

Stella Sueli Lourenço Braga

**Fontes de Luz em Odontologia –
Caracterização e desempenho na ativação
de resinas compostas e cimentos resinosos**

*Light-Curing Units in Dentistry – Characterization
and performance in the activation of resin
composites and resin cements*

Tese apresentada à Faculdade de
Odontologia da Universidade Federal de
Uberlândia, para obtenção do Título de
Doutor em Odontologia na Área de
Clínica Odontológica Integrada.

Uberlândia, 2021

Stella Sueli Lourenço Braga

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Odontológica Integrada.

Orientador: Prof. Dr. Carlos José Soares

Coorientador: Prof. Dr. Richard Bengt Price

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Uberlândia, 2021



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DEDICATÓRIA

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“A coragem não é a ausência de medo,
é a capacidade de enfrentar o medo.

A sorte segue a coragem.”

Mario Sergio Cortella

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Resumo

RESUMO

A fotoativação é um passo importante nos procedimentos restauradores diretos e indiretos. Os equipamentos utilizados para este fim, as fontes fotoativadoras, apresentam características como potência, espectro de emissão, irradiância e formato do equipamento diferentes entre si. Essas características podem influenciar nos procedimentos odontológicos, o que torna importante o estudo e entendimento destes equipamentos para o melhor uso na associação aos materiais restauradores. O objetivo geral desta tese foi avaliar e caracterizar o desempenho de diferentes fontes fotoativadoras na ativação de restaurações diretas e indiretas. Três objetivos integram este estudo: **Objetivo específico 1:** avaliar o efeito do tipo de fonte fotoativadora e do preparo de amostras na microdureza e grau de conversão em diferentes profundidades e regiões de restaurações em dentes posteriores com resina composta *bulk-fill*. **Objetivo específico 2:** Avaliar a influência de diferentes cerâmicas, espessuras e tratamentos de superfície na transmissão de luz em diferentes comprimentos de onda violeta e azul. **Objetivo específico 3:** Avaliar o efeito da emissão de apenas luz violeta, apenas luz azul ou da combinação de ambas por uma fonte fotoativadora de múltiplos espectros, no grau de conversão de cimentos resinosos que apresentam diferentes fotoiniciadores. A partir destes estudos pode-se concluir que o preparo de amostras incluídas em resina de poliestireno para testes de dureza e grau de conversão reduzem a percepção de diferenças entre as diferentes técnicas, já na utilização de método sem inclusão e sem polimento da amostra é possível detectar diferenças produzidas pela fotoativação em áreas profundas da restauração, como caixas proximais de restaurações Classe II. De acordo com o segundo estudo, há uma rápida redução logarítmica em transmissão de luz à medida que a espessura de cerâmica aumenta, transmitindo pouca luz através de 1,5 mm de ambas as cerâmicas testadas. Nos comprimentos de onda curtos a transmissão de luz é ainda menor. A cerâmica Celtra Duo, após glaze, apresenta maior brilho de superfície comparado a IPS e.max CAD, porém não há correlação entre o brilho de superfície e a transmissão de luz. E por fim, demonstrou-se que não é necessária fonte fotoativadora de múltiplos picos para polimerizar o cimento

resinoso Variolink Esthetic LC e, o cimento resinoso RelyX Veneer não tem seu grau de conversão beneficiado pela emissão de luz violeta.

Palavras-Chave: comprimento de onda, fotoiniciadores, grau de conversão, microdureza, potência, transmissão de luz, resina composta *bulk-fill*.

Abstract

ABSTRACT

Photoactivation is an important step for direct and indirect restorative procedures. The light-curing units used for this purpose present characteristics such as radiant power, emission spectrum, irradiance, and equipment design which are different among themselves, and have influenced the procedures. The general objective of this thesis was to evaluate and characterize the performance of different light-curing units in the activation of direct and indirect restorations. Three objectives integrate this study: Specific objective 1: To evaluate the effect of the sample preparation and light-curing units on the Knoop hardness and degree of conversion at different depths and regions of bulk-fill resin-based composite restorations. Specific objective 2: To evaluate the influence of thickness and surface condition on the transmission of violet and blue light through two ceramics. Specific objective 3: To evaluate the effect of delivering isolated violet, blue, or a combination of violet and blue light from a polywave® light-curing unit (LCU) through ceramic on the degree of conversion (DC) of two resin cements with different photoinitiators. Based on the results of these studies, the following conclusions can be drawn: preparation methods that embedded the samples in polystyrene resin and polished the specimens reduced the differences between the KH and DC values obtained by different preparation techniques. The use of the NotPol method was able to detect differences produced by light activation in deeper areas. According to the second study, there is a rapid logarithmic decline in light transmission as the ceramic thickness increases means that little useful light is transmitted through 1.5 mm of either ceramic. Even less of the shorter wavelengths of light are transmitted. Celtra Duo glazed ceramic had a higher surface gloss compared to IPS e.max CAD, however there is no correlation between surface gloss and light transmission. Finally, the third study demonstrated that a polywave® light is not required to cure Variolink Esthetic LC, and RelyX Veneer does not benefit from receiving violet light from the polywave® light.

Keywords: wavelength, photoinitiators, degree of conversion, microhardness, radiant power, light transmission, bulk-fill resin composite.

Introdução

E Referencial Teórico

1. INTRODUÇÃO E REFERENCIAL TEÓRICO

A fotoativação é um passo determinante na adequada polimerização de materiais odontológicos, como as resinas compostas (Faria-e-Silva *et al.*, 2017) e os cimentos resinosos (Li *et al.*, 2021). Este procedimento é uma etapa fundamental no processo restaurador, porém, é habitualmente negligenciado (Rueggeberg, 2011; Santini & Turner, 2011; Shortall *et al.*, 2016; Soares *et al.*, 2018). A incúria deste passo pode resultar em falhas adesivas, dor pós-operatória, descoloração e falha prematura das restaurações (Soares *et al.*, 2017). Assim, cinco fatores são essenciais na adequada fotoativação dos materiais: a fonte fotoativadora utilizada, o protocolo restaurador, a extensão e localização das restaurações, o espectro de emissão da fonte fotoativadora e a exposição recebida pelo material restaurador (Price, 2010; Price *et al.*, 2014; Price *et al.*, 2015).

As fontes fotoativadoras de diodo emissor de luz (LED) substituem suas antecessoras luzes halógena de quartzo-tungstênio e arco de plasma (Rueggeberg, 2011). Embora a luz azul emitida por diferentes fontes de luz possa parecer a mesma ao olho humano, quando a saída de luz é medida usando um espectrofotômetro, a maioria destas fontes emite espectros de emissão (comprimento de onda da luz) muito diferentes (Price *et al.*, 2020). Em suas primeiras gerações, essas fontes LED, apresentam espectro de emissão estreito, em pico único, próximo ao pico de absorção da canforoquinona (450-470 nm). Porém, em sua terceira geração, estas fontes são identificadas pela presença de múltiplos picos, apresentando diferentes comprimentos de onda no intervalo de luz violeta, os quais podem ativar materiais com fotoiniciadores alternativos, além da canforoquinona (Rueggeberg, 2011; Rueggeberg *et al.*, 2017). E, mesmo sendo parte da mesma geração, os picos de comprimento de onda variam de fonte para fonte (Price *et al.*, 2020). Contudo, a menos que essas fontes de terceira geração sejam bem projetadas (Price *et al.*, 2015), elas podem apresentar heterogeneidade do feixe de luz emitido o que pode levar à polimerização heterogênea do material e às falhas prematuras (Michaud *et al.*, 2014). Isso pode ocorrer pois, o comprimento de onda violeta tem menor penetrabilidade no material comparado à luz azul, o que afeta as propriedades

mecânicas, como microdureza e grau de conversão (Shimokawa *et al.*, 2017) e pode aumentar a toxicidade dos materiais (Fujioka-Kobayashi *et al.*, 2019).

Os fabricantes caracterizam seus equipamentos considerando a distância zero entre a fonte e o material a ser fotoativado. Porém, clinicamente, a saída de luz pela ponta do aparelho difere do que é recebido pelo material restaurador. O que é recebido pelo material é dado o nome de irradiância (mW/cm^2), a qual é a potência incidente em uma superfície (Kirkpatrick, 2005; Price *et al.*, 2015). Esta é influenciada pela distância da ponta ativa (área da ponta que realmente emite luz) ao material, pela região da própria ponta da fonte, bem como, pelo tamanho da ponta ativa, que por muitas vezes é menor que o diâmetro externo da ponta do equipamento (Soares *et al.*, 2021). Ademais, quanto menor é a ponta ativa maior é a potência (mW) que, frequentemente, é concentrada em um ponto central (Shortall *et al.*, 2016, Soares *et al.*, 2021).

A fonte fotoativadora deve ser posicionada perpendicularmente ao material restaurador e sua ponta ativa estabilizada o mais próximo possível da área a ser fotoativada, durante toda a exposição à luz (Bhatt *et al.*, 2015; Soares *et al.*, 2017). Esse é um cuidado importante a ser tomado pelo profissional responsável por este passo, seja ele o cirurgião-dentista ou seu auxiliar (Soares *et al.*, 2018). Porém, o correto posicionamento da ponta pode ser dificultado em dentes posteriores devido à pouca visibilidade, ao formato angulado e pouco ergonômico do próprio equipamento (Corciolani *et al.*, 2008; Moreira *et al.*, 2021) e, também, à limitação de abertura interincisal do paciente jovem (Sousa *et al.*, 2008) ou com problemas temporomandibulares (Gupta *et al.*, 2010).

Estes fatores isolados ou associados podem prejudicar a adequada fotoativação (Corciolani *et al.*, 2008; Shortall *et al.*, 2016; Soares *et al.*, 2017; Moreira *et al.*, 2021), formando ângulos entre a ponta e a restauração que geram áreas de baixa irradiância, como nas caixas proximais de cavidades Classe II (Price *et al.*, 2000; Michaud *et al.*, 2014; Shimokawa *et al.*, 2020). Outras situações de baixa irradiância podem ocorrer no uso de resinas compostas de fotoativação única, como as resinas compostas *bulk-fill*, e na fotoativação sobre coroas e laminados cerâmicos, pois poucas fontes de luz possuem ponta ativa que cubra toda a superfície e que tenha feixe de luz homogêneo e colimado (Soares *et al.*, 2017; Shimokawa *et al.*, 2020; Soares *et al.*, 2021). Outro ponto importante, é a presença de matrizes para promoção do ponto de contato, as

quais podem gerar áreas de sombra e aumentar a distância da fonte ao material (de Jong *et al.*, 2007). Estes fatores irão influenciar negativamente nas propriedades mecânicas destes materiais (Agrawal *et al.*, 2015).

Portanto, testes de microdureza Knoop e grau de conversão são frequentemente utilizados para testar diretamente ou indiretamente a polimerização dos materiais restauradores (Park *et al.*, 2000; Chinelatti *et al.*, 2006; Li *et al.*, 2015). As amostras utilizadas nestes testes comumente devem ter alto polimento para a adequada análise da superfície e, por isso são previamente polidas (Park *et al.*, 2000; Chinelatti *et al.*, 2006). No entanto, monômeros não convertidos na superfície destas restaurações são perdidos durante o processo de preparo (Rosatto *et al.*, 2015; Novais *et al.*, 2016). E, também, a reação exotérmica da resina utilizada para inclusão destas restaurações pode produzir uma polimerização adicional destas. Desta forma, torna-se importante o desenvolvimento de estudos que avaliem o processo de preparo de amostras de restaurações para testes de microdureza e grau de conversão, bem como avaliar o efeito da abertura interincisal e do tipo de fonte fotoativadora nestas amostras.

Quanto às restaurações indiretas, os mesmos princípios e cuidados são mantidos como regra. Os cimentos resinosos necessitam de luz para atingir a adequada polimerização (Kilinc *et al.*, 2011). Esses materiais podem usar mais de um tipo de sistema fotoiniciador. A canforoquinona é um iniciador do Tipo II comumente utilizado, o qual é sensível a comprimentos de onda longos de luz azul. Alguns novos materiais que usam fotoiniciadores alternativos do Tipo I livres de amina, como óxido de difenil (2,4,6- trimetilbenzoil) fosfina (TPO) ou à base de germânio (Ivocerin®) tem sido utilizados por apresentarem melhor estabilidade de cor (Alkurt *et al.*, 2018).

A fim de ativar estes materiais uma ampla faixa de comprimentos de onda de luz é utilizada (400-500 nm) (Rueggeberg *et al.*, 2017). Porém, há atenuação da luz à medida que a espessura da cerâmica aumenta (Della Bona *et al.*, 2014; Pacheco *et al.*, 2019), o que reduz a quantidade de luz que chega ao cimento resinoso (Inokoshi *et al.*, 2016; Ilie, 2017; Faria & Pfeifer 2017; Kwon *et al.*, 2018; Martins *et al.*, 2019) e esta redução na quantidade de luz recebida pode reduzir o grau de conversão dos monômeros do material (Pacheco *et al.*, 2019; Martins *et al.*, 2019). Porém, os efeitos dos diferentes picos de

comprimentos de onda da luz emitida por fontes fotoativadoras através de diferentes cerâmicas odontológicas ainda é desconhecido e seu efeito nos cimentos resinosos deve ser estudado.

Portanto, a associação de metodologias torna-se importante ferramenta na caracterização e na avaliação do desempenho das fontes fotoativadoras, obtendo-se importantes informações por meio da correlação de resultados para o maior esclarecimento do comportamento de diferentes espectros de emissão através de resinas compostas e cimentos resinosos.

Objetivos

2. OBJETIVOS

2.1 Objetivo Geral

Avaliar e caracterizar o desempenho de diferentes fontes fotoativadoras na ativação de restaurações diretas e indiretas.

2.2 Objetivos Específicos

Objetivo específico 1

Capítulo 1 - *Effect of the sample preparation and light-curing unit on the microhardness and degree of conversion of bulk-fill resin-based composite restorations*

Avaliar o efeito do tipo de fonte fotoativadora e do preparo de amostras na microdureza e grau de conversão em diferentes profundidades e regiões de restaurações em dentes posteriores, com resina composta *bulk-fill* em dentes.

Objetivo específico 2

Capítulo 2 - *Effect of thickness, glazing, or polishing on the transmission of violet and blue light through two CAD/CAM ceramics*

Avaliar a influência de diferentes cerâmicas, espessuras e tratamentos de superfície, na transmissão de luz, em diferentes comprimentos de onda violeta e azul.

Objetivo específico 3

Capítulo 3 - *Effect of the additional violet light from the light-curing unit on two resin cements that use different photoinitiators*

Avaliar o efeito da emissão de apenas luz violeta, apenas luz azul ou da combinação de ambas por uma fonte fotoativadora de múltiplos espectros no grau de conversão de cimentos resinosos que apresentam diferentes fotoiniciadores.

Capítulos

Capítulos

3.1 CAPÍTULO 1

Effect of the sample preparation and light-curing unit on the microhardness and degree of conversion of bulk-fill resin-based composite restorations

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Effect of the sample preparation and light-curing unit on the microhardness and degree of conversion of bulk-fill resin-based composite restorations

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Running title: Sample preparation and light-curing unit on composite restorations

Keywords: Bulk-fill resin composites; degree of conversion; Knoop microhardness; light-curing unit; sample preparation

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Effect of the sample preparation and light-curing unit on the microhardness and degree of conversion of bulk-fill resin-based composite restorations

RUNNING TITLE: Sample preparation and light-curing unit on composite restorations

ABSTRACT

OBJECTIVES: To evaluate the effect of the sample preparation and light-curing units (LCUs) on the Knoop hardness (KH, N/mm²) and degree of conversion (DC, %) at different depths and regions of bulk-fill resin-based composite restorations.

METHODS: Two molds for specimen preparation were made using molar teeth embedded in acrylic resin. One was a conventional tooth mold where the molar received an MOD preparation. The other was a new method where the molar was sectioned in three slices (buccal, middle, and lingual). The middle slice also received an MOD preparation in the same parameters as the conventional mold. To perform restorations, the molds were placed in the second mandibular molar position in a dentoform with a 44 mm interincisal opening. Restorations were made using Opus Bulk Fill (FGM) high viscosity bulk-fill resin-based composite (RBC) and light-cured using two different lights: VALO Cordless (Ultradent) and Bluephase G2 (Ivoclar Vivadent). The RBC was placed in one increment and light-cured for 40 seconds. The RBC specimens were prepared accordingly: CEmPol – tooth mold, specimen was embedded in polystyrene resin and polished before testing; CPol – tooth mold, specimen was not embedded, but polished before testing; NotPol – sectioned tooth, specimen not embedded nor polished before testing. The KH was measured in different depths and regions of the specimens as well as the DC where Raman spectroscopy was used. Both data were analyzed using a 2-

way analysis of variance for repeated measures followed by Tukey post-hoc test ($\alpha=0.05$).

RESULTS: Preparation method ($P<0.001$), depth of restoration ($P<0.001$) and the interaction between method and depth ($P=0.003$) all influenced the KH values. Preparation method ($P<0.001$), tooth region ($P<0.001$) and the interaction between method and tooth region ($P=0.002$) all influenced DC values. KH values were reduced significantly from the top to the bottom of the restorations, mainly at the proximal boxes. The NotPol method was the most effective method to detect differences for the depth of the restoration and tooth region. A lower DC was found at the gingival regions of the proximal boxes of the restorations. When the KH and DC values were compared, there was no significant differences between LCUs (KH $P=0.4$ and DC $P=0.317$).

CONCLUSION: Preparation methods that embedded the samples in polystyrene resin and polished the specimens reduced the differences between the KH and DC values obtained by different preparation techniques. The use of the NotPol method was able to detect differences produced by light activation in deeper areas.

CLINICAL RELEVANCE: *In vitro* studies should simulate clinical conditions as the resin composite properties are dependent on the sample preparation. Clinicians should pay attention to the proximal boxes of bulk-fill resin composite restorations as those areas may not receive sufficient light for adequate polymerization.

INTRODUCTION

The use of incrementally filled resin-based composites (RBC) can produce restorations that have a higher hardness, a higher degree of conversion, and lower shrinkage stress.¹ However, the use of the incremental layering and photo-curing technique is both time-consuming and more likely to incorporate voids, or contamination between each increment of RBC.² The drive for faster strategies to restore deep cavities has led to the development and commercialization of new materials that have an increased depth of cure and that can be placed in 4 to 5 mm thick increments.³⁻⁵

Using bulk-fill RBCs enables 4-5 mm deep restoration to be entirely filled before light curing and yet still achieve mechanical properties comparable

to restorations made using the incremental filling and incremental light curing technique.⁶ RBC restorations require appropriate polymerization of the material,⁴ and although the light-curing is a critical procedure, it is often overlooked.⁷⁻¹⁰ When the light curing process is performed incorrectly, it may lead to debonding, post-operative pain, discoloration and premature failure of the composite resin restorations.¹¹ Ideally the RBC should require a short exposure time, and yet exhibit low shrinkage stress, and uniform conversion of monomers in all parts of the restoration.¹² Five aspects are essential regarding the polymerization of RBC restorations: the light-curing unit (LCU), the restorative protocol, the extension and location of the restoration, the emission spectrum from the light source, and the radiant exposure received by the composite resin.¹³⁻¹⁵

Restorations in the posterior regions of the mouth are challenging for clinicians due to the presence of dental structures, the type and opacity of restorative material, and the difficulty of positioning the LCU tip perpendicular over the restorations in the mouth.⁹ The extent of the interincisal opening can often affect the access of the tip over restorations. Therefore, the greater interincisal opening, the easier it is to position the tip of the LCU over the posterior teeth.^{11,16} Children,¹⁷ and patients with temporomandibular joint issues often have a limited mouth opening,¹⁸ that will prevent adequate access of the LCU to the teeth. The design of the LCUs, shape and the angulation of the light tip, can also impair the ideal positioning of the LCU tip perpendicular to the surface of the restoration.¹⁹ Limited mouth opening, the presence of matrix bands and poor design of the LCU may lead to an increased distance between the restoration and LCU tips, and may also introduce shadows that will receive less light. This will negatively influence their mechanical properties, solubility, dimensional and color stability, and biocompatibility of the RBC.^{11,20}

Microhardness tests (Knoop or Vickers) and Raman spectroscopy are methods that are often used to measure directly or indirectly the polymerization of RBCs.²¹⁻²³ However, during the sample preparation, the RBC restorations are often embedded in resin and then polished. Additionally, the surface must be flat for adequate hardness and degree of conversion (DC) measurements. Therefore, the restorations are frequently polished.^{21,22}

Unconverted monomers on the surface of the RBCs are lost during the finishing and polishing processes.^{24,25} The exothermic heat produced during polymerization of the embedding material and the heat produced during polishing may produce further polymerization of the RBC.

There is a lack of studies that have analyzed the effect of the preparation method for microhardness and DC analyses and the effect of the LCU design when used in a posterior RBC restoration. Therefore, this study aimed to evaluate, through Knoop microhardness (KH) and DC tests, the effect of the sample preparation of bulk-fill posterior RBC restorations in second molar teeth, prepared in a dentoform with an interincisal mouth opening corresponding to a 12-year-old adolescent.^{17,26} The null hypotheses were: 1) The sample preparation method would not affect the KH or the DC of the bulk-fill restorations, and 2) The choice of LC will have no influence on the KH and DC of the bulk-fill RBCs.

METHODS AND MATERIALS

Cavity preparation

This study was conducted following ethical principles and was approved by the local ethics committee with protocol number 2.985.056. Two extracted intact caries-free human mandibular molar teeth with an average dimension of 10-mm mesiodistal and 4.7-mm of occluso-pulpal distances were used to make two different molds for specimen preparation.

For the conventional sample preparation method (Figure 1A), the molar tooth was embedded in red acrylic resin (Dencrilay, Dencril, Pirassununga, SP, Brazil) up to 2.0 mm below the cemento-enamel junction, to prevent extraneous light exposure. At the same time, allowing some light-curing below the cemento-enamel junction (Figure 1B).²⁷ Using a cavity preparation machine²⁸ a standard class II MOD cavity was prepared using a cylindrical round diamond bur #3146 (KG Sorensen, Barueri, SP, Brazil) in a high-speed handpiece (Kavo do Brasil, Joinville, SC, Brazil) under copious air and water irrigation. The preparations had approximately 4/5 of the intercuspal width, a 6-degree divergence, a 4.0-mm occlusal-pulpal depth, a proximal box that was 2.0-mm wide, and a further 1.0-mm deep, resulting in 5-mm deep proximal boxes (Figure 1B-D).

For the new sample preparation method, a three part matrix was developed.²⁹ The molar tooth was fully embedded leaving the occlusal region exposed (Figure 1E) in chemically activated red acrylic resin (Dencrilay, Dencril, Pirassununga, SP, Brazil) and then sectioned into three parts (buccal, middle, and lingual) (Figure 1F). An impression of the middle part was taken with addition vinyl polysiloxane (Scan Putty Regular, Yllar, Pelotas, RS, Brazil) to record its mesial and distal contours (Figure 1G), then an MOD cavity preparation was made using the same parameters described for the conventional method (Figure 1F). The buccal and lingual parts of the matrix had their transverse surfaces polished using silicon carbide abrasive paper of decreasing grit size (#1200, #1500, #2000 and #2500, Norton, Campinas, SP, Brazil) followed by polishing with diamond pastes (6 µm; 3 µm; 1 µm; 0.25 µm; Arotec, São Paulo, SP, Brazil) on felt discs. All three parts were then clamped together to make an MOD cavity (Figure 1F). This mold enabled easy removal of the restoration by separating the three parts of the matrix after light-curing (Figure 1).

Development of a buccal opening simulation device

In an attempt to simulate the clinical environment, all restorations were light-cured in a tooth positioned in the second mandibular molar region. A device was developed to simulate a standardized mouth opening. A dentoform (MOM, Marília, São Paulo, Brazil) modifying the mandibular second molar so that a cylinder of red acrylic resin (Dencrilay, Dencril, Pirassununga, SP, Brazil) with the molar tooth could be inserted. The prepared teeth were positioned to allow the proximal contact with its adjacent tooth (Figure 2A). Screws were used to fix and stabilize the resin cylinders in the correct position (Figure 2A). The dentoform was positioned in a dental patient simulator (MOM, Marília, São Paulo, Brazil). The interincisal mouth opening was fixed at 44 mm, which corresponds to the mouth opening of a 12-year-old adolescent (Figure 2B).^{17,26}

Restorative procedure

A high viscosity bulk-fill RBC (Opus Bulk fill APS, FGM, Joinville, SC, Brazil) and two multiple peak LCUs: VALO Cordless on Standard power (Ultradent, South Jordan, UT, USA) and Bluephase G2 on High power (Ivoclar Vivadent, Schaan, Liechtenstein) were used. The irradiance (mW/cm²), emission

spectrum (mW/cm²/nm), and radiant exposure (irradiance x time = energy/area = J/cm²) emitted from the LCUs was measured five times using the MARC Resin Calibrator (BlueLight Analytics, Halifax, NS, Canada). The LCU's internal tip diameter was measured using a digital caliper (Mitutoyo, Mississauga, ON, Canada). The sectional matrix band (Unimatrix, TDV Dental Ltda, Pomerode, Santa Catarina, Brazil) was positioned and stabilized using an interdental wooden wedge at the mesial contact point to the groups that used the conventional tooth mold. The bulk-fill RBC was placed, up to 5-mm thick and the LCU was positioned 1 mm above the tooth's occlusal surface. The RBC was light-cured for 40 seconds over the occluso-mesial and 40 seconds over the occluso-distal regions by a well-trained operator, following the manufacturer recommendation to cover the whole tooth area. The light-curing process was performed in a dark room with yellow light to avoid any possible light interfering with the RBC polymerization process. No adhesive system was applied so that the RBC sample could be removed from the mold. After light curing, the restorations were removed and stored in the dark and a controlled humidity at 37 °C for 24h.

Preparation of the samples for microhardness test (n = 5)

The restorations were prepared following the three groups:

CEmPol – after removed from the mold, restorations were embedded in polystyrene resin (Cristal, Piracicaba, SP, Brazil). Before testing, the RBC surfaces were finished with silicon-carbide paper (#1200, 1500, 2000, and 2500 grit sizes; Norton, Campinas, SP, Brazil) followed by cleaning in an ultrasonic bath in distilled water for 5 minutes, and polished with metallographic diamond pastes (6-, 3-, 1-, and 1/4-µm sizes; Arotec, São Paulo, SP, Brazil).

CPol – the RBC restorations were not embedded in polystyrene resin, but were polished as described for the CEmPol Group and fixed on a glass coverslip using cyanoacrylate (Super Bonder Loctite, São Paulo, SP, Brazil).

NotPol – restorations made using the three-part matrix²⁹ were not polished nor embedded in polystyrene resin. Instead the RBCs were removed from the tooth mold and stabilized with cyanoacrylate on a glass slide. The smooth RBC surface was indirectly obtained by the polishing that had been performed on the transverse surfaces of the tooth mold.

Knoop microhardness test (KH)

Knoop microhardness indentations (Shimadzu HMV 2000; Shimadzu Corporation; Kyoto, Japan) were made in the transverse surface of the restorations using 50 g for 10 s at every 1.0 mm from the gingival and pulpal walls of the restorations. Ten indentations were made at proximal box and eight indentations were made at the occlusal box (Figure 3A). The recorded KH data were inserted into the Origin Pro 2020 (OriginLab, Northampton, MA, USA) software to produce the hardness maps of the groups.

Degree of conversion (%)

The degree of conversion (DC) in the mesial and distal proximal boxes was evaluated in 2 spots (an occlusal spot and a proximal box spot being a 2-mm distance from each measurement, Figure 3B), and a spot at the center of the specimen (occlusal spot) using a LabRam HR Evolution Raman spectrometer (Horiba LabRam, Villeneuve d'Ascq, France) operating at an excitation power of 17 mW with radiation emitted by a He-Ne laser (633 nm) was used. Raman signal was acquired using 600 line/mm grating centered between 1000 and 2000 cm^{-1} with 200 μm confocal hole. These settings enabled us to acquire spectra of 1.05 cm^{-1} / pixel of resolution. The spectra were adjusted by polynomial function and by manual multiple point baseline correction. From the Raman vibrational modes, the areas of peaks: aliphatic (1638 cm^{-1}) and aromatic (1608 cm^{-1}) were calculated from polymerized (P), and unpolymerized (NP) bulk-fill RBC samples. The formula used for the calculation of the degree of conversion was: $\text{DC (\%)} = (1 - P / \text{NP}) \times 100$.

Statistical analysis of data

The KH and DC values were tested for normal distribution and equality of variances using the Shapiro-Wilk and Levene tests. Then, data were analyzed using two-way repeated-measures analysis of variance and Tukey post-hoc tests. All tests used a 0.05 level of statistical significance and were performed using the statistical software Sigma Plot version 13.1 (Systat Software Inc, San Jose, CA, USA).

RESULTS

LCUs characterization

The mean and standard deviation of the tip irradiance values for the VALO Cordless was 1298 ± 3.3 mW/cm², and Bluephase G2 was 1394 ± 4.5 mW/cm². The emission spectrum for VALO Cordless ranged between 395-480 nm and Bluephase G2 between 385-515 nm. Each restoration received a radiant exposure of 51.9 J/cm² for VALO Cordless, and 55.8 J/cm² for Bluephase G2 at each light-curing spot: occluso-mesial and occluso-distal regions.

Knoop microhardness – KH (N/mm²)

Means for KH values obtained in the specimens made using the two LCUs for each sample preparation method at various restoration depths are reported in Figure 4. ANOVA results demonstrated that the sample preparation method had a significant effect ($P < 0.001$), the effects of the restoration depth were significant ($P < 0.001$), and there was an interaction between sample preparation method and restoration depth ($P = 0.003$). However, the choice of LCU had no significant effect ($P = 0.475$), neither for interaction between sample preparation method and LCU ($P = 0.734$), LCU and depth of restoration ($P = 0.700$); and there was no interaction among sample preparation method, LCU and depth of restoration ($P = 0.766$). The KH values, in general, were significantly reduced from the top to the bottom of the restoration, mainly at proximal boxes ($P < 0.05$). The NotPol method was the most sensitive method at detecting the effects of restoration depth and tooth region.

Degree of Conversion - DC (%)

Means and standard deviations for DC values obtained in the specimens made using the two LCUs for each sample preparation method at various restoration depths are reported in Figure 5. ANOVA results demonstrated significant influence for sample preparation method ($P < 0.001$), tooth region ($P < 0.001$), and for the interaction between sample preparation method and tooth region ($P = 0.002$). However, no significant influence for LCU ($P = 0.127$), sample preparation method and LCU ($P = 0.104$), LCU and tooth region ($P = 0.114$), sample preparation method, LCU and tooth region ($P = 0.154$). DC values were significantly lower on the gingival region of the proximal boxes than the measurements made on the top of the restorations, irrespective of the local, occlusal or distal areas. The DC measured on the M2

spot (gingival region) of the restoration's mesial proximal box for the NotPol method was lower than the other methods.

DISCUSSION

The sample preparation method clearly influenced the KH and DC results. Therefore, the first null hypothesis was rejected. However, the LCU had no significant influence on these parameters. Thus, the second hypothesis was accepted. *In vitro* studies usually perform restorations under ideal conditions, without any limitation on mouth opening or any difficulty in positioning the LCU over the restorations.^{24,30} Thus these studies are hardly clinically relevant. In clinical procedures, the LCUs are often misaligned when light curing a restoration in the posterior teeth.²³ For this reason, the experimental design used in the present study aimed to simulate the clinical condition.

The microhardness test method that is often used to assess the polymerization of RBCs requires a flat smooth surface. For this reason, the samples are frequently embedded and polished before the hardness assessment.²⁴ The polystyrene resin, commonly used for the embedment of specimens, has an exothermic polymerization reaction. The concern is that the exothermic reaction, it may heat the sample and thus improve the polymerization of the RBC.^{31,32} The CPol and NotPol specimens made in the present study were not embedded and thus received no additional heating effect. The present study found that the increase in the heat of the RBC caused by the exothermic reaction of the polystyrene resin can affect the degree of conversion and Knoop microhardness of the tested bulk-fill RBC. Different bulk-fill RBCs will have different compositions that may also affect the DC and KH results. Another possible effect of the polishing procedure is that the superficial layer that may contain unreacted monomers is removed, thus altering the hardness results. Removing this layer does not reflect the clinical condition where only the superficial layer of RBC is polished. The RBC contacting the cavity walls is never polished. In this study, the DC on the gingival region of the proximal boxes of the restorations made using the NotPol method was lower than the DC measured in the restorations made with the other techniques, this occurred probably due to the effects of the polishing process in CEmPol and CPol. On the other hand, the vinyl polysiloxane material present surrounding the proximal boxes of the NotPol

specimens may affect the light transmission at these regions. However, the three-part matrix method has already been shown to produce a good performance when detecting differences in RBC properties evaluation, mainly at the proximal boxes of the restorations and is recommended for future studies.²⁹

Regarding the polishing process prior to the microhardness testing, other factors might be considered. Such as when polishing RBCs, the use of water coolant to prevent local heat generation may preferentially remove relatively hydrophilic, free monomers, such as residual triethylene glycol dimethacrylate (TEGDMA) or other low molecular weight monomers that have some degree of water solubility,³³⁻³⁶ from surfaces of cut or polished specimens. Some studies polish RBC surfaces using an alcohol-based solution³⁷⁻³⁹ prior to microhardness testing. In the present study, isopropyl alcohol was used on the polishing process of the CEmPol and CPol RBCs samples. This treatment removes even more residual monomer, have an even greater effect on the RBC surface properties, and on the bottom/top hardness ratios than polishing with an aqueous-based solution.⁴⁰⁻⁴²

The KH and DC tests should be performed at a standardized post-cured time, because the restorations do not develop their final mechanical properties immediately after curing.⁴³ Therefore, in the present study, all the specimens were stored for 24 hours before testing. It was possible to notice that KH and DC values in general reduced significantly from the top to the bottom of the restoration, mainly at proximal boxes, which raises the concern of having a premature failure in those areas due to lack of ideal polymerization. Although the use of bulk-fill RBCs reduces the chair time, a lack of adequate polymerization along the bulk of the restoration may result in lower mechanical properties in some regions, compared to a highly filled nano-hybrid RBC restorations that were placed and light cured using an incremental technique.³⁰

In this study, even at the mesial proximal boxes of the NotPol group the DC values were greater than 60%. The high DC could be attributed to the long exposure time of 80 seconds delivered to each RBC restoration. The radiant exposure values were 51.9 J/cm² for VALO Cordless, and 55.8 J/cm² for Bluephase G2 at each light-curing spot: occluso-mesial and occluso-distal regions, and the DC values were higher at the surface spots of the RBC restorations (over 80%), different of other studies results that showed lower DC

values.^{3,5,6} This exposure time was chosen because the manufacturer recommended 40 seconds of exposure time, however, to cover the whole tooth area is recommended to light cure at more than one spot.¹¹ Sufficient polymerization is achieved when the bottom of RBC restorations receive radiant exposure between 0.7-1.5 J/cm².⁴⁴

Different methods are used for the sample preparation for KH and DC tests. Specimens made with the material using a Teflon mold³⁰ or silicon mold between two polyester strips⁴ do not impose difficulties in the positioning of the LCUs over restorations that are frequent in clinical conditions. In the present study, the inter-incisal distance was set to 44 mm, representing the mouth opening of children between 12 and 14 years old.^{17,26} This mouth opening is close to the adults (18 to 70 years old) mouth opening that ranges from 56.6 mm to 49.1 mm for men, and ranges from 49.8 mm to 44.4 mm for woman.⁴⁵ However, an Irish study reported 43 mm of mouth opening for men and 41 mm for women⁴⁶ which only slightly smaller than the mouth opening of Brazilian children.¹⁷

The mouth opening, the location of the cavity, and the operator experience affect the total energy delivered to RBCs.¹⁶ The use of a patient simulator with an adjustable mouth opening enables further studies also to better represent the clinical reality. As shown by the results, the interincisal distance of 44 mm did not impair either of the quality LCUs tested in this study. The Bluephase G2 has a curved tip and Valo Cordless, a straight design showed similar KH and DC results. Therefore, for the mouth opening distance used in this study both of these LCUs show similar results, which may not be true when it comes to a patient who presents a more limited mandibular range of motion or with LCUs that are not so ergonomically designed. The hardness maps (Figure 4) still showed lower polymerization at the bottom of the mesial proximal boxes than at the, distal proximal boxes. This occurred mainly in the Bluephase G2 restorations. It may indicate the difficulty of the Bluephase G2 positioning due to the light tip angle even though the statistical analysis did not indicate any difference between the LCUs. The clinicians should pay attention to this LCU characteristic because of the possibility of reducing the irradiance at the proximal boxes when LCUs with a higher light tip angle are used.¹¹ The additional light exposure by buccal and lingual regions of RBC restoration is commonly used after removing the sectional matrix band to compensate the deficit of

polymerization in this condition. However, the light attenuation through the tooth structure impairs this compensation.⁴⁷

The different designs of the two multi-peak LED lights were used to help elucidate if the tip shape and angulation factors should be considered when choosing an LCU from a quality manufacturer to cure the RBC, especially in areas where the position of the tip over the restoration could be affected. In a recent study comparing 22 contemporary light-curing units,¹¹ it was shown that the tip design will affect the ability to position the light tip over posterior teeth. This condition may affect the beam profile of the LCUs, even though both LCUs tested are multi-peak, both have homogeneous beam profiles.^{48,49} However, further studies are encouraged evaluating the effect of the mouth opening and LCU designs on the beam profile of the LCUs. Further studies could use a mouth opening less than 44 mm, representing a child or a patient with limited mouth opening. Also, the authors suggest that further *in vitro* studies should simulate restorations made under clinical conditions using a dentoform and a three-part matrix, since the properties of the restorations such as DC and KH are dependent of the sample preparation method.

CONCLUSION

Within the limitations of this *in vitro* study, it was concluded that sample preparation that embedded and polished specimens before testing reduced the differences between the KH and DC values of one bulk-fill RBC. However, the use of the NotPol method was able to detect differences produced by light curing in deeper areas of the restorations. No significant differences were found between Valo Cordless and Bluephase G2 when using a 44 mm mouth opening.

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FIGURES

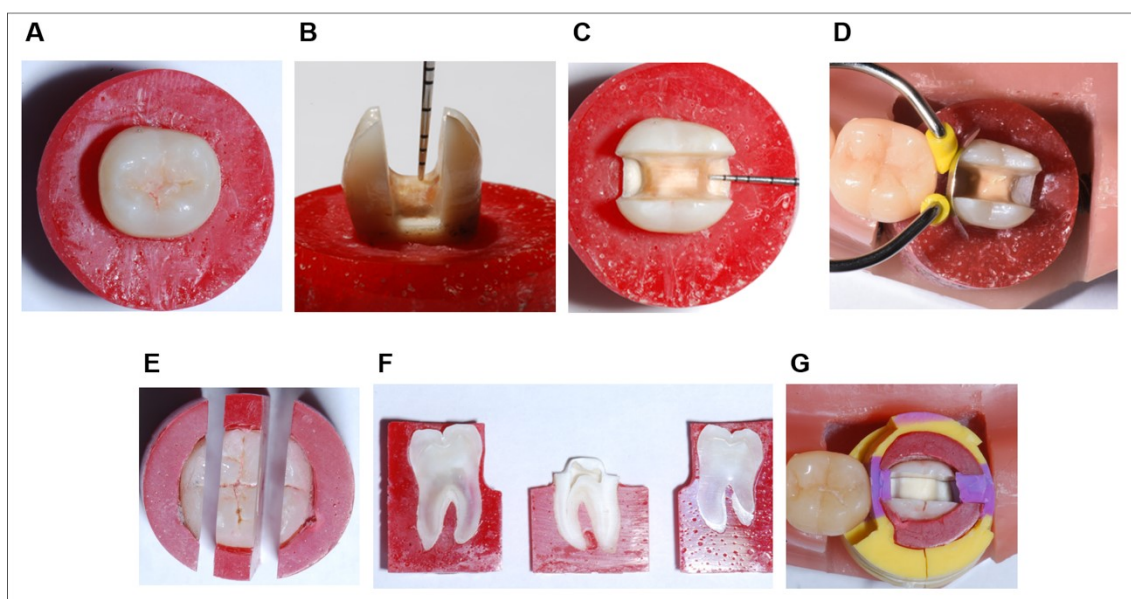


Figure 1. Conventional matrix: (A) Molar tooth embedded in chemically activated acrylic resin in red colour. (B) Cavity preparation with proximal boxes with total of 5 mm depth. (C) Occlusal view of the sample. (D) Sample showing its contact point. Three-part matrix: (E) Molar tooth totally embedded in chemically activated red acrylic resin, and sectioned in 3 parts (buccal, middle, and lingual). (F) View of the buccal, middle, and lingual parts. Middle part with no resin in the coronal third. (G) Sample showing its contact point.

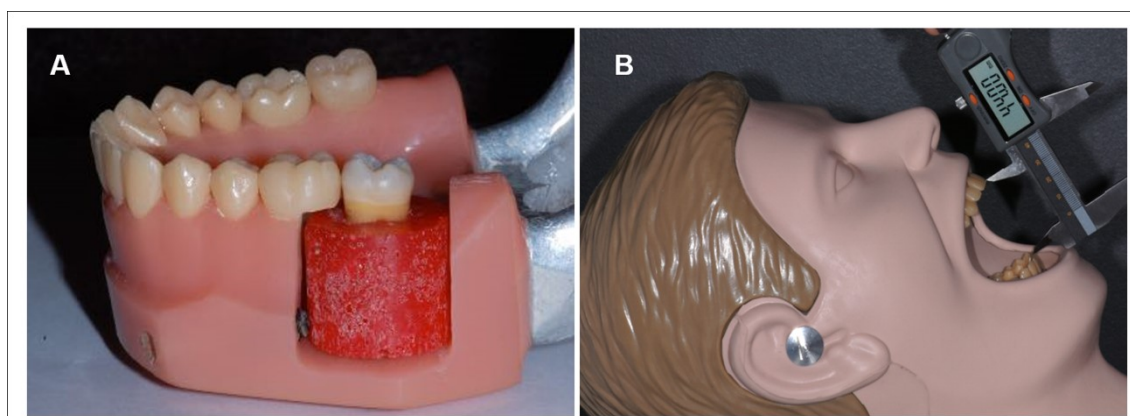


Figure 2. Development of buccal opening simulation device: (A) Dentoform adapted to hold a cylinder sample at the second mandibular molar alveoli and sample showing its contact point. (B) Interincisal distance of 44 mm.

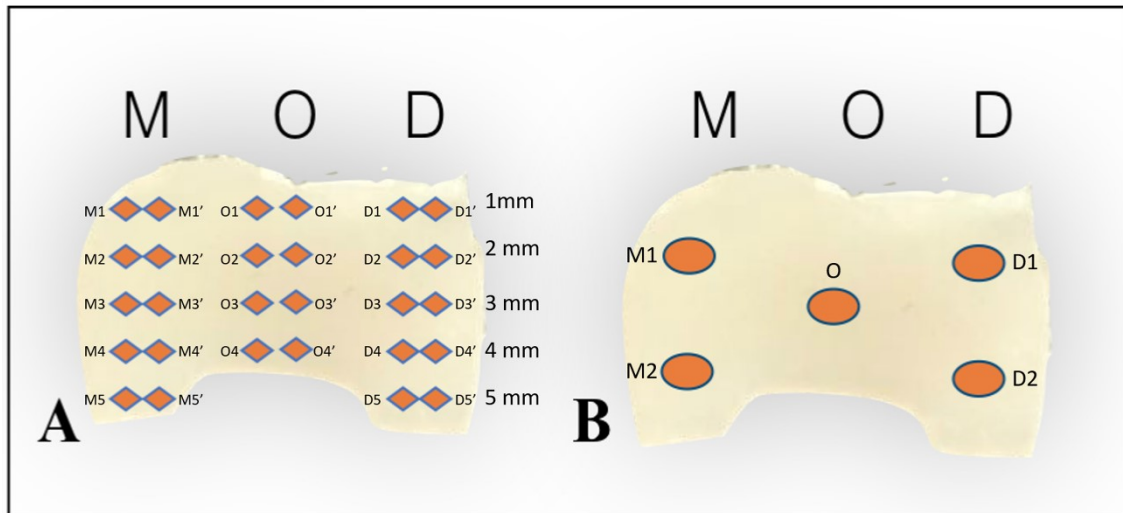


Figure 3. Locations where the tests were performed: (A) Knoop microhardness indentations. (B) Degree of conversion measurement locations.

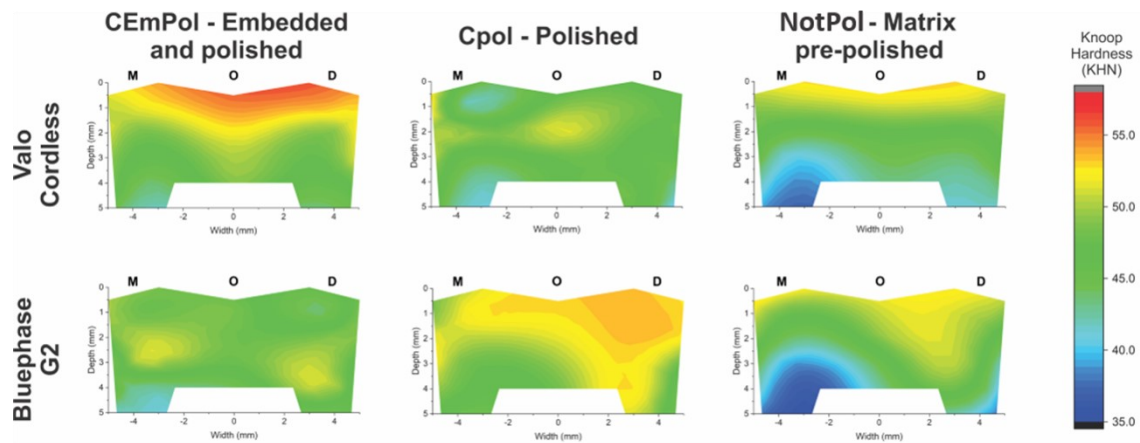


Figure 4. Knoop hardness maps from the means obtained (n=5) comparing the three different sample preparation methods and the two LCUs used in different depths.

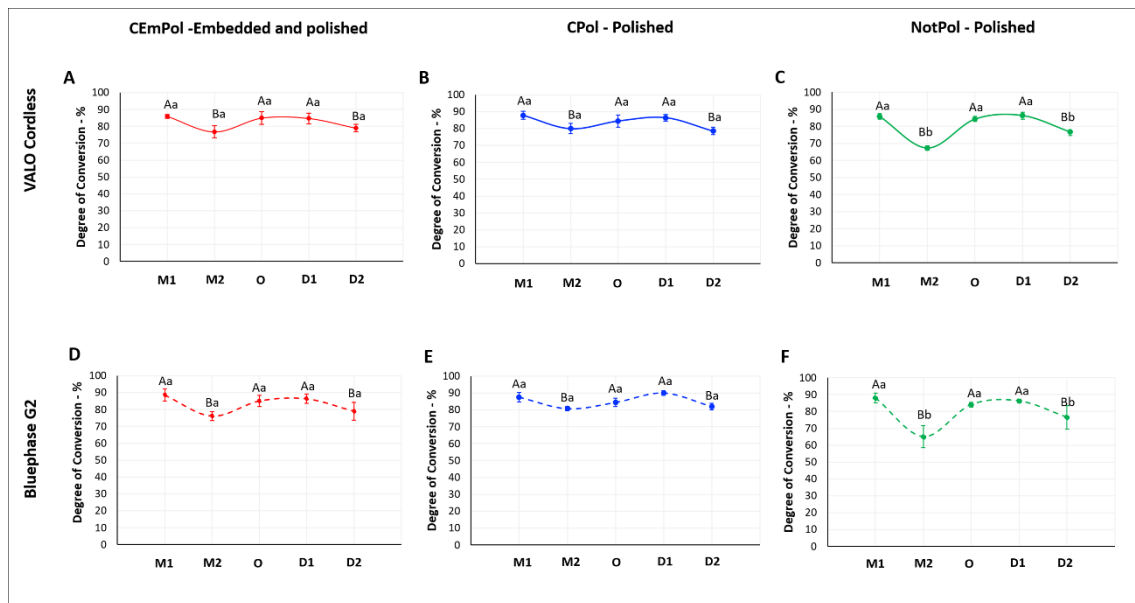


Figure 5. Means and standard deviation of Degree of Conversion (%) performed (n=5) comparing the three different sample preparation methods and the two LCUs used in different depths. Different letters indicate a significant difference: uppercase letters are used for comparing the tooth region, and lowercase letters are used for comparing the sample preparation method ($p < 0.05$).

*M1: occluso-mesial spot; M2: mesial proximal box spot; O: occlusal spot; D1: occluso-distal spot; D2: distal proximal box spot.

Capítulos

3.2 CAPÍTULO 2

Effect of thickness, glazing, or polishing on the transmission of violet and blue light through two CAD/CAM ceramics

Artigo a ser submetido para publicação no periódico **Dental Materials**

Effect of thickness, glazing, or polishing on the transmission of violet and blue light through two CAD/CAM ceramics

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Running title: Transmission of violet and blue light through CAD/CAM ceramics

Declarations of interest: None.

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Effect of thickness, glazing, or polishing on the transmission of violet and blue light through two CAD/CAM ceramics

ABSTRACT

Objectives: Evaluate the influence of thickness and surface condition on the transmission of violet and blue light through two ceramics.

Methods: Celtra Duo (Dentsply Sirona) zirconia-reinforced lithium silicate and IPS e.max CAD lithium disilicate glass-ceramic (Ivoclar Vivadent) were examined. CAD/CAM blocks were sectioned into slices (0.25 - 3.30 mm thick) and treated following their manufacturer's instructions. The light transmission through the Celtra was measured: without treatment (WT), after polishing (P), after polishing with diamond paste (PP), and after glazing (G). Transmission through e.max was measured only after glazing using an integrating sphere attached to a fiber-optic spectrometer (Ocean Insight). The % transmission was measured at 396-, 406-, 447-, 454-, 465-, and 476-nm wavelengths. The surface gloss was measured using a glossmeter (Rhopoint). The beam profiles were evaluated using a CCD camera (Ophir-Spiricon), and they were qualitatively analyzed. Data were subjected to two-way ANOVA and post-hoc Fisher's exact test ($\alpha = 0.05$).

Results: There was a logarithmic decline in light transmission as the thickness increased. At the longer wavelengths, more light was transmitted through Celtra; at the shorter wavelengths, more light was transmitted through e.max. The beam profile images confirmed these results. Celtra glazed ceramic had higher surface gloss values, and there was no difference between the PP or G groups of Celtra.

Significance: The rapid logarithmic decline in light transmission as the ceramic thickness increases means that little useful light is transmitted through 1.5 mm of either ceramic. Even less of the shorter wavelengths of light are transmitted. Celtra glazed ceramic had a higher surface gloss compared to e.max.

Keywords: ceramics; thickness; surface condition; light transmission; radiant power; surface gloss; beam profile; light-curing units.

1 Introduction

The demand by patients for esthetic restorations has increased the use of all-ceramic indirect materials that can offer both aesthetic advantages and acceptable clinical longevity [1]. The evolution in Computer-Aided Designed and Computer-Aided Manufactured (CAD/CAM) technology has accelerated and further promoted these developments [2]. In 2020, it was estimated that dentists worldwide place on average about twenty indirect restorations every month, mostly single crowns [3]. CAD/CAM technology can be a more predictable technique, because it is less time-consuming, and it may be less technique sensitive [4]. The lithium disilicate glass-ceramics are commonly used and have excellent mechanical behaviors; on the other hand, they will reduce the amount of light that reaches the luting cement [5,6] to the extent that the resin cement may receive insufficient light. This attenuation has been reported to be greater as the ceramic thickness increases [7,8], and this reduction in the amount of light received may reduce the degree of conversion of monomers in the resin cement [6, 8], but the effect on the different wavelengths of light emitted by contemporary curing lights is largely unknown. It is already known that shorter wavelengths (violet light) of light do not penetrate through the indirect restorative material as well as longer wavelengths (blue light); this may affect the degree of conversion and microhardness of resin cements that use photoinitiators requiring these shorter wavelengths of light [8, 9].

The material of choice to bond all-ceramic restorations to the tooth is usually a resin cement [10] that can be bonded to the tooth [11]. These dual- and light-cured resin cements are activated by light, and even dual-cured resin cement still benefits from receiving light [10, 12, 13]. The dual- or light-cured resin cements may contain one or more photoinitiator systems that are activated by different wavelengths of visible light [14]. The most common system is a Norrish type II initiator (camphorquinone), which is sensitive to the longer wavelengths (peak at ~ 468 nm) of blue light [15]. However, any unreacted amine or benzoyl peroxide after polymerization can cause discoloration and yellowing of the resin cement over time [16]. To tackle this issue, some cements use Norrish type I systems,

such as diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide (TPO). The initiators are amine-free and sensitive to shorter wavelengths of light (<410 nm) [15]. The use of light-cured resin cements with these alternative initiators (amine-free cements) may reduce the color change and yellowness, thus improving the long-term aesthetic of indirect ceramic restorations [11, 16-18]. In order to light cure restorative materials that use these alternative Norrish type I initiators as well as camphorquinone, light-curing units (LCUs) have been developed that emit multiple-wavelength peaks of light in both the violet and blue range [19]. However, unless well designed, these LCUs may deliver an inhomogeneous light output [20].

Recent studies have shown that the ceramic thickness has a great effect on the light energy delivered to the resin cement [8, 13, 21, 22]. A logarithmic decay in light transmission has been reported as the thickness of the ceramic increases [8, 21]; however, the effect of thickness on the amount of violet and blue light transmitted through the different types requires examination. Additionally, the effect of using a polished surface compared to a glazed ceramic surface requires examination.

Thus, this study aimed to determine the influence of different ceramics, thicknesses, and surface conditions on the light transmission at six discrete wavelengths in the violet and blue wavelength ranges. The null hypotheses were that:

- 1) the thickness and finishing treatments would not influence the light transmission through different types of ceramic;
- 2) the wavelength of light would not affect the light transmission through the ceramic; and
- 3) the finishing treatments would have no effect on the surface gloss of one ceramic.

2 Material and Methods

2.1 Study design

Two ceramic materials used for CAD/CAM restorations were used in this study: Celtra Duo zirconia-reinforced lithium silicate (Dentsply Sirona, York, PA, USA) and IPS e.max CAD lithium disilicate glass-ceramic (Ivoclar Vivadent, Schaan, Liechtenstein). Both ceramics were low translucency A2 shade. The ceramics and their composition are described in Table 1 based on information obtained from their respective manufacturers. The surface gloss was evaluated to determine how the light reflects from the surface. The light transmission through the various ceramic thicknesses was evaluated at 396 nm, 406 nm, 447 nm, 454 nm, 465 nm and 476 nm wavelengths from three different light-emitting diode (LED) curing lights: a single-peak light, SmartLite Focus (Dentsply Sirona), that emitted light with a sharp peak wavelength at 476 nm and, two broad-spectrum multipeak lights VALO Grand (Ultradent, South Jordan, UT, USA), that emitted light with discrete peak wavelengths at 396, 447 and 465 nm and the Bluephase Style (Ivoclar Vivadent) that emitted peak wavelengths of light at 406 and 454 nm. The amount of violet and blue light through the different types and thicknesses of ceramic was determined using a laboratory-grade spectrometer (Ocean Insight, Orlando, FL, USA). A beam profiler (Ophir-Spiricon, Logan, UT, USA) was used to evaluate the beam profile through the ceramic thicknesses in discrete wavelength regions.

2.2 Sample preparation

The CAD/CAM blocks were sectioned into slices that ranged from 0.25-3.30 mm thick using a diamond disc and a precision saw (IsoMet 15LC, Buehler, Lake Bluff, IL, USA). IPS e.max CAD and Celtra Duo have different processing options. Celtra Duo can be either polished or glazed, while IPS e.max should always be glazed. Thus, the slices of Celtra Duo LT A2 were first evaluated without any surface treatment (WT). They were then randomly allocated to receive the processing treatments as recommended by the manufacturer. Some slices of IPS e.max LT A2 were evaluated without any surface treatment only to measure their surface gloss (WT). The light transmission was only measured after they had been glazed as recommended by the manufacturer.

Polishing (P): The slices were cleaned using detergent and water, and dried using a stream of clean air. One side only of the slice was subjected to a pre-polishing

step that used a diamond-polisher (#53 5990 2103, CELTRA Set, TwisTec, DeguDent, Dentsply Sirona). A diamond-polisher (#53 5990 2104, CELTRA Set, TwisTec, DeguDent, Dentsply Sirona), at a speed of 10,000 rpm using a light to medium pressure was used for the final polishing step. All slices were then cleaned with detergent and water to remove any polishing residues.

Polishing with diamond paste (PP): The Celtra Duo slices were cleaned, and one side only was polished following the same instructions as the P group. However, the final step added a fine diamond paste (Dialite Extra-Oral Polishing Paste, Brasseler USA, Savannah, GA, EUA) on a soft Robinson brush (#11/16 SOFT, Brasseler USA) at a speed of 8,000 rpm using a low hand pressure.

Glazing (G): Only one side of the Celtra Duo LT A2 were stained using Universal Stain and glazed, followed by Universal Overglaze (Dentsply Sirona). The ceramic discs were fired twice using the press furnace (Programat EP3000, Ivoclar Vivadent) to glaze the slices. The first firing temperature was at 820 °C, and the second firing was at 770 °C.

The IPS e.max CAD LT A2 slices were evaluated only after glazing because this ceramic would never be used in the blue, unfired condition. One side of each ceramic slice was treated using the IPS e.max CAD Crystallization/Glaze stain and glaze paste (Ivoclar Vivadent) before firing following the manufacturer's instructions (Ivoclar Vivadent) in a press furnace (Programat EP 5010, Ivoclar Vivadent) using the recommended crystallization program at 850 °C and a 20-minute cycle. The thickness of each ceramic slice was measured and was used when plotting the effect of thickness.

2.3 Surface gloss

The surface gloss (in gloss units [GU]) was measured with the Novo-Curve glossmeter (Rhopoint Instruments Ltd, Hastings, Sussex, UK) using a 60° angle of illumination and a 4.5 mm aperture. The equipment was calibrated with a standard plate (93.3 GU) provided by the manufacturer before making the measurements. The gloss was measured four times on the side that had been polished or glazed. The center of the sample was placed on the aperture and adjusted for each measurement. The gloss was measured eight times and

averaged after each treatment (WT, P, PP, and G) with the disc in different orientations over the sensor.

2.4 Light transmitted through the ceramics

The light transmission from each LCU through different ceramics, thicknesses, and polishing conditions was determined through a 10.8 mm by 12 mm aperture into a six-inch integrating sphere (LabSphere, North Sutton, NH, USA) that was connected to a fiberoptic spectrometer (USB 4000, Ocean Insight, Orlando, FL, USA). Before the measurements, the system was calibrated using the LS-1-CAL-INT calibration lamp (Ocean Insight). With the light source perpendicular to the surface, the light transmission through the Celtra Duo thicknesses was measured: WT, P, PP, and G. The light transmission through the IPS e.max CAD slices was determined after glazing. Three measurements of the spectral radiant power received between 350 and 550 nm were made for each condition. The percentage of light transmission was evaluated using discrete peak wavelengths at 396, 406, 447, 454, 465 and 476-nm. The transmission was calculated by dividing the power measured through the ceramic (x_2) by the maximum power recorded at that wavelength in the absence of the ceramic (x_1) (Figure 1).

The amount of violet and blue light was fitted using Microsoft Excel software (Microsoft Corporation, Redmond, Washington, USA), with the following formula that followed the Beer-Lambert law:

$$T(t) = T_0 e^{-t/d_0}$$

where T is transmission as a function of distance, T_0 is the transmission at zero thickness, t is thickness, and d_0 is a characteristic decay length called the extinction length.

2.5 Beam profile of the light transmitted through the ceramics

The light beam profiles of light transmitted through the 0.25-mm and 1.50-mm thick ceramic slices were measured using a laser beam profiler camera (USB-L070, Ophir-Spiricon, Logan, UT, USA) before treatment and after glazing. The LCU guide tip was placed against the polished or glazed side of the ceramic slice and the transmitted light beam was examined from the other unfinished side using

a digital camera with a 50 mm focal length lens (USB-L070, Ophir-Spiricon) that used a custom-made blue filter (International Light Technologies, Peabody, MA, USA) to flatten the spectral response of the CCD camera. Images of the transmitted light beam were taken through narrow bandpass filters that had a 10-nm full width half maximum bandwidth centered at either 400 ± 5 nm or 460 ± 5 nm (Edmund Industrial Optics, Barrington, NJ, USA). Images were collected using BeamGage Professional 6.14 software (Ophir-Spiricon).

2.6 Statistical analysis

The light transmission through Celtra Duo LT A2 was analyzed using a two-way analysis of variance (ANOVA) followed by a post-hoc Fisher's exact test to compare the effects of thickness and the different surface processing treatments at each wavelength. The light transmission through IPS e.max CAD LT A2 was analyzed by one-way ANOVA followed by post-hoc Fisher's exact test to compare the effects of thickness and surface processing treatments at each wavelength. The surface gloss data for Celtra Duo LT A2 was analyzed by one-way ANOVA, and the surface gloss data for both ceramics without any treatment and after glazing was analyzed by two-way ANOVA. Multiple comparisons were made using Tukey's test. The correlation between the surface gloss on light transmission for each wavelength for each ceramic type was tested using the Pearson correlation test. All tests were made using Sigma Plot version 13.1 (Systat Software, Inc. San Jose, CA, USA) with the significance level set at $\alpha = 0.05$. The beam profiling images were analyzed qualitatively.

3 Results

3.1 Surface gloss

There was a significant difference in the gloss between the various processing treatments and type of ceramic ($p < 0.001$). After glazing, Celtra Duo LT A2 ceramic had higher surface gloss values than the IPS e.max CAD LT A2. There was no significant difference between the PP and G groups of Celtra Duo LT A2 (Figure 2). Table 2 show that there was a weak correlation between gloss surface values and the percentage of light transmission through ceramic thicknesses, independent of the wavelength ranges evaluated ($R^2 < 0.25$).

3.2 Light transmitted through the ceramics

The thicknesses of the ceramics and the wavelengths of light all had a significant effect ($p < 0.001$) on the amount of light transmission; however, the various processing treatments had no significant difference on the light transmission at each wavelength. There was a rapid logarithmic decay ($R^2 > 0.97$) in the amount of light transmitted as the ceramic thickness increased. The lower wavelengths of light were more affected (Figures 3, 4, 5 and 6). Using a logarithmic curve fit, at 396 nm, the predicted light transmission through 0.25 mm of IPS e.max CAD LT and through Celtra Duo LT A2 was 41% and 47%, respectively. Table 3 shows that only 6% of the light at 396 nm passed through 1.50 mm of both ceramics, and 1% or less of the original light at 396 nm could be detected through 3.00 mm of each ceramic. At the longer wavelengths of light, more light was transmitted through Celtra Duo LT A2 than through IPS e.max CAD LT (Figure 5). At 476 nm, the amount of light transmission through 0.25 mm of IPS e.max CAD LT and through Celtra Duo LT A2 was 55% and 67%, 21% and 26% through 1.50 mm of ceramic, but only 6% and 9% passed through 3.00 mm of each ceramic, respectively. When the full range of the wavelengths was analyzed, there was a rapid logarithmic decay until 1 mm thickness of both ceramics and the SmartLite Focus showed higher light transmission at thicker ceramics (Figure 3). The emission spectra of the LCU's light transmission through the ceramic thicknesses are presented in Figure 4.

3.3 Beam profile of the light transmitted through the ceramics

The three-dimensional representations of the beam profile recorded from the Bluephase Style, SmartLite Focus and VALO Grand through Celtra Duo LT A2 (before and after glazing) and IPS e.max CAD LT A2 (after glazing) through the narrow bandpass filters are shown in Figure 7. The beam profile from the Bluephase Style used in the present study was less homogeneous than the VALO Grand. Less light was transmitted through IPS e.max CAD LT A2 after glazing compared to Celtra Duo LT A2 after glazing. The images supported the light transmission measurements and clearly show the effect of ceramic thickness on the amount of transmitted light and how the shorter wavelengths are more affected than longer wavelengths of light.

4 Discussion

This study determined the influence of different types of ceramic, thicknesses, and surface conditions on the light transmission at six discrete wavelengths (in the violet and blue wavelengths). As the ceramic thickness increased, there was a rapid logarithmic decay in the amount of light transmitted through the different types of ceramic. The processing treatments did not influence the light transmission. Therefore, the first hypothesis was rejected.

The light was logarithmically attenuated ($R^2 > 0.97$) as the ceramic thickness increased, as described by the Beer-Lambert law, at all the wavelengths of light that were evaluated [17, 21]. Thus, the second hypothesis was rejected. This attenuation was greater at the shorter wavelengths than longer wavelengths of light. This is thought to occur because the lower wavelengths of light are more highly scattered as they pass through the ceramic [17]. A similar finding was reported in a previous study that showed that the thickness and the wavelength of light affected the light transmission through resin-based composites [23]. These aspects may impair the light activation of resin cements that use Norrish Type I photoinitiators that require this violet light [17, 18].

Although the blue light emitted from different LCUs may appear to be the same to the human eye [24], when the light output is measured using a spectrophotometer, most LCUs emit very different emission spectra (wavelength of light). In this study, six wavelength peaks in the violet and blue wavelengths could be identified in the three commercial LED LCUs. These wavelengths illustrate the wide range of wavelengths that are delivered from contemporary LCUs and highlight that great differences exist in the output from dental LCUs.

To provide a range of contemporary ceramics used in CAD/CAM Dentistry, a zirconia-reinforced lithium silicate (Celtra Duo) and a lithium disilicate glass-ceramic (IPS e.max CAD) were tested. The Celtra Duo has 10% of zirconia embedded into the lithium silicate (Li_2SiO_3) glass matrix. This results in smaller silicate crystals leading to a higher translucency and higher glass content than found in conventional lithium disilicate glass-ceramics [25]. In a previous study, the percentage of light transmitted decreased irrespective of the ceramic

composition and the wavelength analyzed [21]. In another study, Celtra Duo showed higher translucency than IPS e.max CAD, but only when polished [25]. In the present study, a slightly amount of light was transmitted through and observed in the beam profile images of glazed Celtra Duo.

To adequately polymerize resin cements, enough light must penetrate through the ceramic and reach the resin cement. The radiant exposure is affected by different conditions, and at the shorter wavelengths, very little light is transmitted through the ceramic (less than 1 J/cm²) [26]. However, these data were not evaluated individually, and they are included in the total radiant exposure of the LCUs that also delivered the longer wavelengths of blue light [26]. When using the full range of light output from the LCU, the minimum radiant exposure delivered to achieve adequate polymerization of the light-cured resin cement has been reported to be 6 J/cm² [27]. Light-cured resin cements have been reported to reach an adequate polymerization when the ceramic is no more than 1.0 mm thick, and the mechanical properties of both light-cured and dual-cured resin cements have been reported to be unaffected when the ceramic is between 0.3 and 0.9-mm thick [28, 29]. The present study presented a rapid logarithmic decay until 1 mm thick using all LCUs (Figure 3). Other studies have reported that the ceramic should be no more than 3 mm thick for both light-cured and dual-cured resin cements [10, 30]. In the present study, the percentage of light transmitted showed that only 31% and 38% of the light was transmitted through 1 mm thick IPS e.max CAD LT A2 and Celtra Duo LT A2, respectively, at the longer wavelengths, and only 13% of the light was transmitted through both ceramics at the shorter wavelengths. At 3 mm thick, SmartLite Focus presented better performance than the other LCUs which can be explained by the presence of the longer wavelength in this LCU. The beam profile images (Figure 7) confirmed that very little light was transmitted through the 1.5 mm thick glazed ceramics. Thus, when the ceramic thickness is 1.5 mm and above, dual-curing resin cements should be used [31]. The angulation and distance of the LCU from the ceramic will also negatively affect the light transmission and the polymerization process [32]. Also, the radiant exposure is affected by the position and exposure distance of the LCU from the target [26].

The method used to evaluate the logarithmic decay of the transmission with ceramic thickness is based on a formula according to the Beer-Lambert law. The calculated percentages of light transmission at 396, 454, and 476-nm wavelengths through IPS e.max CAD LT A2 and Celtra Duo LT A2 are reported in Table 3. From this table, the clinician can estimate how much light will be transmitted based on the type and thickness of their ceramic. They can then estimate for how long they will have to have the LCU on to deliver 6 J/cm² [27].

Regarding the processing treatments, CAD/CAM restorations are usually made in a laboratory with a final glazed surface created by firing in an oven (e.g., IPS e.max CAD). However, other chairside CAD/CAM ceramics, as Celtra Duo, can just be hand-finished and polished [33]. Finishing and polishing steps have been reported to improve the esthetics of the milled ceramic, resulting in similar refraction and reflection characteristics of a natural tooth and can produce a glossy surface [33]. Highly polished and smooth surfaces will decrease bacterial adhesion [34] and minimize abrasive wear of the antagonist teeth or restorations [35]. Also, according to another study [35], highly polished surfaces may produce less antagonist wear than glazed surfaces. Despite the processing treatment, it is recommended that after any adjustment, the ceramic should receive a final high-gloss polishing to recover the smooth surface and to provide optimal clinical performance [36, 37]. According to the American Dental Association (ADA) [38], a surface gloss of 40 to 60 GU is considered adequate for dental restorations. This gloss level was exceeded for both glazed ceramics and for Celtra Duo LT A2 when it was polished with the diamond paste. A previous study has also reported that the polished surface of ceramic can be smoother than the glazed ceramic surface [33]. The polishing technique can alter the surface of indirect restorations [39]. However, the surface gloss did not affect the light transmission, and only a low correlation between surface gloss and the percentage of light transmission was found ($R^2 < 0.25$).

In vitro studies have their limitations, and further studies are necessary to determine the effect of light transmission on the polymerization of light and dual-curing resin cements. Also, the light transmitted through other types of ceramics with higher opacity, such as 3Y-zirconia and alumina-based ceramics, should be studied as the light transmission may be even more affected by the thickness of

these ceramics. However, based on the present study, the clinician should recognize that violet light will be attenuated at a faster rate than blue light and any benefits of including violet light from the LCU will be quickly lost as the thickness of ceramic increases. In the region of 0.5 to 1.0 mm thickness, even small increases in the thickness of the ceramic will have a large effect on the amount of transmitted light, and that little light is transmitted through more than 1.5 mm of ceramic.

5 Conclusion

Within the limitations of this *in vitro* study, the following conclusions can be drawn:

1. There is a rapid logarithmic decay in the amount of light transmitted through the ceramic as the thickness increases. This decay occurs faster at the shorter wavelengths.
2. Celtra Duo glazed ceramic had higher surface gloss values compared to e.max CAD ceramic. However, there was no difference between Celtra Duo polished with diamond paste and glazed Celtra Duo.
3. There was no correlation between the surface gloss and the amount of light transmitted.

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Tables

Table 1. Ceramics used in the study (data provided by the manufacturers).

Ceramic Material	Classification	Composition	Lot No.	Manufacturer
<i>IPS e.max CAD LT A2</i>	Lithium disilicate glass-ceramic	SiO ₂ , Li ₂ O, K ₂ O, MgO, Al ₂ O ₃ , P ₂ O ₅ and other oxides	X42573	Ivoclar Vivadent, Schaan, Liechtenstein
<i>Celtra Duo LT A2</i>	Zirconia-reinforced lithium silicate ceramic	SiO ₂ , Li ₂ O, ZrO ₂ (~10%), P ₂ O ₅ , Al ₂ O ₃ , K ₂ O, CeO ₂ , pigments	18031165	Dentsply Sirona, York, USA
Abbreviations: Al ₂ O ₃ , aluminum trioxide; CeO ₂ , cerium dioxide; K ₂ O, potassium oxide; Li ₂ O, lithium oxide; MgO, magnesium oxide; P ₂ O ₅ , phosphorus pentoxide; SiO ₂ , silicon dioxide; ZnO, zinc oxide; ZrO ₂ , zirconium dioxide.				

Table 2. Pearson correlation between surface gloss value and the percentage of light transmission at six discrete wavelengths (nm).

Wavelength (nm)	r	r ²	P value
396	- 0.109	0.012	0.223
406	- 0.119	0.014	0.186
447	- 0.147	0.019	0.101
454	- 0.139	0.021	0.119
465	- 0.147	0.025	0.101
476	- 0.149	0.022	0.096

Table 3. Predicted light transmission (%) at the 396, 454 and 476-nm wavelengths.

Ceramic	Predicted Light Transmission (%)																					
	396							454							476							
	0.25	0.50	1.00	1.50	2.00	3.00	R²	0.25	0.50	1.00	1.50	2.00	3.00	R²	0.25	0.50	1.00	1.50	2.00	3.00	R²	
	mm	mm	mm	mm	mm	mm		mm	mm	mm	mm	mm	mm		mm	mm	mm	mm	mm	mm		mm
IPS e.max CAD LT A2	41	28	13	6	3	1	0.994	53	42	27	17	11	5	0.985	55	45	31	21	14	6	0.974	
Celtra Duo LT A2	47	31	13	6	2	0	0.996	62	51	33	22	14	6	0.996	67	56	38	26	18	9	0.996	

Abbreviations: R², is the coefficient of determination what is the proportion of the variance in the dependent variable that is predictable from the independent variable; 396 nm, is the peak of the short wavelength of light emitted from the VALO Grand; 454 nm is the peak of the long wavelength of light emitted from the Bluephase Style; 476 nm is the single-peak of blue light emitted from the SmartLite Focus.

Table 4. Radiant power (mW) through glazed ceramic thicknesses using VALO Grand, Bluephase Style and SmartLite Focus.

Ceramic	Light-Curing Units																	
	VALO Grand: Average Power at the Top Surface 863 mW						Bluephase Style: Average Power at the Top Surface 603 mW						SmartLite Focus: Power at the Top Surface 413 mW					
	<i>0.25 mm</i>	<i>0.50 mm</i>	<i>1.00 mm</i>	<i>1.50 mm</i>	<i>2.00 mm</i>	<i>3.00 mm</i>	<i>0.25 mm</i>	<i>0.50 mm</i>	<i>1.00 mm</i>	<i>1.50 mm</i>	<i>2.00 mm</i>	<i>3.00 mm</i>	<i>0.25 mm</i>	<i>0.50 mm</i>	<i>1.00 mm</i>	<i>1.50 mm</i>	<i>2.00 mm</i>	<i>3.00 mm</i>
IPS e.max CAD LT A2	430	324	158	142	64	38	318	242	122	111	49	29	240	187	104	102	51	32
Celtra Duo LT A2	501	438	239	173	126	39	379	301	187	138	90	30	276	230	161	123	85	35

Figures

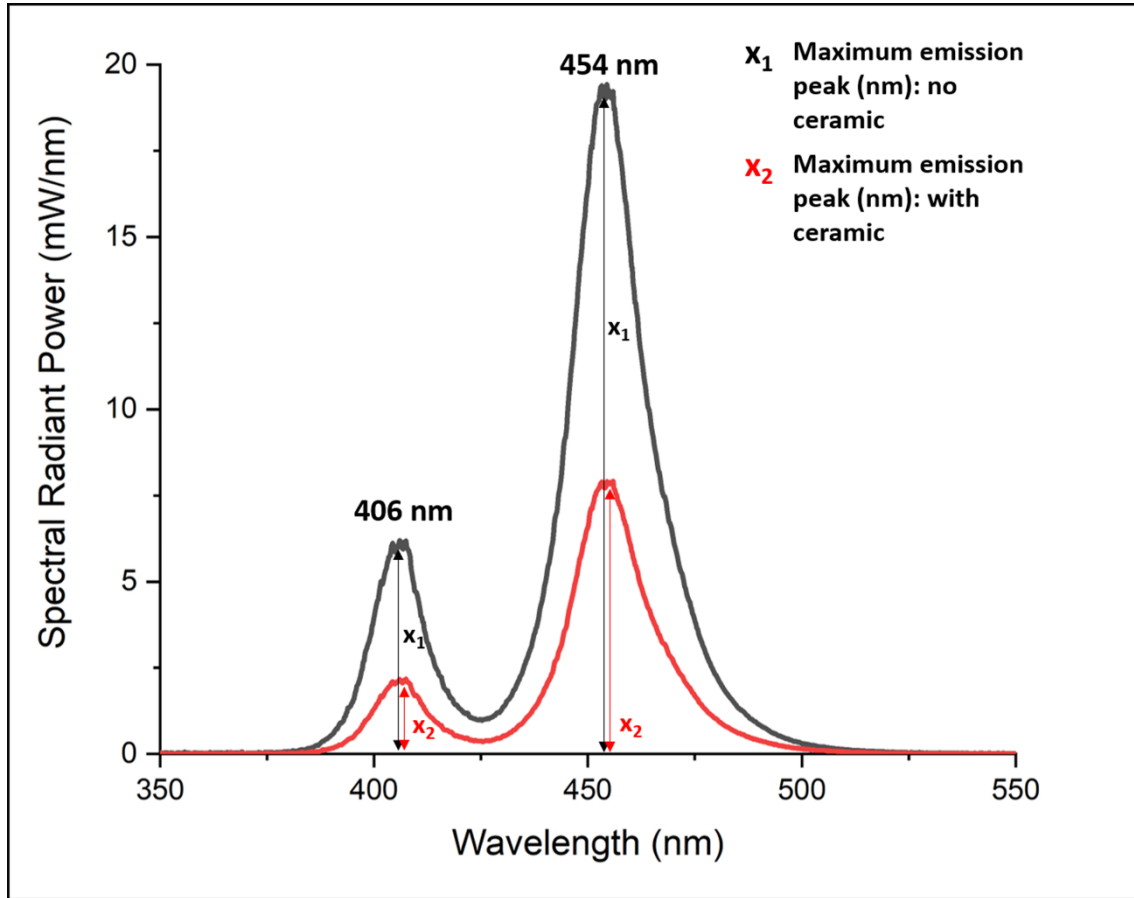


Figure 1. Representative diagram of maximum peaks that were used to calculate the light transmission through the ceramics. The percentage of light transmission was evaluated using discrete peak wavelengths of light at 396, 406, 447, 454, 465 and 476-nm. The transmission of light was calculated by dividing the power measured through the ceramic (x_2) by the maximum power recorded at that wavelength in the absence of ceramic (x_1). Note the ceramic thickness present in this diagram is 0.50 mm (t).

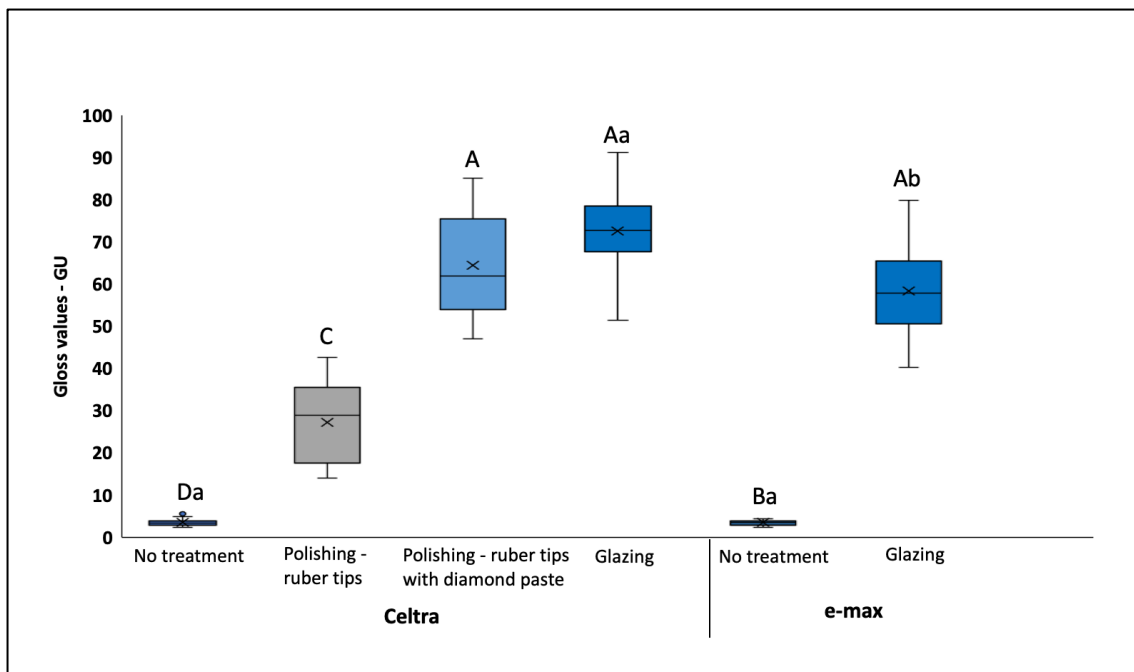


Figure 2. Surface gloss values measured for both ceramics after different processing treatments. Upper case letter used to compare different processing treatments for each ceramic; lower case letter used to compare between the same processing treatments for the different ceramics ($P < 0.05$).

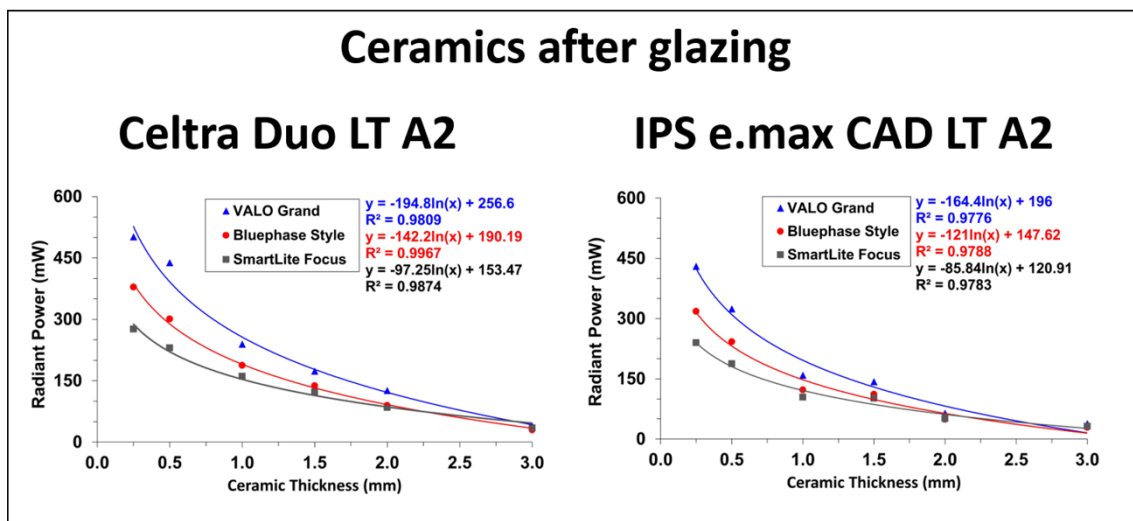


Figure 3. Comparison of the radiant power (mW) at the full range of the wavelengths of VALO Grand, Bluephase Style and SmartLite Focus. Note the effect of thickness and the wavelength on the transmission of light through the ceramics.

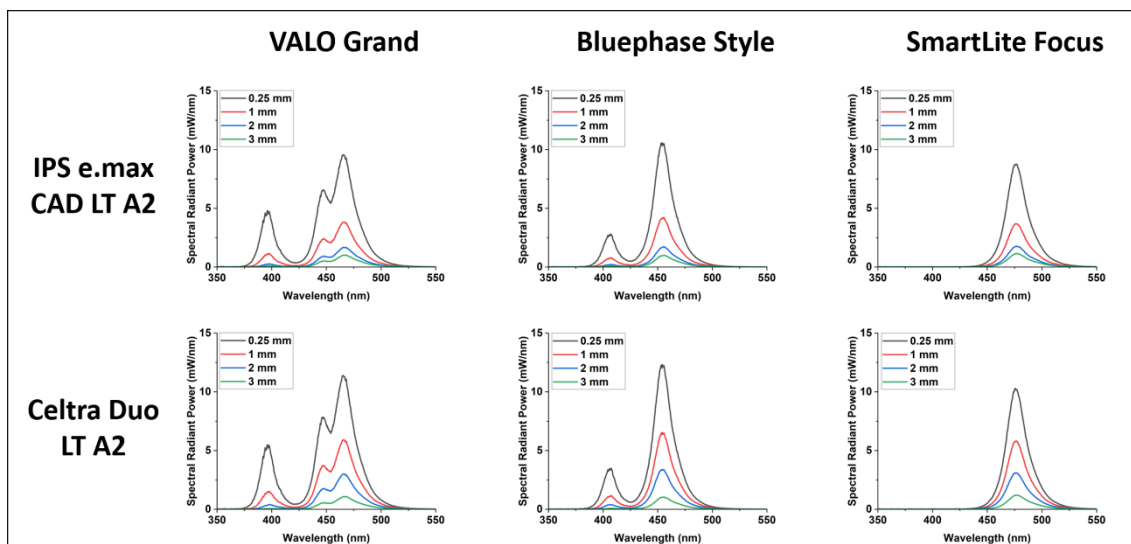


Figure 4. Comparison of the emission spectra (mW/nm) at the full range of the wavelength of VALO Grand, Bluephase Style and SmartLite Focus. Note the effect of thickness and the wavelength on the transmission of light through the ceramics.

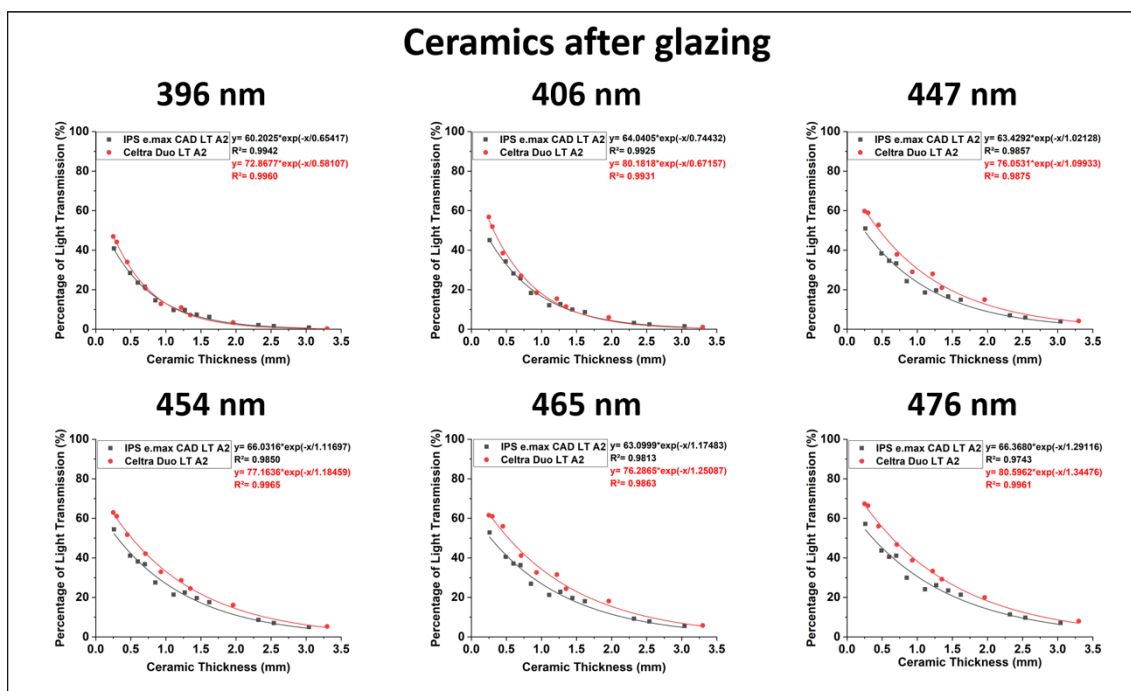


Figure 5. Percentage of light transmission (%) at each wavelength. Note the effect of thickness and the effect wavelength on the transmission of light through the ceramics.

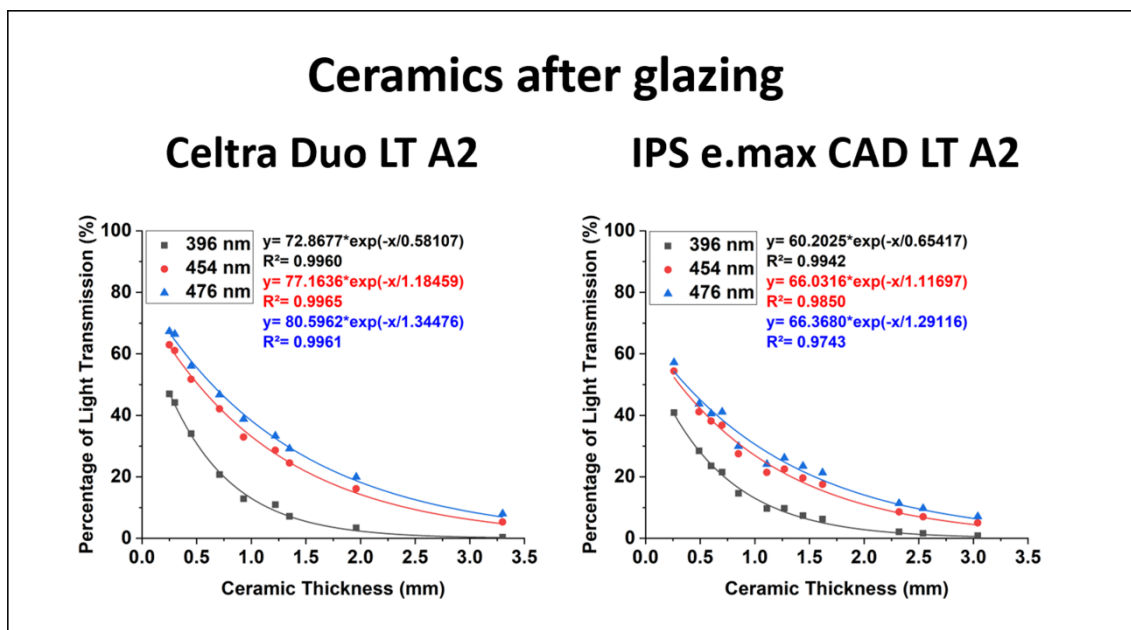


Figure 6. Comparison of the light transmission (%) at 396, 454 and 476-nm. Note the effect of thickness and the wavelength on the transmission of light through the ceramics.

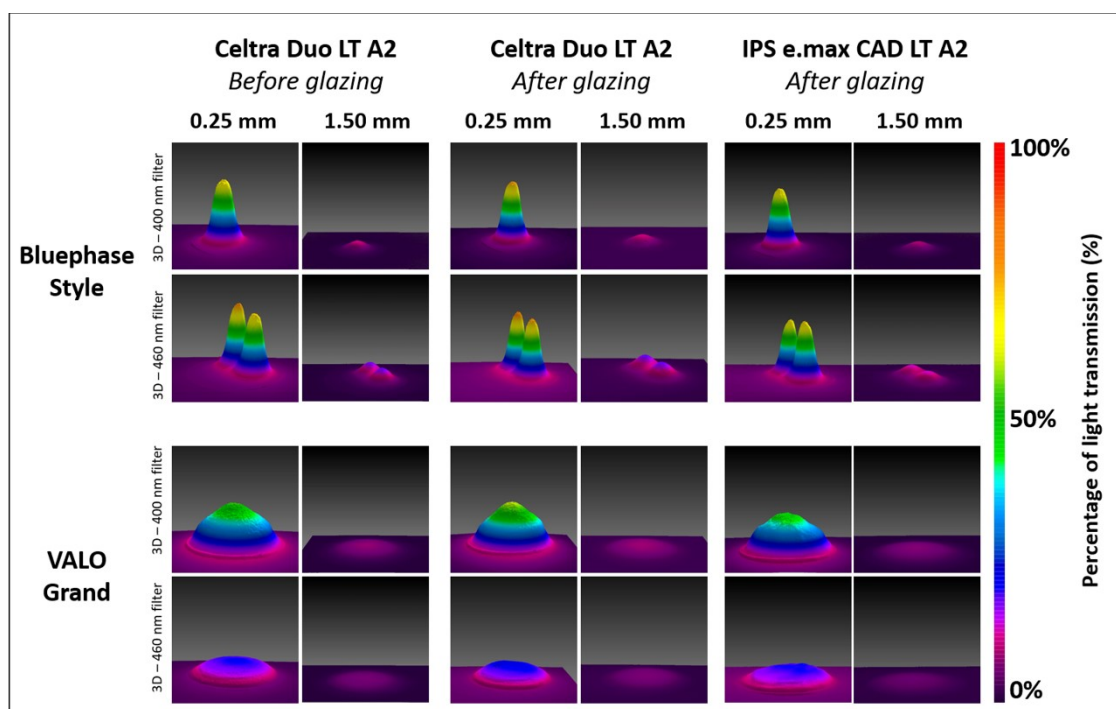


Figure 7. The three-dimensional representations of the beam profile recorded through a 400 nm bandpass filter (violet light), and through a 460 nm bandpass filter (blue light) from the standard output mode of Bluephase Style, and VALO Grand through Celtra Duo LT A2 (before and after glazing), and through IPS

e.max CAD LT A2 (after glazing). Note the images taken through the 400 and the 460 nm bandpass filters are on their own individual percentage scales specific to that wavelength.

Capítulos

3.3 CAPÍTULO 3

Effect of the additional violet light from the light-curing unit on two resin cements that use different photoinitiators

Artigo submetido para publicação no periódico **Dental Materials**

Effect of the additional violet light from the light-curing unit on two resin cements that use different photoinitiators

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Running Title: Violet light through resin cements and ceramic

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Highlights

- Very little of the violet light passed through 1.5 mm of e.max ceramic to reach the resin.
- Depending on the photoinitiator used in the resin, polywave® lights may not be required.
- Future studies should report the emission spectrum and radiant exposure received by the resin.

Effect of the additional violet light from the light-curing unit on two resin cements that use different photoinitiators

ABSTRACT

Objectives. To evaluate the effect of delivering violet, blue, or a combination of violet and blue light through ceramic on the degree of conversion (DC) of two resin cements that use different photoinitiators.

Methods. A Bluephase Style (BS, Ivoclar Vivadent) polywave[®] light-emitting diode (LED) light-curing unit (LCU) was used to deliver a total of 6.4 J/cm² of violet and blue light (385-515 nm). This was comprised of 1.4 J/cm² of violet (385-420 nm) and 5.0 J/cm² of blue light (420-515 nm). The LED emitters in a second modified BS LCU were connected directly to a power supply so that either just violet (1.4 J/cm²) or just blue (5.0 J/cm²) light was emitted. RelyX Veneer (RV, 3M) and Variolink Esthetic LC (VE, Ivoclar Vivadent) resin cements were photo-cured through either 0.5 or 1.5 mm of IPS e.max CAD (Ivoclar Vivadent). The DC was monitored using Fourier-Transform Infrared spectroscopy (Vertex 70, Bruker). Data were subject to a three-way analysis of variance followed by Tukey test ($\alpha = 0.05$).

Results. All factors had a significant effect ($P < 0.001$). Increasing the ceramic thickness decreased the DC most notably for RV when just violet light was delivered. There was no significant difference ($P = 0.948$) in the DC of RV when just the blue, or a combination of violet and blue wavelengths of light were delivered. Adding the violet light produced a small but significant increase in the DC of the VE cement.

Significance. A polywave[®] light is not required to photo-cure VE, and RV did not benefit from receiving the additional 1.4 J/cm² of violet light from the LCU.

Keywords: resin cements, ceramic thicknesses, degree of conversion, light-curing unit, violet light, photoinitiators, multi-peak.

1 Introduction

The use of indirect ceramic restorations cemented adhesively using light-cured resin cement has become increasingly popular. Currently, both light and dual-cure resin cements require light to reach an adequate level of polymerization [1]. These resin cements may use more than one type of photoinitiator system. Camphorquinone (CQ) is a commonly used Type II initiator, and it is most sensitive to the longer wavelengths of blue light. Some new materials that use additional amine-free Type I photoinitiators such as diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide (TPO), or the new germanium-based photoinitiator (Ivocerin®) have shown better color stability [2] because these resin cements contain less or even no CQ.

While the absorption peak of CQ is at 468 nm, the absorption peak for TPO is in the violet range and extends down into the UV-A region [3]. Thus, to light cure restorative materials that use a combination of these Type I and Type II initiators, it has been recommended that the light-curing unit (LCU) deliver a broad range of wavelengths of light (400 to 500 nm). The newest germanium-based initiator, Ivocerin®, also has a peak absorption near 410 nm in the violet spectrum, but it also can be activated by light up to 460 nm [4]. When quartz tungsten halogen LCUs were used, this was not a problem because they emit a broad range of wavelengths of light. However, nowadays, most dentists use light-emitting diode (LED) curing lights that produce a much narrower range of wavelengths. Often, a single LED with a full width half maximum emission (FWHM) range that is less than 25 nm is used in the LCU. Consequently, these LCUs cannot deliver both violet and blue wavelengths of light. To overcome this problem, some manufacturers have introduced polywave® LED curing units that contain several different LED emitters to produce a broader range of wavelengths of light [5]. While these multiple peak LED curing lights can now activate the resin cements that use other photoinitiators [6], there is a logarithmic decay in the overall irradiance received by the resin as the ceramic thickness increases [7,8].

Furthermore, the shorter wavelengths of violet light do not penetrate through indirect restorative materials as well as the longer wavelengths of blue light [8,9].

This lack of penetration may adversely affect the degree of conversion and microhardness of the resin [9], and it may increase the toxicity of resin cements [10] that require these shorter wavelengths. However, so far, no studies have separated the individual effects of delivering just the violet or just blue light from a commercial LED curing light on resin polymerization through the ceramic.

The purpose of this in-vitro study was to evaluate the effect of the photo-curing the resin using just the violet, the blue, or a combination of violet and blue wavelengths delivered from a polywave® LCU on the degree of conversion (DC) of two resin cements that use different photoinitiators through two thicknesses of a lithium disilicate glass-ceramic. The research hypotheses tested were that:

- 1) the different ceramic thicknesses would not affect the degree of conversion of light-cured resin cement;
- 2) the degree of conversion of light-cured resin cements that use different photoinitiators would not be influenced by the wavelengths of light from a polywave® LCU.

2 Materials and Methods

2.1 Study Design

Two light-cured resin cements that use different photoinitiators were used in this study: RelyX Veneer translucent shade (RV, 3M Oral Care, Saint Paul, MN, USA) that uses CQ as the photoinitiator; and Variolink Esthetic LC neutral shade (VE, Ivoclar Vivadent, Schaan, Liechtenstein). The resin cements and their compositions, as provided by their manufacturers, are described in Table 1. The RV and VE cements were light-cured at a distance of 0 mm, or through two thicknesses (0.5 and 1.5 mm) of glazed, low translucency, A2 shade IPS e.max CAD lithium disilicate glass-ceramic (Ivoclar Vivadent). The composition of the ceramic, as provided by their manufacturer, is described in Table 2. The DC was monitored using Fourier-Transform mid-Infrared Spectroscopy.

2.2 The adjustment of the light output from the modified Bluephase Style

Ivoclar Vivadent provided a modified Bluephase Style (BS, Ivoclar Vivadent) curing light that had the LED emitters attached directly to a DC power supply (GPS-3030DD, GW Instek, New Taipei City, Taiwan) so that it could deliver light

from just the violet LED (385-420 nm), or from just the blue LED (420-515 nm) light (Figure 1). A second unmodified Bluephase Style (385-515 nm) curing light was used to emit the full range of wavelengths from this LCU. Using a laboratory-grade spectrometer (Ocean Insight, Orlando, FL, USA) to monitor the light output, the violet and the blue outputs of the modified LCU were adjusted to match the power output from the unmodified Bluephase Style (Ivoclar Vivadent). The same light guide was used on both LCUs. The current to the violet and the blue LED emitters was adjusted to match the light output emitted at both the violet and blue wavelength ranges from the unmodified BS light. The spectral radiant powers of the modified and unmodified BS curing lights were measured three times before and three times after the degree of conversion (DC) tests. The light output was measured through a 6.4 mm aperture into a six-inch integrating sphere (LabSphere, North Sutton, NH, USA) connected to a fiberoptic spectrometer (USB 4000, Ocean Insight, Orlando, FL, USA). This 6.4 mm aperture was chosen to correspond to the 6 mm diameter of the resin specimens. The emission spectra of the three light conditions evaluated in this study are shown in Figure 2, and the characteristics of the lights are reported in Table 3.

2.3 Ceramic veneer fabrication

The CAD/CAM block of IPS e.max LT A2 was sectioned into two slices that were 0.5 and 1.5 mm thick using a diamond disc and a precision saw (IsoMet 15LC, Buehler, Lake Bluff, IL, USA). One side of each ceramic slice was then glazed following the manufacturer's instructions (Ivoclar Vivadent) with crystallization and glazing carried out in one step using IPS e.max CAD Crystallization/Glaze paste and stain paste (Ivoclar Vivadent) in a press furnace (Programat EP 5010, Ivoclar Vivadent) for 20 minutes. The glazed IPS e.max CAD LT A2 slices were interposed between the resin cements and LCU tips during the DC tests.

2.4 Sample preparation of the resin cements

The tests were performed in a controlled condition with either 0 mm between the LCU tip and resin cement or with a slice of either 0.5 or 1.5 mm thick glazed IPS e.max CAD LT A2 ceramic interposed between the LCU tip and the RV and VE resin cements (Figure 3). A 0.1 mm thick mylar strip (DuPont Teijin Films, Chester, VA, USA) with a 6 mm diameter hole was placed on the Attenuated Total Reflectance (ATR) crystal of the Vertex 70 (Bruker Optik GmbH, Ettlingen,

Germany). The resin cement was inserted into the 6 mm diameter hole in the 0.1 mm thick mylar strip to produce resin specimens that were all 0.1 mm thick. Another mylar strip was placed over the resin cement, and for the 0 mm control specimens, a microscope slide (Microscope Cover Glass, Mahwah, NJ, USA) was used to press the material against the ATR crystal and the cement was photocured, and then removed before the photocure. For the 0 mm control specimens, the cement was photocured with the LCU tip touching the mylar strip that covered the cement specimen. Alternatively, the slice of either 0.5 or 1.5 mm thick glazed IPS e.max CAD LT A2 ceramic was interposed between the LCU tip and the RV and VE resin cements and the cement was photocured through the ceramic. Five specimens were made for each condition.

2.5 Degree of Conversion (DC)

The DCs of the RV and VE resin cements were evaluated after they had been photocured using just the violet, just the blue, or a combination of violet and blue light from the curing light ($n = 5$). To not affect the resin cement polymerization, the specimens were prepared directly over the ATR crystal in a dark room with yellow light. The DCs were assessed using FT-mid-IR (Vertex 70, Bruker Optik GmbH, Ettlingen, Germany) with a Golden Gate ATR crystal (Specac Ltd, Orpington, Kent, UK) attachment. The spectra were obtained between internal standard aromatic C = C bonds (1608 cm^{-1}) and aliphatic C = C bond (1638 cm^{-1}), at a resolution of 4 cm^{-1} . All the experiments were performed under controlled temperature ($23 \pm 1\text{ }^{\circ}\text{C}$) and humidity ($60 \pm 5\%$) conditions. The DC at 5 minutes after the resin was exposed to light was calculated using the average of the last 40 scans ($t = 5\text{ s}$) from the equivalent aliphatic (1638 cm^{-1}) and aromatic (1608 cm^{-1}) ratios of cured (C) and uncured (U) resin composite specimens. The formula used to calculate the degree of conversion was $\text{DC (\%)} = (1 - \text{C/U}) \times 100$.

2.6 Statistical Analysis

The DC data (%) of RV and VE were analyzed using a three-way analysis of variance (ANOVA) to compare the effects of the wavelengths of light (3 levels), ceramic thickness (2 levels), and resin cement (2 levels). Post-hoc multiple comparisons were made using Tukey's test. All tests used $\alpha = 0.05$ significance level using Sigma Plot version 13.1 (Systat Software, Inc. San Jose, CA, USA).

3 Results

3.1 *Modified and Unmodified Bluephase Style Outputs*

Table 3 reports the emitted radiant power (mW), calculated irradiance (mW/cm²), and radiant exposure (J/cm²) values from each of the three light conditions through a 6.4 mm aperture into the integrating sphere. The unmodified polywave® BS light had a violet light peak emission at 406 nm and a full width half maximum (FWHM) from 398 to 413 nm, and blue light peak emission at 454 nm with a FWHM emission spectrum from 445 to 465 nm. The violet light had a peak emission at 412 nm and a FWHM from 404 to 420 nm. The blue light had a peak emission at 457 nm (FWHM 447 to 467 nm). These small differences between the two LCU were within the manufacturer's specifications. In 10 s, the unmodified BS delivered 6.4 J/cm² at the 0 mm distance, the blue light delivered 5.0 J/cm², and the violet light delivered an additional 1.4 J/cm². The irradiance from the modified BS was 138 mW/cm² when only violet light was delivered, which corresponds to a radiant exposure of only 1.4 J/cm² and 507 mW/cm² or a radiant exposure of 5.0 J/cm² when just the blue light was emitted.

3.2 *Percentage of Degree of Conversion (%) comparing lights, materials, and ceramic thicknesses*

The DC means and standard deviation values are reported in Figure 4. The two resin cements behaved differently to the violet light component from the LCU ($P < 0.05$). Three-way ANOVA revealed that the resin cement ($P < 0.001$), the ceramic thickness ($P < 0.001$), and the light wavelengths ($P < 0.001$) factors and all the different combinations of factors had a significant effect on the DC values ($P < 0.001$). As the ceramic thickness increased, there was a decrease in the DC for both resin cements. However, the DC of VE was affected less than the DC of RV as the ceramic thickness increased.

Delivering just the violet portion did not cure RV cement very well, notably through 1.5 mm of IPS e.max CAD LT A2 when the DC was 26 % compared to a DC of 75 % for VE. Delivering just the violet component of light was significantly ($P < 0.001$) worse than delivering just the blue or when the polywave® light was used. For both thicknesses of ceramic, there was no significant difference (Tukey test

$P = 0.948$) in the DC of RV when just the blue or a combination of violet and blue wavelengths of polywave® light were delivered.

The DC of VE was not significantly affected ($P = 0.898$) when just the violet component or the light from the unmodified polywave® light was delivered. Delivering blue light alone will cure VE, but using either just violet or a combination of violet and blue light (polywave®) will produce a small increase in DC, even though 1.5 mm of ceramic ($P < 0.05$).

4 Discussion

This study determined the effect of delivering 1.4 J/cm² of just violet light, 5.0 J/cm² of just blue light or a 6.4 J/cm² in a combination of violet and blue light wavelengths from a polywave® LCU on the DC of two light-cured resin cements through two thicknesses (0.5 and 1.5 mm) of a lithium disilicate glass-ceramic. As the ceramic thickness increased, there was a small decrease in DC for both resin cements. This decrease was much greater for the RV cement when only violet light was delivered. Thus, the first hypothesis was rejected ($P < 0.05$). This supported previous work [11] and previous studies that reported the mechanical properties of both light- and dual-cured resin cements were not affected when the ceramic was less than 0.9 mm thick [6,12].

The degree of conversion of RV and VE was affected by the wavelengths of light from the LCU. This effect was much greater for the RV cement when only violet light (412 nm with a FWHM 404 to 420 nm) was delivered. Therefore, the second hypothesis was rejected ($P < 0.05$). The lowest conversion result (26 %) was the RV resin cement when it was exposed to just violet light through 1.5 mm of IPS e.max CAD. This occurs because shorter wavelengths of light are affected more by the Rayleigh scattering effect and cannot penetrate through the restorative materials as well as the longer wavelengths of blue light [8,9]. This is highly relevant because RV uses only CQ as its photoinitiator. It is activated mainly by the longer wavelengths of blue light, with a peak absorption at 468 nm, and the lower wavelengths of violet light are less able to activate CQ. Consequently, RV has higher DC results (66 %) when only the blue light (457 nm with a FWHM emission spectrum 447 to 467 nm) or the polywave® light was delivered. Of note, RV did photocure when only violet light was delivered, but the maximum DC was

only 58 % at the 0 mm distance showing that violet light is less able to activate CQ compared to blue light (DC=66%).

CQ is the most widely used photoinitiator in dental resins. However, the bright yellow color of the CQ can cause undesirable discoloration through thin ceramic veneer restorations. To tackle this issue, the Norrish Type I photoinitiators were developed. Most of these initiators require the lower wavelengths of violet light to be activated [3]. Variolink Esthetic LC was chosen for this study because it is amine-free and uses only the germanium-based system, Ivocerin[®], Norrish Type I photoinitiator. Due to the wider absorption range of Ivocerin[®], which ranges from 390 to 460 nm [3], this initiator appears to be more forgiving to the wavelengths of light that it receives. VE will photocure when it receives blue light only (FWHM from 445 to 465 nm) and as previously reported [6], there is only a very small increase in DC when the violet light was delivered.

The unmodified LCU tested in the present study is a multiple peak third-generation LCU that has three LED emitters (two emitting blue light and one emitting violet light) [3]. This LCU has a heterogeneous beam profile when a regular light guide is used instead of the more recently introduced homogenizer light guide [13]. The violet component of the unmodified LCU corresponded to a radiant exposure of 1.4 J/cm² in 10 s at the 0 mm distance. This value was similar to another study that reported a radiant exposure of 1.9 J/cm² under similar conditions [14]. However, even this amount of violet light was sufficient to produce a small increase in the DC of VE. However, this increase may not be clinically relevant because even just the blue wavelengths of light from the LCU can activate the Ivocerin[®] photoinitiator [6]. Unless the LCU delivers mostly the longer wavelengths of blue-green light (e.g., those LCUs that peaking at 476 nm) and which deliver little light below 460 nm [15], this Ivocerin[®] initiator can be activated by a wide range of contemporary LCUs [3].

There is a wide range in the emission spectra from contemporary LCUs [15,16] or even among LCUs from the same manufacturer due to differences in the LED emitters used in the LCUs. This occurs because LED emitters that have a tighter color, lumen output, or forward voltage specification are built from full reels of LEDs. Such LEDs will have identical 'bin' numbers, and these LEDs cost more.

To reduce costs, manufacturers may choose to use LEDs that have lower quality bin numbers where the LEDs come from different reels and batches from the manufacturer. In an attempt to overcome this problem, both LCUs used in the present study both came from the same manufacturer and used the same optics. Even so, the violet peaks had FWHM values near 16 nm, whereas the blue peaks had broader FWHM close to 20 nm. Although this result may be manufacturer-specific, a previous study also reported that the violet LED emitters had a narrower FWHM than the LED emitters that deliver longer wavelengths [16].

In the unmodified LCU used in the present study, the contribution of the blue peak was 3.6 times greater than the violet peak on the High output setting of LCU (5 J/cm² compared to the violet light component of 1.4 J/cm²). In dentistry, the heat generated when light-curing is a concern [17,18]. The fact that the manufacturer knows that most of the violet light is effectively blocked from reaching the resin and the fact that the manufacturer uses Ivocerin[®] which has a broader absorption spectrum may help to explain why the emission spectrum in the violet range from LCUs made by this manufacturer (Ivoclar Vivadent) does not increase as the overall output is increased; only the output from the blue LED chip changes [16]. The manufacturer knows that the energy of a photon is equal to its frequency multiplied by Planck's constant [19]. Photons delivered at the shorter violet wavelengths will have a higher frequency and more energy. Consequently, they will raise the temperature of the restoration, the tooth, pulp, and soft tissues, but they will be scattered more and be less penetrating than the photons in the blue range. Thus, if the violet light does not penetrate as well as blue, why include the violet light that will only produce more heat? However, the decision not to increase the violet component at the higher power settings from the LCU may be manufacturer-specific, illustrating the importance of knowing the emission spectrum from the LCU on all its output settings.

For optimum photocuring of the resin beneath, the LCU should have an effective tip diameter that fully covers a molar tooth, adequate radiant power, irradiance, and a homogeneous beam profile [20]. In the present study, the light tip completely covered the 6 mm diameter specimen, and the light output was measured through an aperture that corresponded to the 6 mm diameter of the resin specimens. Thus, the light that was received by the specimen was

measured, not the total output of the light, some of which fell beyond the edges of the specimen.

LCUs with a heterogeneous beam profile will have 'hot and cold' spots of high and lower irradiance values. This will affect the radiant exposure delivered across the surface of the resin composite and affect its mechanical properties [9,21]. In the present study, the LCUs were both from the same manufacturer and used the same optics. Thus, the optics variable was removed from the study. The emission spectra in the violet and the blue ranges from both LCUs were almost identical, and therefore it was possible to isolate the effects of delivering the violet or the blue component, or both components.

The results of this study are limited because only one lithium disilicate glass-ceramic ceramic that was A2 shade with low translucency was used, and it is known that the ceramic translucency, ceramic type, and ceramic thickness can all affect the light transmission through the ceramic [22]. Therefore, when a thicker ceramic is used (1.5 mm and above), dual-curing resin cements should be the first choice for cementation [23]. Further studies are necessary to determine the effect of different LCUs and different wavelengths on other types of resin cements resins and restorative ceramics. Also, because the resin specimens were only 0.1 mm thick, we were unable to measure the hardness of the specimens. Another study evaluated the hardness of both resin cements, however the results showed that longer radiant exposure led to a greater hardness, and it is necessary to evaluate the effect of different wavelengths. [24] Future studies could measure the impact of the emission of different light wavelengths on the nanohardness of light- and dual-cured resin cements.

Based on this study, the clinician should recognize that a light-cured resin cement that uses only CQ photoinitiator (e.g., RV) will not benefit from receiving 1.4 J/cm² violet light provided that at least 5.0 J/cm² of blue light or a 6.4 J/cm² combination of violet and blue light wavelengths is delivered. Thus, there is no need to use a broad spectrum polywave[®] light when using RV and, if used, the violet light energy will likely just cause additional heating. Since both light-cured resin cements achieved an adequate DC when only the blue light component was delivered, it can be concluded that a single peak blue light LCU will produce

similar DC values as a polywave® LCU, provided that the same radiant exposure is delivered.

5 Conclusions

Within the limitations of this *in vitro* study, the following conclusions can be drawn:

1. The Variolink Esthetic LC and RelyX Veneer resin cements behaved differently to the violet light component from the LCU ($P < 0.05$).
2. Delivering blue light alone will cure Variolink Esthetic LC, but using either just violet or a combination of violet and blue light will produce a small increase in DC, even though 1.5 mm of ceramic ($P < 0.05$).
3. Delivering violet light alone does not cure RelyX Veneer cement very well, notably through 1.5 mm of ceramic (DC = 26 % compared to a DC of 75 % for Variolink Esthetic LC).
4. Provided that the ceramic thickness is ≤ 1.5 mm thick, Variolink Esthetic LC appears to be more forgiving to the wavelengths of light delivered.

Acknowledgements

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Tables

Table 1. Resin cements used in the study (data provided by the manufacturers).

Resin Cement	Code	Shade	Composition	Filler (wt%)	Lot No.	Manufacturer
RelyX Veneer	RV	Translucent	Bis-GMA, TEGDMA, zirconia/silica and fumed silica filler, a dimethacrylate polymer CQ photoinitiator, pigments	66	N932443	3M Oral Care, Saint Paul, MN, USA
Variolink Esthetic LC	VE	Neutral	Bis-GMA, UDMA, TEGDMA, ytterbium trifluoride, boroaluminofluorosilicate glass, spheroidal mixed oxide, Ivocerin® benzoyl peroxide, pigments	60-68	X48332/ W11132	Ivoclar Vivadent, Schaan, Liechtenstein

Table 2. Computer-aided design/computer-aided manufacturing (CAD/CAM) material used in the study (data provided by the manufacturers).

Ceramic Material	Classification	Composition	Lot No.	Manufacturer
IPS e.max CAD LT A2	Lithium disilicate glass- ceramic	SiO ₂ , Li ₂ O, K ₂ O, MgO, Al ₂ O ₃ , P ₂ O ₅ and other oxides	X42573	Ivoclar Vivadent, Schaan, Liechtenstein

Abbreviations: Al₂O₃, aluminum trioxide; K₂O, potassium oxide; Li₂O, lithium oxide; MgO, magnesium oxide; P₂O₅, phosphorus pentoxide; SiO₂, silicon dioxide; ZnO, zinc oxide; ZrO₂, zirconium dioxide.

Table 3. Violet, blue, and polywave® light output from the two LCUs measured through a 6.4 mm aperture into an integrating sphere.

Lighting conditions	Aperture (mm)	Effective tip area (mm ²)	Emission Peak (nm)	Mean (±SD) Radiant Power (mW)	Mean Irradiance (mW/cm ²)	Mean (±SD) Radiant Exposure delivered in 10 s (J/cm ²)
Bluephase Style	6.4	32.2	406 and 454	410 (14.5)	644.5	6.4 (0.2)
Blue Light			457	322 (12.8)	507.0	5.0 (0.2)
Violet Light			412	88 (6.2)	138.2	1.4 (0.1)

Figures

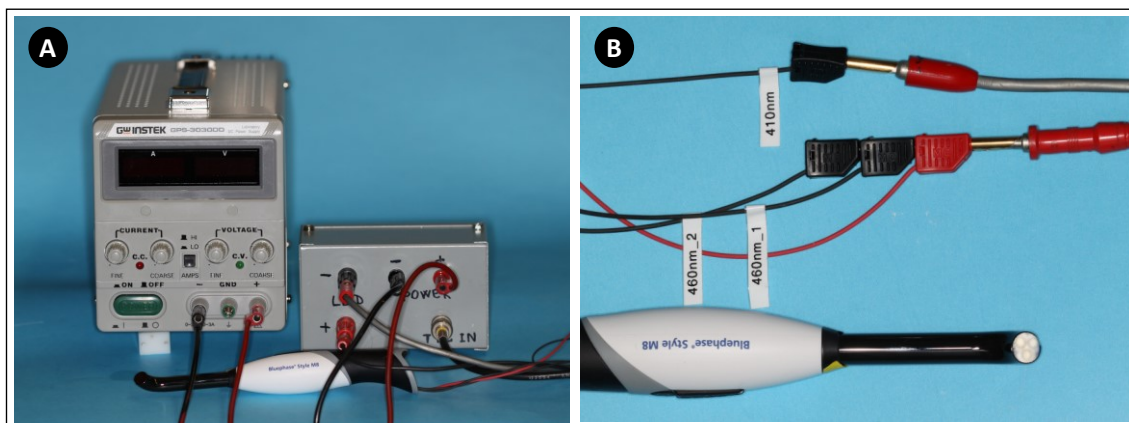


Figure 1. The LED light-curing unit: A- a modified Bluephase Style (Ivoclar Vivadent) curing light was attached to a power supply so that either violet light (peak emission at 412 nm), or blue light (peak emission at 457 nm) could be delivered; B- the modified Bluephase Style curing light showing the different connections used to select the violet or the blue light outputs.

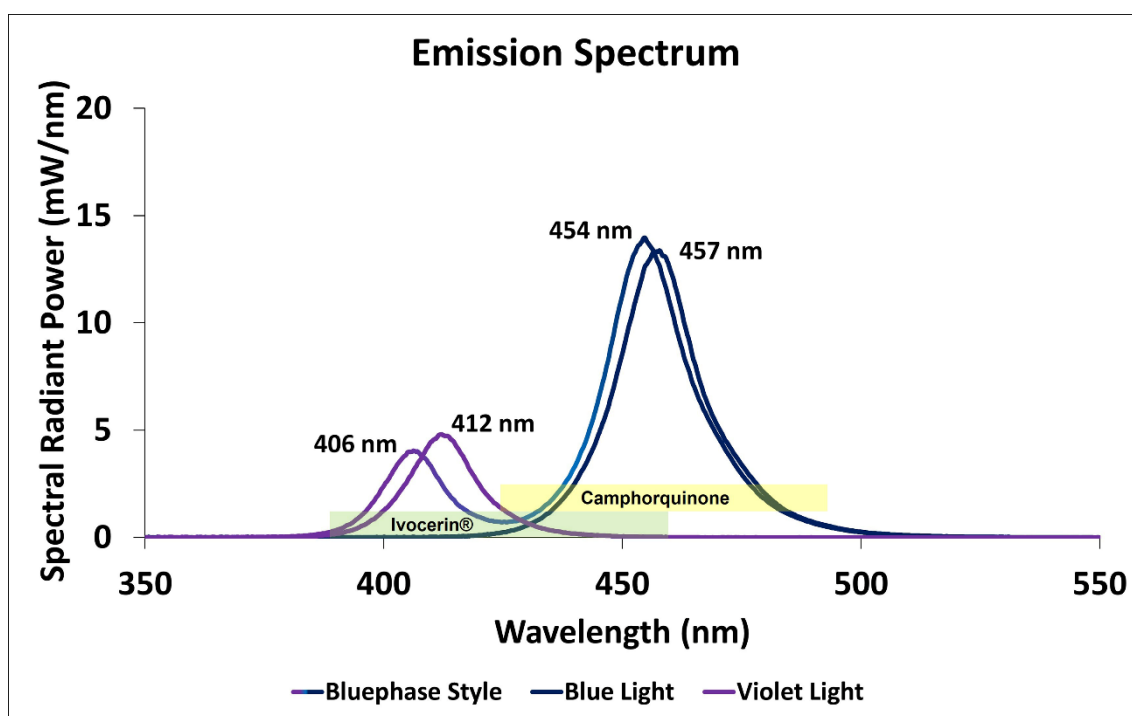


Figure 2. Emission spectra for the three light conditions examined in this study (note the small difference in the emission peaks), and the visible light absorption range of camphorquinone (425 to 495 nm) and Ivocerin® (390 to 460 nm).

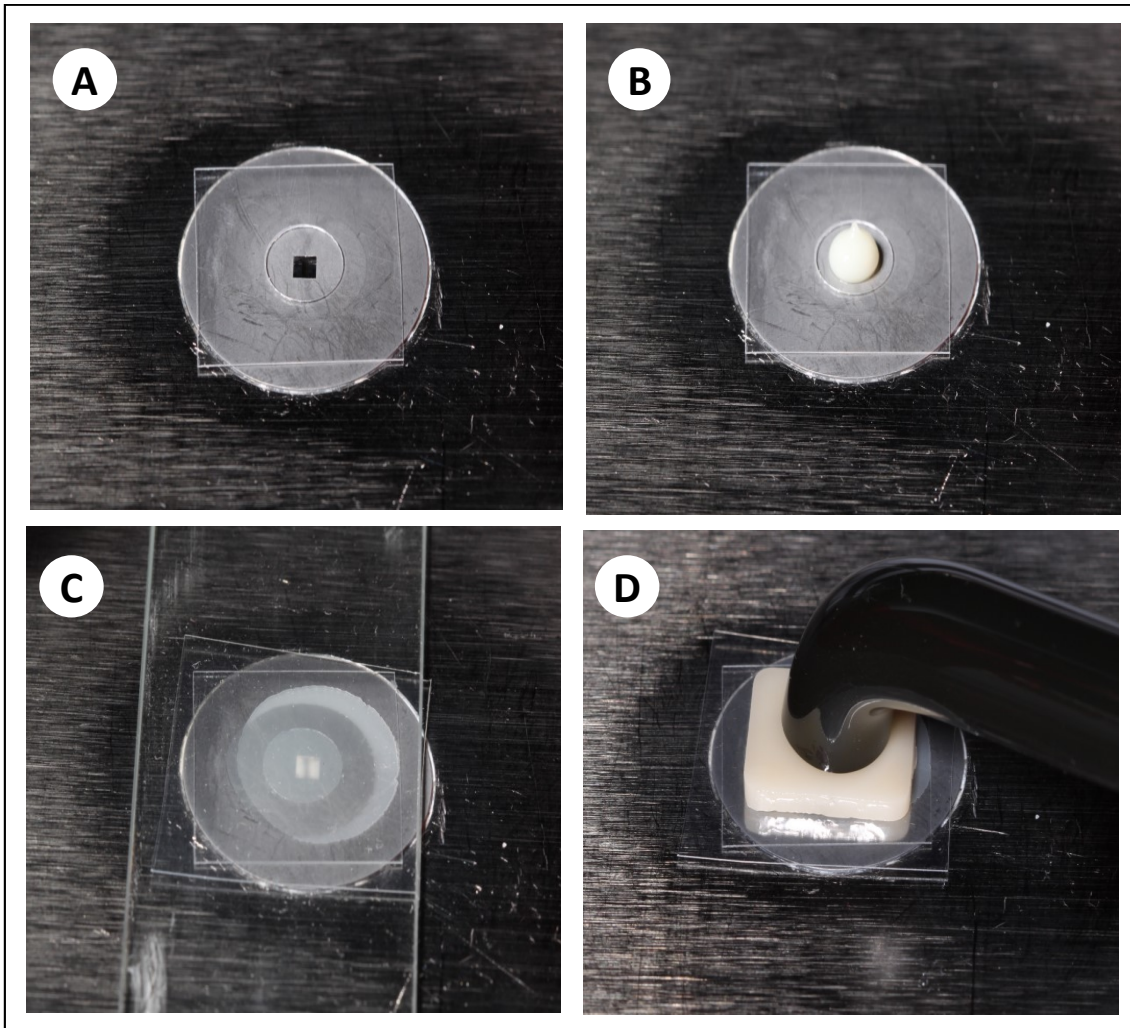


Figure 3. Sample preparation (n=5). A- 0.1 mm thick transparency film positioned on the Golden Gate ATR crystal; B- veneer cement increment positioned inside the hole in the Mylar film; C- Mylar film covering the veneer cement (at a distance of 0 mm); microscope slide used to press the material against the diamond; D – the 1.5 mm thick piece of e.max in position over the Mylar film covering the veneer cement; and light-curing through this 1.5 mm thick ceramic.

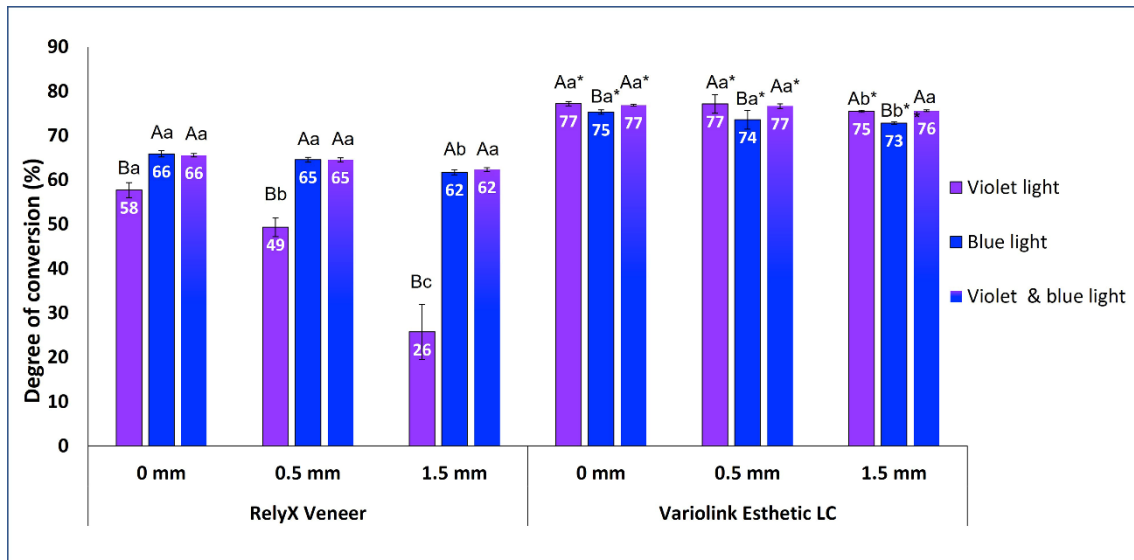


Figure 4. Degree of conversion (%) comparing the type of light received, resin cement, and ceramic thicknesses. Capital letters are used to compare the effect of delivering violet, blue or polywave[®] light, lower case letters are used to compare the effect of ceramic thickness. Tukey multiple comparisons test * used to compare the resin cement at each wavelength range, and the overall effect of ceramic thickness by a *t*-Student test. All tests used $\alpha = 0.05$ significance level.

Considerações finais

4. CONSIDERAÇÕES FINAIS

A partir dos resultados dos estudos *in vitro* realizados nesta tese, pode-se concluir pontos importantes quanto à técnica e ao tipo de fonte fotoativadora a ser utilizada na ativação de resinas compostas e cimentos resinosos. No primeiro estudo, é demonstrada a importância da aproximação da realidade dos estudos *in vitro* da realidade clínica. Visto que, as propriedades da resina composta são dependentes do preparo das amostras para os testes de microdureza e grau de conversão. Os clínicos devem se atentar às caixas proximais das restaurações de resina composta bulk-fill devido a estas áreas estarem sujeitas a não receber luz suficiente para adequada polimerização. Outra conclusão relevante deve-se a inclusão em resina de poliestireno e polimento das amostras mascarar as diferenças entre grupos, quanto menos interferência a amostra de restauração tiver durante o preparo para os testes, maior é a habilidade de detecção das diferenças produzidas pelas fontes fotoativadoras em regiões profundas da restauração.

Já o segundo estudo, comprova que há redução logarítmica na transmissão de luz à medida que a espessura de cerâmica aumenta, o que significa que pouca ou nenhuma luz é transmitida através de 1,5 mm ou mais de espessura de IPS e.max CAD e Celtra Duo, ambas de baixa translucidez e cor A2. A redução é ainda mais acentuada em comprimentos de onda curtos (luz violeta). Quanto ao brilho de superfície, a cerâmica Celtra Duo após glaze apresentou melhor brilho de superfície comparado à IPS e.max CAD após glaze. Entre os tratamentos de superfície da Celtra Duo, nenhuma diferença foi encontrada entre a cerâmica após glaze e a cerâmica após polimento com pasta de diamante. E por fim, não foi encontrada correlação entre o brilho de superfície e a quantidade de luz transmitida.

Os resultados do terceiro estudo demonstram que o Variolink Esthetic LC, um cimento que possui Ivocerin® como fotoiniciador, não necessita de fonte fotoativadora de múltiplos picos para a sua polimerização adequada. Além disso, verificou-se que a emissão de luz no comprimento de onda violeta não melhora a polimerização do cimento resinoso RelyX Veneer. Ambos os cimentos resinosos se comportam de forma diferente à luz violeta. Quando apenas a luz

azul é emitida, o Variolink Esthetic LC é polimerizado, porém ao utilizar apenas a luz violeta ou a combinação de luz violeta e azul é observado um pequeno aumento no grau de conversão, mesmo através de 1,5 mm de IPS e.max CAD. O Variolink Esthetic LC apresenta adequada polimerização independente da luz emitida e da espessura da cerâmica (0,5 e 1,5 mm). E por fim, a emissão da luz violeta isolada polimeriza de forma inadequada o RelyX Veneer, principalmente através de espessuras maiores (1,5 mm).

Há muito o que se estudar sobre os diferentes espectros de emissão das fontes fotoativadoras presentes no mercado e seus efeitos nos mais diversos materiais fotoativáveis, a fim de se alcançar a melhor compreensão dos processos e educação dos profissionais para obter como resultados a longevidade das restaurações diretas e indiretas.

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Parecer do Comitê de Ética – Capítulo 1



PARECER CONSUBSTANCIADO DO CEP

DADOS DO PROJETO DE PESQUISA

Título da Pesquisa: Efeito da fonte de luz e abertura de boca na polimerização de resinas compostas "bulk fill" em molares

Pesquisador: Carlos José Soares

Área Temática:

Versão: 1

CAAE: 96032718.7.0000.5152

Instituição Proponente: Universidade Federal de Uberlândia/ UFU/ MG

Patrocinador Principal: Financiamento Próprio

DADOS DO PARECER

Número do Parecer: 2.985.056

Apresentação do Projeto:

Conforme apresenta o protocolo:

O projeto "in vitro" tem como proposta avaliar de forma experimental o efeito de duas fontes de luz (corpo retilíneo e angulado) e a abertura de boca (normal e limitada) sobre a polimerização de resinas compostas "bulk fill" em molares, definindo protocolos de análise de comportamento biomecânico via espectroscopia Raman, teste de dureza. Serão selecionados, após aprovação pelo CEP da UFU, 60 terceiros molares inferiores humanos, livres de cárie, defeitos estruturais ou restaurações, oriundos de indivíduos com idade semelhante, coletados nas Clínicas de Cirurgia da Faculdade de Odontologia da Universidade Federal de Uberlândia com prévio consentimento dos pacientes. Serão excluídos dentes terceiros molares com cáries, trincas, defeitos estruturais ou restaurações e doadores com idade abaixo de 18 anos, dentes que apresentarem diferença anatômica das suas dimensões coronárias com variação maior que 10% da média serão descartados. Os 60 molares selecionados serão distribuídos aleatoriamente em 4 grupos experimentais (n=15) pela interação entre os dois fatores em estudo, fontes de luz (2 níveis, Valo Cordless - Ultradent Products, Inc, South Jordan, UT, USA e Radium Cal - SDI Ltd, Bonywater, Victoria, Aus). Os fatores de estudo serão: A) Fontes de luz (2 níveis): retilínea, Valo Cordless (Ultradent, South Jordan, UT, EUA), e angulada, Radium Cal (SDI, Bonywater, Victoria, Austrália) e B) abertura de boca (2 níveis): limitada (25 mm) e normal (45 mm). O método de análise será por

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Espectroscopia Raman e FTIR para medição do grau de conversão; teste de indentação dinâmica para medida de dureza Knoop, e microscopia eletrônica para avaliar microinfiltração por nitrato de prata. Após análise da distribuição normal e de homogeneidade entre os valores obtidos será empregada ANOVA fatorial (2x2), caso exista diferença entre os grupos, será empregado teste de comparação de médias de Tukey ($\alpha = 0,05$). Para a infiltração marginal será utilizada análise não paramétrica de Kruskal Wallis. Todos os testes serão aplicados em nível de significância de 5%.

Objetivo da Pesquisa:

Nos termos do projeto

Objetivo Primário:

O presente estudo "in vitro" tem como proposta avaliar de forma experimental o efeito de duas fontes de luz (corpo retilíneo e angulado) e a abertura de boca (normal e limitada) sobre a polimerização de resinas compostas "bulk fill" em molares, definindo protocolos de análise de comportamento biomecânico via espectroscopia Raman, teste de dureza.

Objetivos Secundários:

- Caracterizar o grau de conversão de restaurações em resina composta "bulk fill" de viscosidade regular realizadas em molares utilizando espectroscopia confocal Raman em três porções, sendo mesial, distal e oclusal;
- Mensurar o grau de dureza Knoop (KHN) e módulo de elasticidade (E) da resina composta nas condições experimentais;
- Mensurar o grau de infiltração marginal, sob efeito da termociclagem nas margens cervicais utilizando microscopia de varredura.

Avaliação dos Riscos e Benefícios:

Nos termos do projeto

Riscos:

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A coleta dos dentes será realizada pelo mestrando Rodrigo José Moreira, o qual abordará os pacientes após o atendimento no Pronto Socorro Odontológico e nas clínicas de cirurgia realizadas por alunos de graduação do 6º ao 10º período da Faculdade de Odontologia da UFU. Anteriormente a doação do dente, haverá o procedimento cirúrgico de exodontia, no qual será necessária anestesia e utilização de material previamente esterilizado. Haverá para o paciente o risco de detecção de complicações biológicas, como dor e sangramento, porém o paciente será informado sobre a possível ocorrência e orientado a comparecer nos locais específicos de atendimento para que o tratamento seja instituído. O único risco é a identificação do sujeito de pesquisa no momento da coleta do dente, o que contraria a Resolução 466/12/CNS, porém a equipe executora se compromete a tratar os sujeitos participantes de forma sigilosa, não fazendo a identificação dos mesmos.

Benefícios:

Com o presente estudo poderá ser definido técnicas e protocolos clínicos para reabilitação de molares com perda estrutural utilizando resinas bulk fill em situações clínicas de dificuldade de acesso, permitindo aumentar o nível de sucesso nos tratamentos reabilitadores.

Comentários e Considerações sobre a Pesquisa:

Não há.

Considerações sobre os Termos de apresentação obrigatória:

Apresentados e adequados.

Recomendações:

Revisão de português no TCLE. Pois no TCLE consta que "Nesta pesquisa nós estamos buscando entender sobre resinas em dentes posteriores (molares) e o quanto a abertura de boca interfere nessas restaurações. Para isso precisamos que os terceiros molares indicados para exodontia sejam doados. O Termo de Consentimento Livre e Esclarecido será obtido pelo pesquisado Rodrigo José Moreira nas clínicas dos 6º, 7º e 8º período das Clínicas de Cirurgia da Universidade Federal de Uberlândia".

Conclusões ou Pendências e Lista de Inadequações:

De acordo com as atribuições definidas na Resolução CNS 466/12, o CEP manifesta-se pela aprovação do protocolo de pesquisa proposto.

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O protocolo não apresenta problemas de ética nas condutas de pesquisa com seres humanos, nos limites da redação e da metodologia apresentadas.

Considerações Finais a critério do CEP:

Data para entrega de Relatório Final ao CEP/UFU: fevereiro de 2019.

OBS.: O CEP/UFU LEMBRA QUE QUALQUER MUDANÇA NO PROTOCOLO DEVE SER INFORMADA IMEDIATAMENTE AO CEP PARA FINS DE ANÁLISE E APROVAÇÃO DA MESMA.

O CEP/UFU lembra que:

- a- segundo a Resolução 466/12, o pesquisador deverá arquivar por 5 anos o relatório da pesquisa e os Termos de Consentimento Livre e Esclarecido, assinados pelo sujeito de pesquisa.
- b- poderá, por escolha aleatória, visitar o pesquisador para conferência do relatório e documentação pertinente ao projeto.
- c- a aprovação do protocolo de pesquisa pelo CEP/UFU dá-se em decorrência do atendimento a Resolução CNS 466/12, não implicando na qualidade científica do mesmo.

Orientações ao pesquisador :

- O sujeito da pesquisa tem a liberdade de recusar-se a participar ou de retirar seu consentimento em qualquer fase da pesquisa, sem penalização alguma e sem prejuízo ao seu cuidado (Res. CNS 466/12) e deve receber uma via original do Termo de Consentimento Livre e Esclarecido, na íntegra, por ele assinado.
- O pesquisador deve desenvolver a pesquisa conforme delineada no protocolo aprovado e descontinuar o estudo somente após análise das razões da descontinuidade pelo CEP que o aprovou (Res. CNS 466/12), aguardando seu parecer, exceto quando perceber risco ou dano não previsto ao sujeito participante ou quando constatar a superioridade de regime oferecido a um dos grupos da pesquisa que requeiram ação imediata.
- O CEP deve ser informado de todos os efeitos adversos ou fatos relevantes que alterem o curso normal do estudo (Res. CNS 466/12). É papel de o pesquisador assegurar medidas imediatas adequadas frente a evento adverso grave ocorrido (mesmo que tenha sido em outro centro) e enviar notificação ao CEP e à Agência Nacional de Vigilância Sanitária – ANVISA – junto com seu posicionamento.
- Eventuais modificações ou emendas ao protocolo devem ser apresentadas ao CEP de forma clara e sucinta, identificando a parte do protocolo a ser modificada e suas justificativas. Em caso de

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projetos do Grupo I ou II apresentados anteriormente à ANVISA, o pesquisador ou patrocinador deve enviá-las também à mesma, junto com o parecer aprobatório do CEP, para serem juntadas ao protocolo inicial (Res.251/97, item III.2.e).

Este parecer foi elaborado baseado nos documentos abaixo relacionados:

Tipo Documento	Arquivo	Postagem	Autor	Situação
Informações Básicas do Projeto	PB_INFORMAÇÕES_BÁSICAS_DO_PROJETO_1103544.pdf	13/08/2018 15:55:58		Aceito
Projeto Detalhado / Brochura Investigador	Projeto_Rodrigo_Jose_Moreira.docx	13/08/2018 15:51:58	Rodrigo Jose Moreira	Aceito
Recurso Anexado pelo Pesquisador	Modelo_Coleta_Dados.pdf	09/08/2018 12:07:04	Rodrigo Jose Moreira	Aceito
TCLE / Termos de Assentimento / Justificativa de Ausência	Termo_de_Consentimento_Livre_e_Escelarecido.docx	04/08/2018 19:35:52	Rodrigo Jose Moreira	Aceito
Declaração de Pesquisadores	Curriculum_Lattes_dos_pesquisadores.docx	02/08/2018 15:28:07	Rodrigo Jose Moreira	Aceito
Declaração de Instituição e Infraestrutura	declaracao_coparticipante.pdf	02/08/2018 15:26:18	Rodrigo Jose Moreira	Aceito
Folha de Rosto	rosto.pdf	02/08/2018 10:50:23	Rodrigo Jose Moreira	Aceito
Declaração de Pesquisadores	Termo_de_compromisso.pdf	04/04/2018 21:43:50	Rodrigo Jose Moreira	Aceito

Situação do Parecer:

Aprovado

Necessita Apreciação da CONEP:

Não

UBERLÂNDIA, 26 de Outubro de 2018

Assinado por:
Karine Rezende de Oliveira
(Coordenador(a))

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Normas dos periódicos

1. Operative Dentistry

<https://jopdent.com/author-review-for-journal/instructions-to-authors/>

2. Dental Materials

<https://www.elsevier.com/journals/dental-materials/0109-5641/guide-for-authors>

3. Dental Materials

<https://www.elsevier.com/journals/dental-materials/0109-5641/guide-for-authors>