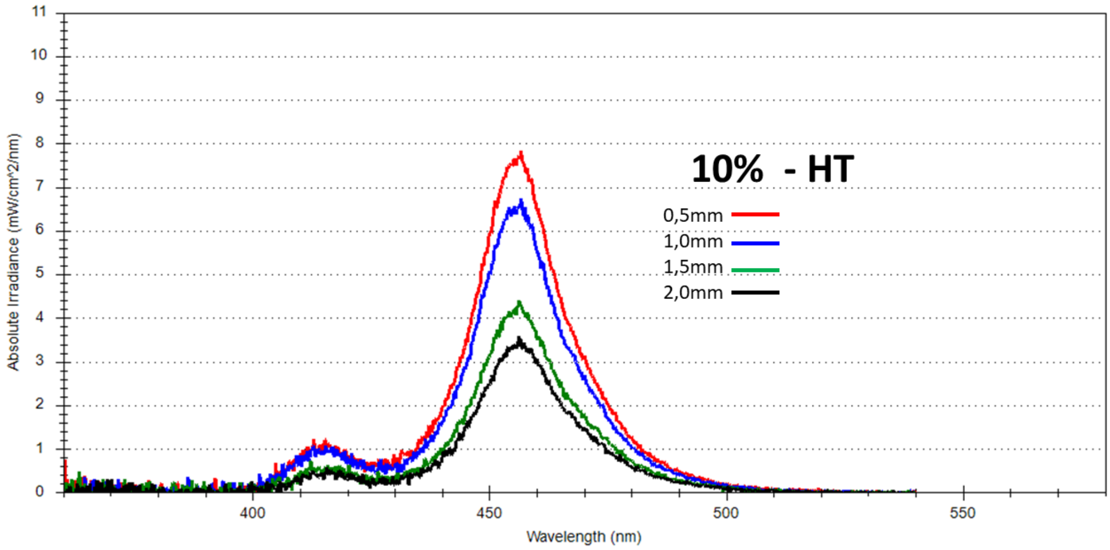


Figure. 3 - Absolute Irradiance (mW/cm^2/nm) x wavelength (nm): transmittance through the different ceramic thickness and translucency at 50% battery level.



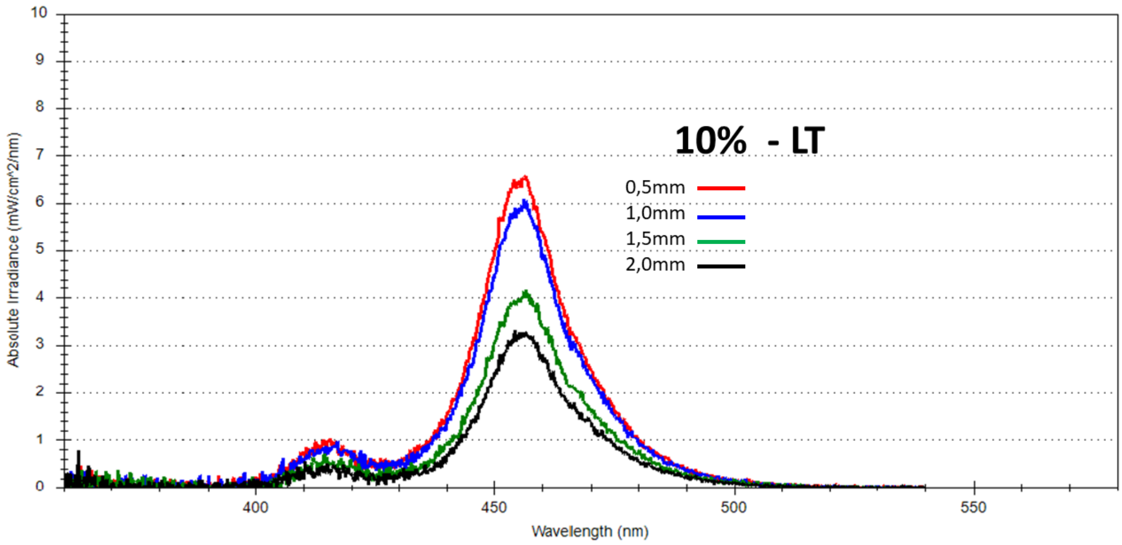


Figure. 4 - Absolute Irradiance ($mW/cm^2/nm$) x wavelength (nm): transmittance through the different ceramic thickness and translucency at 10% battery level.

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