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CAMILA FERREIRA LEITE MADRUGA

INFLUÊNCIA DA TRANSLUCIDEZ DE LAMINADOS DE CERÂMICA NO GRAU DE CONVERSÃO E TAXA DE POLIMERIZAÇÃO DE UM CIMENTO RESINOSO CONTENDO FOTOINICIADOR ALTERNATIVO EXPOSTO A DIFERENTES UNIDADES DE FOTOATIVAÇÃO

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Dissertação apresentada à Universidade Guarulhos para obtenção do título de Mestre em Odontologia, Área de Concentração em Dentística.

Orientador: Prof. Dr. Cesar A. Galvão Arrais
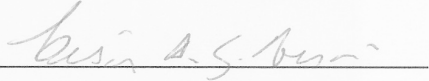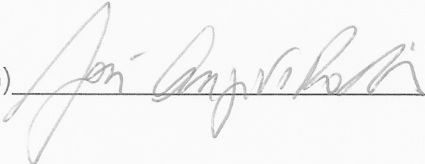
Co-orientador: Prof. Dr. André Figueiredo Reis

GUARULHOS

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A Comissão Julgadora dos trabalhos de Defesa de Dissertação de MESTRADO, intitulada "INFLUÊNCIA DA TRANSLUCIDEZ DE LAMINADOS DE CERÂMICA NO GRAU DE CONVERSÃO E TAXA DE POLIMERIZAÇÃO DE UM CIMENTO RESINOSO CONTENDO FOTOINICIADOR ALTERNATIVO EXPOSTO A DIFERENTES UNIDADES DE FOTOATIVAÇÃO" em sessão pública realizada em 27 de Junho de 2011, considerou a candidata CAMILA FERREIRA LEITE MADRUGA aprovada.

COMISSÃO EXAMINADORA:

1. Prof. Dr. César Augusto Galvão Arrais (UnG)_____

2. Prof. Dr. Paulo Francisco Cesar (FOUSP)_____

3. Prof. Dr. José Augusto Rodrigues (UnG)_____

Guarulhos, 27 de Junho de 2011.

DEDICATÓRIA

Dedico este trabalho,

A Deus
"O Senhor iluminou o meu caminho para que eu sempre pudesse segui-lo."

Ao meu marido, Marcelo
Eu gostaria de agradecer o seu apoio e estímulo para realizar coisas que eu pensava que permaneceriam apenas em sonhos.

A minha mãe, Margareth
Você sempre esteve ao meu lado em todos os momentos da minha vida, me instruindo, me ajudando, me apoiando ou simplesmente me dando muito amor. Obrigada pela dedicação, força e sabedoria.

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Você despertou em meu coração e em minha alma um amor que jamais imaginava!

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Minha amiga e confidente, sempre me aconselhando e dividindo pensamentos e, muitas vezes, me ajudando a enxergar o caminho correto.

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Você me inspira e me motiva. Apesar de não estar mais aqui, a sua presença continua forte e eterna.

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"Aprendemos quando compartilhamos experiências."

(John Dewey)

RESUMO

Este estudo avaliou o grau de conversão (GC) e a taxa máxima de polimerização (Tp_{max}), utilizando espectroscopia infravermelha com transformada de Fourier (FTIR), de um cimento resinoso fotopolimerizável de cor transparente exposto à unidade de fotoativação (UFA) tanto de lâmpada halógena (*quartz-tungsten halogen* – QTH) quanto de lâmpada LED (*light-emitting diodes*) de segunda e terceira gerações sendo que a luz foi atenuada pela presença de laminados cerâmicos com diferentes níveis de translucidez. O cimento resinoso fotopolimerizável (Variolink II Base, cor transparente) foi aplicado na superfície de uma unidade horizontal atenuada (ATR) acoplada ao espectroscópio do FTIR. O cimento resinoso dispensado, foi recoberto por um disco de 1 mm de espessura feito com cerâmicas (IPS e.max Press) de alta translucidez (HT) e de baixa translucidez (LT), ou através de uma lâmina de vidro (LV) e então fotoativado por 40 segundos com QTH (Optilux 501) ou com LED (bluephase G1 e bluephase G2). Os espectros foram obtidos durante 10 minutos (1 espectro/s, 16 *scans*/espectro, resolução de 4 cm^{-1}) imediatamente após a aplicação na ATR. O GC foi calculado usando técnicas padronizadas de avaliação das alterações das razões entre picos C=C alifáticos e aromáticos antes e 10 minutos após a polimerização, bem como a cada 1 segundo de intervalo. A análise de cinética de polimerização foi usada para determinar Tp_{max}. Os dados de GC e a Tp_{max} (n=7) foram analisados por meio da ANOVA 2 fatores e teste de Tukey (p=0,05). Não foram encontradas diferenças significativas no GC entre os grupos LV, HT e LT, independente da UFA. Os valores de GC para os grupos bluephase G2 foram maiores que aqueles obtidos para os grupos QTH (p=.0218). Não houve diferença significativa no grau de conversão tanto entre os grupos bluephase G1 e bluephase G2 quanto entre os grupos bluephase G1 e QTH. O grupo LV apresentou maior Tp_{max} que os grupos HT e LV (p<.0001), mas não foram detectadas diferenças significativas entre HT e LT. Os grupos bluephase G1 apresentarem menor Tp_{max} que bluephase G2 (p=.0234). A presença de laminados cerâmicos interpostos entre a ponta da UFA e o cimento resinoso diminuiu a Tp_{max} sem afetar o GC, enquanto que as diferenças entre os níveis de translucidez da cerâmica não interferiu no GC e na Tp_{max} da camada de cimento resinoso. A utilização de uma UFA de pico duplo não comprometeu o GC e a Tp_{max} do cimento resinoso fotoativado através dos laminados cerâmicos.

Palavras-chave: cimentos resinosos, polimerização, grau de conversão.

ABSTRACT

This study evaluated the degree of conversion (DC) and rate of polymerization (Rp_{max}), using Fourier Transformed Infrared analysis (FTIR), of one commercial, transparent shade resin cement exposed to second and third generation light-emitting diodes (LED), or quartz-tungsten halogen (QTH) curing units (LCUs) with curing light attenuated by the presence of ceramic laminates with varying translucency. One resin cement (Variolink II Base, transparent shade) was applied to the horizontal diamond element of an attenuated total reflectance (ATR) unit attachment in the optical bench of a FTIR spectrometer. The deposited resin cement was covered with 1-mm thick discs with either high (HT) or low (LT) translucence (IPS e.max Press), or with a glass slide (GS, control group), and the resin cement was light-activated for 40 s using QTH (Optilux 501) or LED (Bluephase G1 and G2). FTIR spectra were recorded for 10 min (1 spectrum/s, 16 scans/spectrum, resolution 4 cm^{-1}) immediately after application to the ATR. DC was calculated using standard techniques of observing changes in aliphatic-to-aromatic peak ratios pre- and 10-min post-curing, as well as during each 1-second interval between. Time-based monomer conversion analysis was used to determine Rp_{max}. DC and Rp_{max} data (n=7) were analyzed by two-way ANOVA and Tukey's post-hoc test (p=.05). No significant differences in DC were found among GS, HT, and LT groups, regardless of LCU. DC values in bluephase G2 groups were higher than those in QTH groups (p=.0218). No significant difference in DC was noted either between bluephase G2 and bluephase G1 groups or between bluephase G1 and QTH groups. GS group showed higher Rp_{max} than HT and LT groups (p<.0001), but no significant differences in Rp_{max} were noted between HT and LT. Bluephase G1 groups exhibited lower Rp_{max} than bluephase G2 (p=.0234). The presence of ceramic laminates interposed between LCU tip and resin cement decreased Rp_{max} without affecting DC, while differences in ceramic translucency did not affect DC nor Rp_{max} of resin cement layer. The use of dual peak LCU neither compromised DC nor Rp_{max} of translucent resin cement base paste underlying ceramic laminates.

Keywords: resin cements, polymerization, degree of conversion.

SUMÁRIO

1. INTRODUÇÃO E JUSTIFICATIVA

Atualmente, a busca pela odontologia estética está em constante crescimento, juntamente com a demanda pelas restaurações cerâmicas. Como as coroas totais são restaurações bastante invasivas, necessitando de reduções consideráveis na estrutura dental, as facetas laminadas e as restaurações livres de metais tem sido muito utilizadas (Calamia e Calamia, 2007). Diferentes materiais foram desenvolvidos para confeccionar estas restaurações, como as cerâmicas feldspáticas, as cerâmicas reforçadas e os compósitos de uso laboratorial. No entanto, quando se trata de estética, resistência e propriedades ópticas semelhantes ao dente natural, a cerâmica é o material de eleição (Peumans et al., 2000).

Dentre as propriedades ópticas existentes, a translucidez é considerada uma das mais importantes, pois está relacionada às falhas clínicas em relação ao mimetismo entre o material restaurador e o dente natural (Brodbelt et al., 1980). Em função disso, cerâmicas com diferentes níveis de translucidez foram desenvolvidas visando atender todas as necessidades clínicas. De acordo com Brodbelt (1980), translucidez é a quantidade relativa de luz transmitida ou a reflectância difusa de um substrato através de um meio. Com isso, baixa translucidez de uma cerâmica significa que pouca quantidade de luz é transmitida através da mesma.

Além dessas considerações estéticas, o sucesso de uma restauração cerâmica depende da longevidade da união entre cerâmica e agente cimentante, e consequentemente da união entre agente cimentante e superfície dental (esmalte e dentina). Atualmente, os compósitos têm sido utilizados como base dos principais materiais para cimentação. Para a cimentação dos laminados cerâmicos (até 1mm de espessura), os cimentos resinosos fotopolimerizáveis são os mais indicados (Peumans et al., 2000). No entanto, a boa *performance* desses compósitos depende de uma polimerização adequada e, fotopolimerizar através das cerâmicas é um desafio, pois uma quantidade considerável de luz é perdida através da absorção, reflexão ou transmissão (Ilie e Hickel; 2008). Considerando que a eficácia desta união depende de uma ótima polimerização do cimento resinoso (Cekic-Nagas et al., 2010; Lohbauer et al., 2005), é importante que uma quantidade de energia adequada penetre na camada de cimento. Por esta razão, a fonte ativadora deve assegurar que esta energia alcance o cimento resinoso.

Baseado nisso, as unidades de fotoativação (UFA) tornaram-se uma parte integral da Odontologia moderna, sendo que as UFA de lâmpada halógena (QTH)

foram por muitos anos a principal fonte ativadora. Unidades com lâmpadas LED (*Light Emitting Diodes*) foram desenvolvidas em função de apresentarem propriedades superiores em relação às lâmpadas halógenas (QTH) como por exemplo: estável emissão de energia ao longo do tempo e maior longevidade. Com isso, são atualmente utilizadas como as mais recentes fontes de luz para polimerização.

Ambas as UFAs, de lâmpada halógena e de LED, foram desenvolvidas para emitirem um comprimento de onda compatível ao de absorção da canforoquinona (CQ), o principal fotoiniciador utilizado em resinas compostas e cimentos resinosos, cujo pico de absorção é de aproximadamente 470 nm. No entanto, como a canforoquinona possui uma coloração amarela intensa, outros iniciadores alternativos, como a fenilpropanodiona (PPD) ou a lucerina TPO (Ilie e Hickel, 2008), têm sido adicionados na composição destes produtos, principalmente quando é necessária a formulação de compósitos claros e selantes incolores. A lucerina TPO ganhou popularidade porque torna-se completamente clara após o término da reação química, no entanto, possui um pico de absorção curto, de aproximadamente 390 nm. Desta forma, o comprimento de onda emitido por UFAs LED (entre 425 e 470 nm) não consegue polimerizar de forma ideal materiais resinosos translúcidos ou de cores claras que apresentam este fotoiniciador em sua composição (Ilie e Hickel, 2008; Uhl et al., 2004).

Diante disto, uma nova geração de LEDs ("terceira geração") foi desenvolvida para tentar suprir as limitações impostas pelas primeiras vesões (Price et al., 2010; Rueggeberg, 2011). Em contraste com as primeiras gerações, as novas unidades de polimerização se assemelham às lâmpadas halógenas (QTH) por possuírem um comprimento de onda entre 380 e 515 nm, pois dois LEDs são utilizados, um com aproximadamente 410 nm e o outro com aproximadamente 470 nm (comprimentos de onda dominantes) (Price et al., 2005; Price et al., 2010; Rueggeberg, 2011). Com esta característica, a luz emitida por tais unidades é capaz de excitar estes fotoiniciadores alternativos tanto quanto a canforoquinona e polimerizar adequadamente os compósitos mesmo os de cores claras ou translúcidas (Park et al., 1999). Entretanto, a fotoativação dos cimentos resinosos com os fotoiniciadores alternativos tornou-se um desafio para os clínicos, visto que as cerâmicas são menos permeáveis ao comprimento de onda de absorção desses fotoiniciadores que ao da canforoquinona (Ilie e Hickel, 2008).

Em contrapartida, a luz emitida por essas unidades de polimerização geralmente excede a irradiância de 1.000 mW/cm^2 com isso, elevados valores de grau de conversão (GC) tem sido observados em resinas compostas contendo apenas CQ quando LEDs de segunda e terceira gerações são utilizados (Ilie e Hickel, 2008). No entanto, estes fotoativadores também promovem alta taxa de polimerização (Tp$_{max}$), o que pode ocasionar alta tensão de contração de polimerização (Pfeifer et al., 2008), que quando combinada com a termociclagem, pode ser responsável pela propagação de fraturas nos laminados de cerâmica (Magne et al., 1999).

Apesar de existirem informações na literatura sobre a efetividade de tais fontes de luz de alta intensidade nas características de polimerização de resinas compostas (Palin et al., 2008), não há estudos disponíveis que estejam relacionados ao GC e à Tp$_{max}$ de cimentos resinosos contendo fotoiniciador alternativo e expostos à luz transmitida de diferentes tipos de unidade de fotoativação através de laminados cerâmicos com diferentes níveis de translucidez.

O grau de conversão (GC) é uma medida de avaliação da qualidade da polimerização das resinas compostas, uma vez que consiste na análise do percentual de duplas ligações de carbono que foram convertidas em ligações simples para formar uma cadeia polimérica (Ruyter e Györösi, 1976). Quando a polimerização dos compósitos dentais a base de metacrilato é iniciada, um radical livre de polimerização do grupo metacrilato e acrilato segue a ativação do fotoiniciador através da luz. Durante a polimerização, as duplas ligações C=C são convertidas, e quanto maior a quantidade de duplas ligações convertidas em ligações simples, maior será o GC dos compósitos (Wendl, 2004). Os valores de GC podem ser calculados de várias maneiras sendo que a análise utilizando espectrofotometria infravermelha com transformada de Fourier (FTIR) tem se mostrado uma metodologia eficaz e confiável. Deste modo, o objetivo deste estudo foi avaliar por meio de espectroscopia infravermelha com transformada de Fourier (FTIR) o GC e a Tp$_{max}$ de cimentos resinosos fotopolimerizáveis expostos a diferentes unidades de fotoativação e utilizados sob laminados cerâmicos com diferentes níveis de translucidez. As hipóteses de estudos foram: (1) a presença de laminados cerâmicos interpostos entre a ponta da unidade de fotoativação (UFA) e a camada de cimento resinoso fotopolimerizável diminui o GC e a Tp$_{max}$, independente do tipo de fotoativador; (2) laminados cerâmicos menos translúcidos promovem

menores valores de GC e Tp_{max} que cerâmicas mais translúcidas; (3) a utilização de um LED de "terceira geração" é tão efetiva quanto às unidades de lâmpada QTH referente ao GC e Tp_{max} de cimentos resinosos com baixo conteúdo de canforoquinona e alto de lucerina TPO.

2. PROPOSIÇÃO

O objetivo geral deste estudo foi avaliar por meio de FTIR a relação entre a translucidez de laminados cerâmicos e o GC e Tp_{max} de um cimento resinoso fotoativado com baixo conteúdo de canforoquinona e alto de lucerina TPO. Os objetivos específicos foram:

- A presença de laminados cerâmicos interpostos entre a ponta da unidade de fotoativação (UFA) e a camada de cimento resinoso fotopolimerizável diminui o GC e a Tp_{max}, independente do tipo de fotoativador.

- Averiguar se laminados cerâmicos menos translúcidos promovem menores valores de GC e Tp_{max} que cerâmicas mais translúcidas.

- Avaliar se a utilização de um LED de "terceira geração" é tão efetiva quanto às unidades de lâmpada QTH referente ao GC e Tp_{max} de cimentos resinosos com baixo conteúdo de canforoquinona e alto de lucerina TPO.

3. METODOLOGIA E RESULTADOS

A presente dissertação está baseada no artigo "Effect of ceramic laminate translucency on polymerization kinetics of one luting agent with high content of alternative photoinitiators exposed to second and third generation light curing units".

Effect of ceramic laminate translucency on polymerization kinetics of one luting agent with high content of alternative photoinitiators exposed to second and third generation light curing units

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ABSTRACT

Objectives: This study evaluated the degree of conversion (DC) and rate of polymerization (Rp_{max}), using Fourier Transformed Infrared analysis (FTIR), of one commercial, transparent shade resin cement exposed to second and third generation light-emitting diodes (LED), or quartz-tungsten halogen (QTH) curing units (LCUs) with curing light attenuated by the presence of ceramic laminates with varying translucency. **Materials and Methods:** One resin cement (Variolink II Base, transparent shade) was applied to the horizontal diamond element of an attenuated total reflectance (ATR) unit attachment in the optical bench of a FTIR spectrometer. The deposited resin cement was covered with 1-mm thick discs with either high (HT) or low (LT) translucence (IPS e.max Press), or with a glass slide (GS, control group), and the resin cement was light-activated for 40 s using QTH (Optilux 501) or LED (Bluephase G1 and G2). FTIR spectra were recorded for 10 min (1 spectrum/s, 16 scans/spectrum, resolution 4 cm^{-1}) immediately after application to the ATR. DC was calculated using standard techniques of observing changes in aliphatic-to-aromatic peak ratios pre- and 10-min post-curing, as well as during each 1-second interval between. Time-based monomer conversion analysis was used to determine Rp_{max}. DC and Rp_{max} data (n=7) were analyzed by two-way ANOVA and Tukey's post-hoc test (p=.05). **Results:** No significant differences in DC were found among GS, HT, and LT groups, regardless of LCU. DC values in bluephase G2 groups were higher than those in QTH groups (p=.0218). No significant difference in DC was noted either between bluephase G2 and bluephase G1 groups or between bluephase G1 and QTH groups. GS group showed higher Rp_{max} than HT and LT groups (p<.0001), but no significant differences in Rp_{max} were noted between HT and LT. Bluephase G1 groups exhibited lower Rp_{max} than bluephase G2 (p=.0234). **Conclusion:** The presence of ceramic laminates interposed between LCU tip and resin cement decreased Rp_{max} without affecting DC, while differences in ceramic translucency did not affect DC nor Rp_{max} of resin cement layer. The use of dual peak LCU neither compromised DC nor Rp_{max} of translucent resin cement base paste underlying ceramic laminates.

Keywords: resin cements, polymerization, degree of conversion.

INTRODUCTION

Over the last years, patients' demand for aesthetic restorations led clinicians to choose ceramic laminates, the most conservative treatment among all indirect restorations. These materials have many advantages over resin composites, such as better mechanical properties and optical properties similar to those of natural teeth.[1] Among all optical properties, translucency is considered the most important one as differences in translucency are responsible for the clinical failure of ceramic laminates regarding the mimetic effect between restorative material and prepared tooth.[2] Because of this issue, manufacturers have developed ceramic systems with different translucency to ensure success of ceramic laminates in all clinical scenarios.

According to Brodbelt (1980),[2] translucency is the relative amount of light transmitted or the diffuse reflectance from a substrate surface through a turbid medium, so low ceramic translucency means low amount of light transmitted through ceramic to underlying bonding material. Considering that reliable and durable bonding between ceramic laminate and tooth structure depends upon optimum polymerization of the resin cement underlying the ceramic material,[3,4] a minimum amount of radiant energy must be delivered to the resin cement layer. For this reason, light source must ensure that optimal radiant exposure is delivered to resin cement layer, while ceramic translucency should allow light to pass through the indirect restoration and reach the resin cement with the minimum light intensity required to provide high degree of conversion (DC). These aspects deserves more attention mainly when ceramic laminates are used, as they are usually cemented with light-curing luting composites.[1]

With this regard, high-power light-emitting diodes (LED) light-curing units (LCUs) were developed to provide enough radiant exposure to the underlying resin cement and warranty its optimum polymerization. However, the narrow wavelength from 425 to 470 nm emitted by these LCUs cannot be effective when translucent or bright white resin cement base pastes are used,[5,6] as they have camphorquinone (CQ) partially replaced by so-called "white" photo-initiators, such as 2,4,6-trimethylbenzoyl-diphenylphosphine oxide (Lucirin-TPO) and 1-phenyl-1,2-propanedione (PPD),[7] which have the maximum absorbance spectral peak located in lower wavelength ranges (~390nm peak).

Most recently, so-called "third generation" dual peak LED LCUs were developed to overcome all limitations imposed by earlier LED LCUs versions.[8,9] In

contrast to older LED generations, these new LCUs emit light with broad spectral range of wavelengths from 385 to 515 nm, as two LEDs are used, one with approximately 410 nm and the other with approximately 470 nm (dominant wavelengths).[8-10] With this feature, such LCUs are capable of providing optimum polymerization to resinous materials even when alternative photoinitiators are included in their composition.[6,11,12] However, light activation of resin cements with these alternative photoinitiators becomes an issue to clinicians as ceramics are less permeable for maximum absorption wavelength of these alternative photoinitiators than for CQ absorption length.[13]

Conversely, light emitted by these LCUs usually exceeds the irradiance of 1000 mW/cm^2, so high DC values have been observed in resin composites when second and third generation LED LCUs are used.[13] As a consequence, the resulting fast rate of polymerization (Rp_{max}) leads to high contraction stress,[14] which combined with cyclic thermal loads may be responsible for crack development in these ceramic restorations.[15] Based on this argument, it would be desirable that resin cement layer achieved high degree of conversion (DC) with moderate Rp_{max}. Although there is available information in the literature about the effectiveness of such high power light sources on the polymerization characteristics of resin composites,[12] no information is available concerning the effects of one dual peak LCU on the degree of conversion (DC) and Rp_{max} of translucent luting composite, with CQ partially replaced by Lucerin TPO, exposed to attenuated light due to the presence of ceramic laminates with varying translucency.

The aim of the current study was to evaluate DC and Rp_{max} of resin cement base paste exposed to different curing units having ceramic laminates with different levels of translucency interposed between the light curing unit tip end and the luting composite layer. The research hypotheses were: (1) the presence of ceramic laminates interposed between LCU tip and resin cement layer decreases the DC and Rp_{max} values, regardless of curing unit; (2) less translucent ceramic laminates promotes lower DC and Rp_{max} values than more translucent ceramics; (3) "third generation" LED LCU is as effective as quartz-tungsten halogen (QTH) LCU regarding DC and Rp_{max} of resin cement with lower content of CQ and higher content of Lucirin TPO.

MATERIALS AND METHODS

Specimen preparation

The base paste of one dual-curing (light and self-curing) luting composite (Variolink II Base, transparent shade, Ivoclar Vivadent, Schaan, Liechtenstein) was evaluated. Ceramic discs with varying translucency were selected to simulate ceramic laminates that offer different levels of light transmission to an underlying resin cement base paste: 1.0 mm thick by either high (HT) or low (LT) translucency (IPS e.max Press, A2 shade, Ivoclar Vivadent, Schaan, Liechtenstein).

The light-curing luting composite was then applied to the horizontal diamond element of an attenuated total reflectance (ATR) unit attachment (Golden Gate, Specac, Woodstock, GA, USA) in the optical bench of a Fourier transform infrared spectrometer (Tensor 27, Bruker Optik GmbH, Ettlingen, German). Prior to resin placement, adhesive tape (3M) was placed around the diamond surface to act as a spacer, ensuring standard thickness for all specimens (100 – 120 μm). When exposing cement using different translucency of overlying ceramic discs, the deposited resin cement was covered with a Mylar strip and the discs were placed directly between the Mylar sheet and the emitting light guide end (Figure 1). Light-activation of the resin cement base paste was performed through the discs for 40 s as recommended by the manufacturer, using either a QTH LCU with emitting irradiance of 600 mW/cm^2 (Optilux 501, Demetron Kerr, Danbury, CA, USA) or a second (LED, irradiance of 650 mW/cm^2 in the low power mode, bluephase G1, Ivoclar Vivadent, Schaan, Liechtenstein) and third generation LED curing units (irradiance of 650 mW/cm^2 in the low power mode, bluephase G2, Ivoclar Vivadent). As control group, emitting light guide end was placed directly against a 1-mm thick glass slide, positioned directly over top of the Mylar-covered resin specimen, so the specimens were exposed without any overlying restorative material (DLE - direct light exposure – control group).
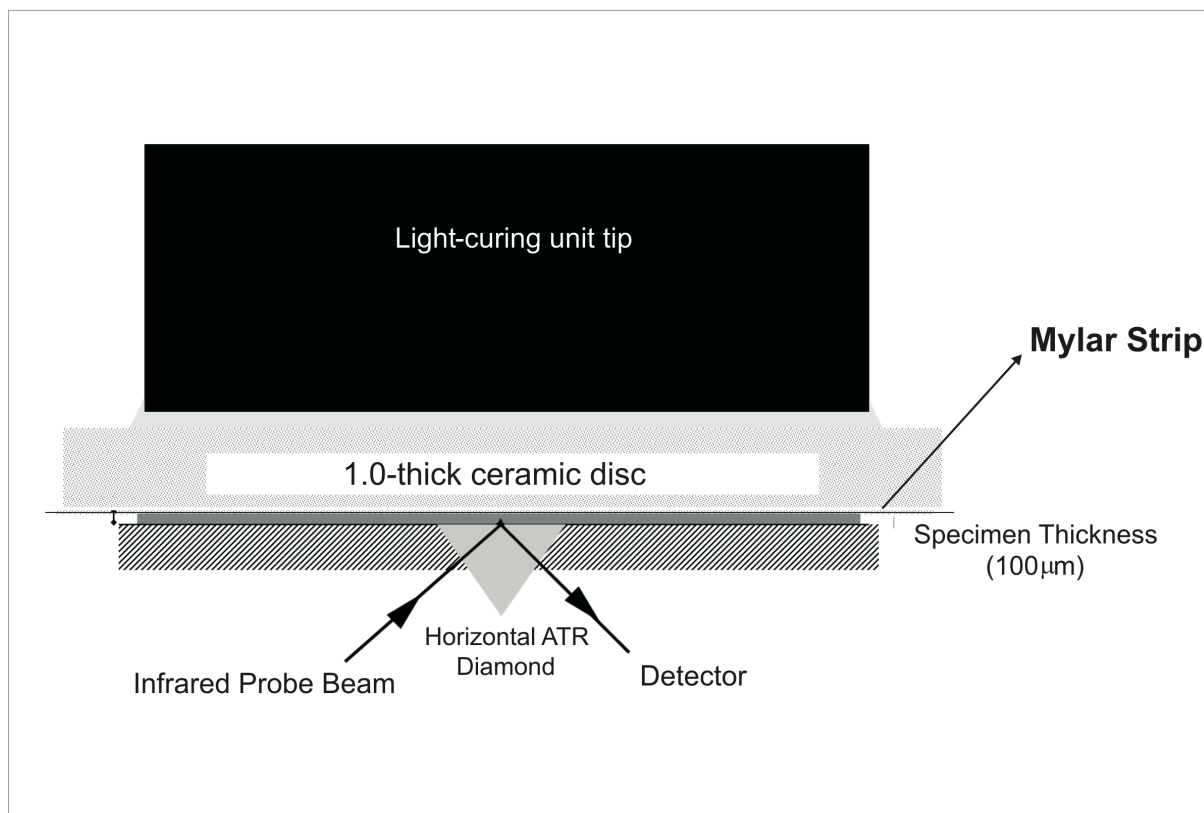
Figure 1 – Illustrative diagram demonstrating the interaction between the infrared beam and the specimen, as well as the position of the ceramic discs or the glass slide and the light-curing unit tip.

Degree of conversion

Infrared (IR) spectra were collected between 1680 and 1500 cm^{-1} at a rate of one spectrum per second (16 scans per spectrum) at 4-cm^{-1} resolution. Data were counted from the moment the IR scan demonstrated the resin was stabilized on the ATR surface and any overlying objects had been placed. Spectra were recorded continuously during the following 10 min. Seven replications were made for each test condition (n=7). Monomer conversion was calculated using standard methods that evaluated changes in the ratios of aliphatic-to-aromatic C=C absorption peaks (1636 cm^{-1}/1608 cm^{-1}) in the uncured and cured states obtained from the infrared spectra.[16,17] Prior to determining conversion, calibration graphs were made relating the absorbance ratios of known molar concentrations of aliphatic and aromatic C=C to their respective absorbance height ratios. Conversion values among all curing modes were compared statistically at 10 min from the time the resin cement was stabilized on the ATR surface. All polymerized specimens were carefully removed from the ATR plate and measured for thickness to the nearest 0.01 mm using a

digital micrometer (Series 406; Mitutoyo America Corp., Aurora, IL, USA) to ensure similar thickness among all specimens.

Polymerization Rate

Polymerization kinetic values were calculated following previously described methods.[18] The Rp_{max} (percent/s) corresponded to the highest rate of polymerization and was calculated based on the differences between DC values measured in sequential, 1-second intervals throughout the 10-min analysis of each specimen.

Statistical analyses

Degree of conversion and Rp_{max} data were evaluated using a two-way ANOVA (factors: ceramic translucency (3 levels) and light source (3 levels)). Significant differences were analyzed by Tukey's post-hoc test. All testing was performed at a pre-set alpha of 0.05 using personal statistical software (SAS 8.0 for Windows; SAS Institute Inc., Cary, USA). Post hoc power analysis was performed for the statistical analysis of DC and Rp_{max} data using personal statistical software (IBM SPSS 19, SPSS Inc., IBM Company, Armonk, NY, USA).

RESULTS

Degree of conversion

For the number of specimens used (n=7), the study was adequately powered for both factors, "ceramic translucency" and "light source" (over 95%; α=0.05) regarding DC and Rp_{max} analyses. The results of DC values are exhibited in Table 1. ANOVA did not indicate statistical significance for "ceramic translucency" factor, so no significant differences in DC values were found among DLE, LT, and HT groups, regardless of LCU. On the other hand, ANOVA indicated statistical significance for "light source" factor and Tukey's post hoc test indicated that DC values of specimens exposed to light from bluephase G2 were higher than those when specimens were exposed to QTH LCU (p=0.0218). However, no significant difference in DC was noted between groups exposed to bluephase G2 and those exposed to bluephase G1 or between groups exposed to bluephase G1 and those exposed to QTH LCUs.

Table 1: DC (%) values of Variolink II translucent base paste exposed to different light sources and activation conditions.

	Direct light exposure (DLE)	High translucent ceramic (HT)	Low translucent ceramic (LT)
Optilux 501	47.4 (0.5) Ba	47.6 (0.5) Ba	47.3 (0.4) Ba
bluephase G1	47.3 (0.4) ABa	47.9 (0.6) ABa	48.1 (1.0) ABa
bluephase G2	48.0 (0.5) Aa	48.3 (0.5) Aa	48.4 (0.7) Aa

Significant differences are indicated by different letters (Upper case letter, comparison within column; Lower case letters, comparison within row).

Polymerization Rate

Rp$_{max}$ values are displayed in Table 2. ANOVA detected statistical significance for "ceramic translucency", "light source" factors, as well as the interaction between factors. Therefore, Variolink II from DLE group showed higher Rp$_{max}$ than Variolink II from the other groups (p<.0001). No significant difference in Rp$_{max}$ was neither noted between HT and LT groups nor between groups exposed to different LED LCUs. For DLE groups, no significant difference in Rp$_{max}$ was observed among groups exposed to different LCUs. On the other hand, for HT and LT groups, groups exposed to bluephase G1 exhibited lower Rp$_{max}$ values than those exposed to bluephase G2 (p=0.0234), while no significant difference was observed between groups exposed to QTH and those exposed to bluephase G2. Within HT groups, no significant difference in Rp$_{max}$ values was observed between groups light-activated by QTH and those light-activated by bluephase G1, while within LT groups QTH LCU promoted higher Rp$_{max}$ values than bluephase G1 (p=0.0234).

Table 2: Rp$_{max}$ (% / s) values of Variolink II translucent base paste exposed to different light sources and activation conditions.

	Direct light exposure (DLE)	High translucent ceramic (HT)	Low translucent ceramic (LT)
Optilux 501	10.0 (0.7) Aa	7.6 (0.4) ABb	7.7 (0.7) Ab
bluephase G1	9.4 (0.4) Aa	6.9 (0.4) Bb	6.4 (0.5) Bb
bluephase G2	10.0 (0.3) Aa	8.3 (0.4) Ab	8.2 (0.4) Ab

Significant differences are indicated by different letters (Upper case letter, comparison within column; Lower case letters, comparison within row).

DISCUSSION

Neither the presence of ceramic laminates between LCU tip and resin cement layer nor ceramic translucency ranges decreased DC values, so the first and second research hypotheses were rejected regarding DC values. These results imply that light exposure of attenuated light for 40 s was capable of providing optimal polymerization to the resin base paste. According to El-Mowafy et al.,[19] 1-mm thick ceramic disc allows approximately 40% of light to be transmitted to the underlying resin cement. Therefore, it is possible that light from any of the tested LCUs was delivered to the resin cement with approximately 240 mW/cm^2. If this assumption is true, the expected radiant exposure of approximately 9.6 J/cm^2 in all groups having ceramic laminates interposed between the curing unit light tip end and resin cement was lower than the recommended minimum value, which may range from 12 to 24 J/cm^{2}.[20,21] However, differently from resin composites, resin cements have low viscosity, which allows higher radical mobility and collision frequency of unreacted active groups and radicals.[22,23] Thus, it is reasonable to assume that lower radiant exposure is required for resin cements than that necessary for resin composites to promote optimum polymerization.

Despite the lack of differences in DC values among groups, lower radiant exposure due to the presence of ceramic laminates resulted in decreased Rp_{max} values in comparison to maximum radiant exposure (use of glass slide), validating the first research hypothesis regarding Rp_{max} values. This result was expected as Rp_{max} depends on free radical production rate, which is in turn directly proportional to the irradiance absorbed by the resin cement.[24,25] Nevertheless, the significant differences in radiant energy due to the range in ceramic translucency did not affect Rp_{max} values, demonstrating that small changes in radiant exposure have no effect on polymerization kinetics of resin cements. Thus, the second hypothesis was invalidated for Rp_{max} analysis.

Even though conversion is usually dependent upon the rate of polymerization (Rp_{max}),[26] the higher in Rp_{max} values observed in control groups and in bluephase G2 groups than in other groups was not reflected in the DC values. As previously established, high irradiance leads to high Rp_{max}, which in turn promotes high rates of primary radical termination, compromising final monomer conversion.[27] On the other hand, low power density decreases Rp_{max} and shift the maximum rates to longer time, in contrast to the short maximum rates observed when resins are

exposed the high power density.[26] As a consequence, lower Rp_{max} results in slower and longer radical termination, which allow further conversion to occur at this stage.[26] Therefore, the lower Rp_{max} values due to the low power density in groups having ceramic laminates allowed further post cure conversion to compensate for the small differences between Rp_{max} during light-activation, leading to similar 10-min DC values among groups regardless of the differences in Rp_{max}.

In the current study, DC values of resin cement base paste exposed to LED bluephase G2 LCU were higher than those when either QTH or bluephase G1 LCUs were used, so the third hypothesis was validated. Similar findings were also observed by Price et al (2010),[11] who confirmed the effectiveness of dual peak LED LCUs by a hardness analysis of resin composites. One could state that high intensity light emitted by these polywave LCUs would compensate for the low intensity of light emitted in low wavelength to excite the alternative photoinitiators. For this reason, in order to avoid the influence of higher light intensity on the results, the LED LCUs were set to operate in the low power mode, which consists of an operating option available in the LCU that allows the clinician to work with light intensity of 650 mW/cm^2. Besides, the lack of significant difference in DC values between groups exposed to bluephase G1 and those exposed to QTH LCU demonstrated that CQ was capable of compensating for the poor effectiveness of blue light to activate alternative photoinitiators.

In this study, it should be pointed out that FTIR method with ATR table attached to the IR spectroscopy focused only on one specific region in the middle of the specimen surface. As irradiance uniformity from dual peak LCUs depends on the position of the LED chips and their emission wavelengths,[8] it is possible that DC and Rp_{max} values do not represent monomer conversion on the entire resin cement layer, as previously demonstrated by Pallin et al (2008),[12] who noted differences in hardness from the center to the periphery of resin composite surfaces exposed to these LED LCUs. Nevertheless, the differences between second and third generation LED LCUs regarding light features was evidenced on Rp_{max} results, as bluephase G1 promoted lower Rp_{max} values than bluephase G2 when Variolink II was exposed to attenuated light in HT and LT groups. This finding confirms previous evidences[6,12] that second generation LED LCUs are less effective in exciting alternative photoinitiators such as Lucirin TPO than polywave third generation LED LCUs.

Based on these results, clinicians may rely on dual peak LED LCUs to cement ceramic laminates with varying translucency even when translucent resin cements with lower CQ content are used. However, it should be emphasized that only one specific translucent resin cement base paste and one specific dual peak LED LCU were evaluated in the current study. Besides, restricted information about photoinitiator content was provided by the manufacturer, so the concentration of CQ and Lucirin TPO in the translucent resin cement base paste is unknown. Differences in concentration of photoinitiatiors in other resin cements as well as differences in wavelength of light emitted from other third generation LED LCUs may lead to other results different from those observed in the current study. Therefore, further studies evaluating other third generation LED LCUs as well as other resinous materials with alternative photoinitiators are required to confirm the effectiveness of such LED LCUs in resin cement polymerization.

CONCLUSION

Within the limitations of this study, it was possible to conclude that:

- The presence of ceramic laminates interposed between LCU tip and resin cement base paste decrease Rp_{max} without affecting DC.
- Differences in ceramic translucency did not affect DC and Rp_{max} of resin cement layer.
- The use of dual peak LCU neither compromised DC nor Rp_{max} of translucent resin cement base paste underlying ceramic laminates with varying translucency.

REFERENCES

1. Peumans M, Van Meerbeek B, Lambrechts P & Vanherle G (2000) Porcelain veneers: a review of the literature *J Dent* **28(3)** 163-177.

2. Brodbelt RH, O'Brien WJ & Fan PL (1980) Translucency of dental porcelains *J Dent Res* **59(1)** 70-75.

3. Cekic-Nagas I, Egilmez F & Ergun G (2010) The effect of irradiation distance on microhardness of resin composites cured with different light curing units *Eur J Dent* **4(4)** 440-446.

4. Lohbauer U, Rahiotis C, Kramer N, Petschelt A & Eliades G (2005) The effect of different light-curing units on fatigue behavior and degree of conversion of a resin composite *Dent Mater* **21(7)** 608-615.

5. Uhl A, Sigusch BW & Jandt KD (2004) Second generation LEDs for the polymerization of oral biomaterials *Dent Mater* **20(1)** 80-87.

6. Ilie N & Hickel R (2008) Can CQ be completely replaced by alternative initiators in dental adhesives? *Dent Mater J* **27(2)** 221-228.

7. Park YJ, Chae KH & Rawls HR (1999) Development of a new photoinitiation system for dental light-cure composite resins *Dent Mater* **15(2)** 120-127.

8. Price RB, Labrie D, Rueggeberg FA & Felix CM (2010) Irradiance differences in the violet (405 nm) and blue (460 nm) spectral ranges among dental light-curing units *J Esthet Restor Dent* **22(6)** 363-377.

9. Rueggeberg FA (2011) State-of-the-art: dental photocuring--a review *Dent Mater* **27(1)** 39-52.

10. Price RB, Felix CA & Andreou P (2005) Evaluation of a dual peak third generation LED curing light *Compend Contin Educ Dent* **26(5)** 331-332, 334, 336-338 passim; quiz 348.

11. Price RB, Fahey J & Felix CM (2010) Knoop hardness of five composites cured with single-peak and polywave LED curing lights *Quintessence Int* **41(10)** e181-191.

12. Palin WM, Senyilmaz DP, Marquis PM & Shortall AC (2008) Cure width potential for MOD resin composite molar restorations *Dent Mater* **24(8)** 1083-1094.

13. Ilie N & Hickel R (2008) Correlation between ceramics translucency and polymerization efficiency through ceramics *Dent Mater* **24(7)** 908-914.

14. Pfeifer CS, Ferracane JL, Sakaguchi RL & Braga RR (2008) Factors affecting photopolymerization stress in dental composites *J Dent Res* **87(11)** 1043-1047.

15. Magne P, Versluis A & Douglas WH (1999) Effect of luting composite shrinkage and thermal loads on the stress distribution in porcelain laminate veneers *J Prosthet Dent* **81(3)** 335-344.

16. Rueggeberg FA & Craig RG (1988) Correlation of parameters used to estimate monomer conversion in a light-cured composite *J Dent Res* **67(6)** 932-937.

17. Rueggeberg FA, Hashinger DT & Fairhurst CW (1990) Calibration of FTIR conversion analysis of contemporary dental resin composites *Dent Mater* **6(4)** 241-249.

18. Arrais CA, Giannini M & Rueggeberg FA (2009) Kinetic analysis of monomer conversion in auto- and dual-polymerizing modes of commercial resin luting cements *J Prosthet Dent* **101(2)** 128-136.

19. el-Mowafy OM, Rubo MH & el-Badrawy WA (1999) Hardening of new resin cements cured through a ceramic inlay *Oper Dent* **24(1)** 38-44.

20. Rueggeberg FA, Caughman WF & Curtis JW, Jr. (1994) Effect of light intensity and exposure duration on cure of resin composite *Oper Dent* **19(1)** 26-32.

21. Sobrinho LC, Goes MF, Consani S, Sinhoreti MA & Knowles JC (2000) Correlation between light intensity and exposure time on the hardness of composite resin *J Mater Sci Mater Med* **11(6)** 361-364.

22. Daronch M, Rueggeberg FA & De Goes MF (2005) Monomer conversion of pre-heated composite *J Dent Res* **84(7)** 663-667.

23. Daronch M, Rueggeberg FA, De Goes MF & Giudici R (2006) Polymerization kinetics of pre-heated composite *J Dent Res* **85(1)** 38-43.

24. Watts DC (2005) Reaction kinetics and mechanics in photo-polymerised networks *Dent Mater* **21(1)** 27-35.

25. Calheiros FC, Kawano Y, Stansbury JW & Braga RR (2006) Influence of radiant exposure on contraction stress, degree of conversion and mechanical properties of resin composites *Dent Mater* **22(9)** 799-803.

26. Halvorson RH, Erickson RL & Davidson CL (2002) Energy dependent polymerization of resin-based composite *Dent Mater* **18(6)** 463-469.

27. Cook WD (1992) Photopolymerization kinetics of dimethacrylates using camphorquinone/amine initiator system *Polymer* **33(3)** 600-609.

4. CONCLUSÃO

Dentro das limitações deste estudo, foi possível concluir que:

- A presença de laminados cerâmicos interpostos entre a ponta da UFA e o cimento resinoso diminui a Tp_{max} sem comprometer o GC.

- A variação da translucidez da cerâmica não afetou o GC e a Tp_{max} da camada de cimento resinoso.

- A utilização de uma UFA de pico duplo não interferiu no GC e na Tp_{max} do cimento resinoso fotoativado através de laminados cerâmicos com diferentes níveis de translucidez.

REFERÊNCIAS BIBLIOGRÁFICAS

Arrais CA, et al. Kinetic analysis of monomer conversion in auto- and dual-polymerizing modes of commercial resin luting cements. J Prosthet Dent. 2009;101 (2): 128-136.

Calamia JR, Calamia CHS. Ceramic laminate veneers: reasons for 25 years of success. Dent Clin N Am. 2007;51: 399-417.

Brodbelt RH, et al. Translucency of dental porcelains. J Dent Res. 1980;59 (1): 70-75.

Calheiros FC, et al. Influence of radiant exposure on contraction stress, degree of conversion and mechanical properties of resin composites. Dent Mater. 2006;22 (9): 799-803.

Cekic-Nagas I, et al. The effect of irradiation distance on microhardness of resin composites cured with different light curing units. Eur J Dent. 2010;4 (4): 440-446.

Cook WD. Photopolymerization kinetics of dimethacrylates using camphorquinone/amine initiator system. Polymer. 1992;33 (3): 600-609.

Daronch M, et al. Monomer conversion of pre-heated composite. J Dent Res. 2005; 84 (7): 663-667.

Daronch M, et al. Polymerization kinetics of pre-heated composite. J Dent Res. 2006; 85 (1): 38-43.

Halvorson RH, et al. Energy dependent polymerization of resin-based composite. Dent Mater. 2002;18 (6): 463-469.

Ilie N, Hickel R. Can CQ be completely replaced by alternative initiators in dental adhesives? Dent Mater. 2008;27 (2): 221-228.

Ilie N, Hickel R. Correlation between ceramics translucency and polymerization efficiency through ceramics. Dent Mater. 2008;24 (7): 908-914.

Lohbauer U, et al. The effect of different light-curing units on fatigue behavior and degree of conversion of a resin composite. Dent Mater. 2005;21 (7): 608-615.

Magne P, Douglas WH. Interdental design of porcelain veneers in the presence of composite fillings: finite element analysis of composite shrinkage and thermal stresses. Int J Prosthodont. 2000;13 (2): 117-124.

Magne P, et al. Effect of luting composite shrinkage and thermal loads on the stress distribution in porcelain laminate veneers. J Prosthet Dent. 1999;81 (3): 335-344.

Palin W, et al. Cure width potential for MOD resin composite molar restorations. Dent Mater. 2008;24 (8): 1083-1094.

Park YJ, et al. Development of a new photoinitiation system for dental light-cure composite resins. Dent Mater. 1999;15 (2): 120-127.

Peumans M, et al. Porcelain veneers: a review of the literature. J Dent. 2000;28 (3): 163-177.

Pfeifer CS, et al. Factors affecting photopolymerization stress in dental composites. J Dent Res. 2008;87 (11): 1043-1047.

Price RB, et al. Evaluation of a dual peak third generation LED curing light. Compend Contin Educ Dent. 2005;26 (5): 331-338.

Price RB, et al. Knoop hardness of five composites cured with single-peak and polywave LED curing lights. Quintessence Int. 2010;41 (10): 181-191.

Price RB, et al. Irradiance differences in the violet (405 nm) and blue (460 nm) spectral ranges among dental light-curing units. J Esthet Restor Dent. 2010;22 (6): 363-377.

Rueggeberg FA. State-of-the-art: dental photocuring--a review. Dent Mater. 2011;27 (1): 39-52.

Rueggeberg FA, Craig RG. Correlation of parameters used to estimate monomer conversion in a light-cured composite. J Dent Res. 1988;67 (6): 932-937.

Rueggeberg FA, et al. Calibration of FTIR conversion analysis of contemporary dental resin composites. Dent Mater. 1990;6 (4): 241-249.

Rueggeberg FA, et al. Effect of light intensity and exposure duration on cure of resin composite. Oper Dent. 1994;19 (1): 26-32.

Ruyter IE e Györösi PP. An infrared spectroscopic study of sealants. Scand J Dent Res. 1976;84(6): 396-400.

Sobrinho LC, et al. Correlation between light intensity and exposure time on the hardness of composite resin. J Mater Sci Mater Med. 2000;11 (6): 361-364.

Uhl A, et al. Second generation LEDs for the polymerization of oral biomaterials. Dent Mater. 2004;20 (1): 80-87.

Watts DC. Reaction kinetics and mechanics in photo-polymerised networks. Dent Mater. 2005;21 (1): 27-35.

Wendl B, Droschl H, Kern W. A comparative study of polymerization lamps to determine the degree of cure of composites using infrared spectroscopy. Eur J Orthod. 2004; 26: 545-551.

ANEXOS

ANEXO A – Figura ilustrativa da pasta base do cimento resinoso utilizado neste estudo

Pasta base do cimento resinoso, Variolink II T (Ivoclar Vivadent).

ANEXO B – Figuras ilustrativas das unidades de fotoativação utilizadas neste estudo

LED de terceira geração, bluephase G2 (Ivocla
Vivadent).

LED de segunda geração, bluephase (Ivocla
Vivadent).

Unidade de lâmpada halógena (quartz-tungste
halogen – QTH), Optilux 501 (Kerr).

ANEXO C – Figura ilustrativa do espectroscópio infravermelho com transformada de Fourier (FTIR)
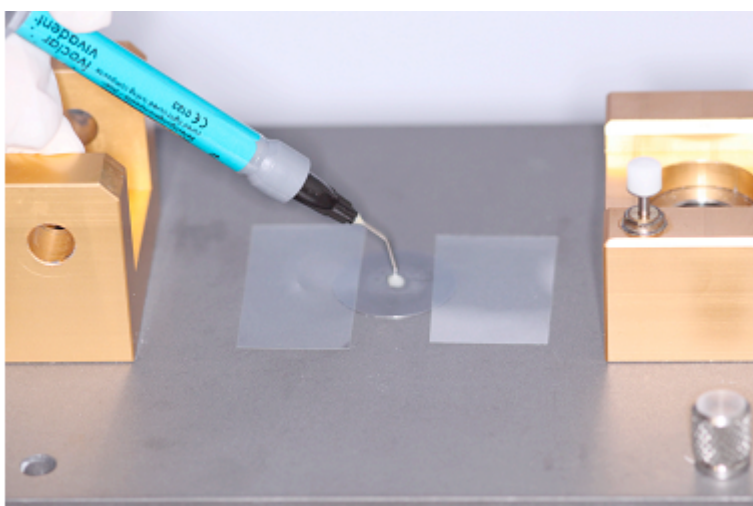

Espectrofôtometro infravermelho com transformada de Fourier – FTIR (Tensor 27, Bruker Comp), unidade de reflectância total atenuada – ATR (Satandard Golden Gate, Specac).
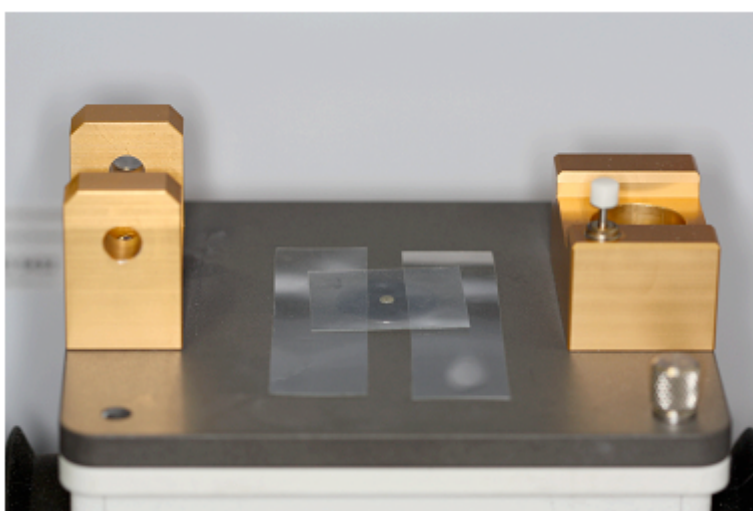
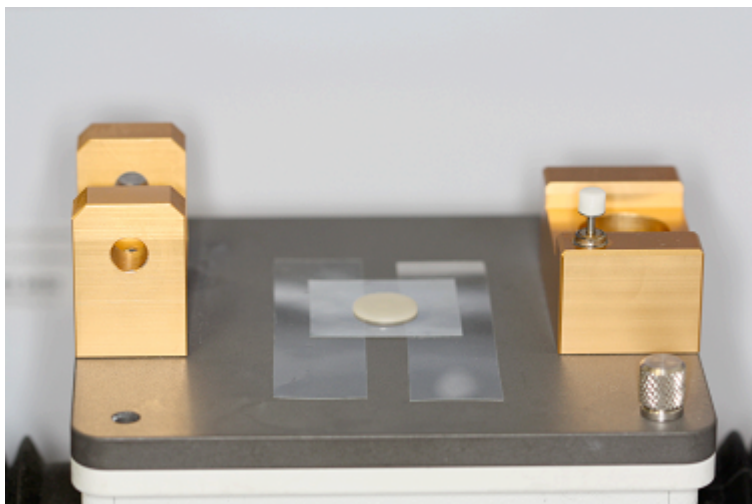**ANEXO D – Preparação dos espécimes**
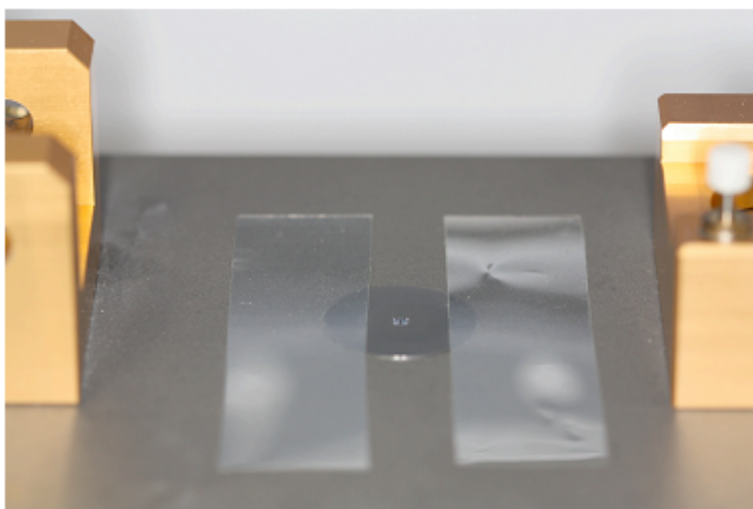
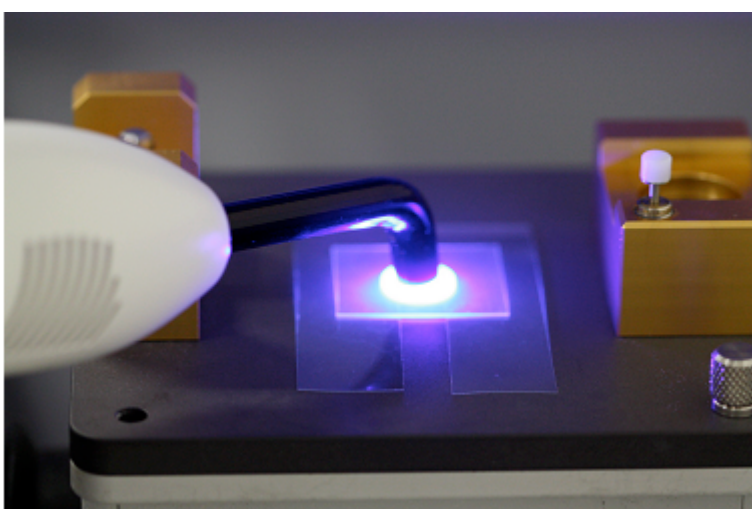
Posicionamento das tiras Mylar.

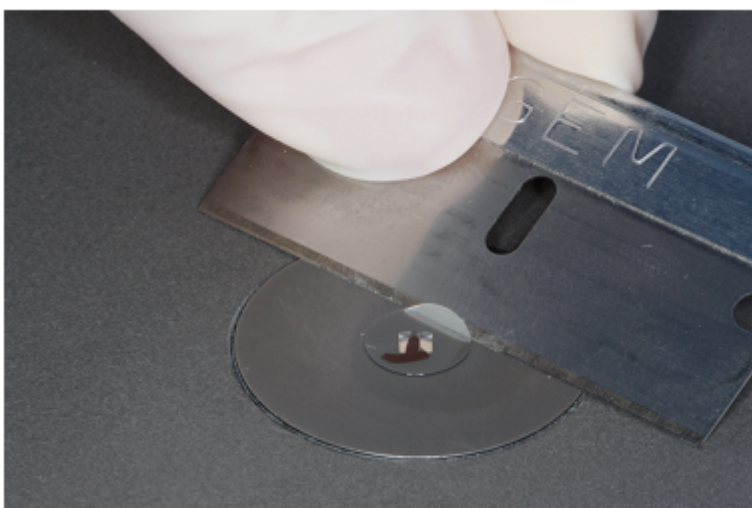Aplicação do cimento resinoso na superfície de diamante da mesa de ATR.

Posicionamento da tira Mylar sobre o cimento ainda não polimerizado.

Colocação do disco de cerâmica.

Fotoativação conforme recomendação dos fabricantes.

Remoção do filme de cimento superfície de diamante da mesa de ATR.

ANEXO E – Análise estatística para os resultados de grau de conversão

The GLM Procedure

Dependent Variable: DC

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	8	8.84396429	1.10549554	3.30	0.0045
Error	47	15.73157143	0.33471429		
Corrected Total	55	24.57553571			

R-Square	Coeff Var	Root MSE	DC Mean
0.359869	1.211386	0.578545	47.75893

Source	DF	Type I SS	Mean Square	F Value	Pr > F
ceramica	2	1.51374300	0.75687150	2.26	0.1154
foto	2	6.42631487	3.21315744	9.60	0.0003
ceramica*foto	4	0.90390642	0.22597660	0.68	0.6126

Source	DF	Type III SS	Mean Square	F Value	Pr > F
ceramica	2	1.89439288	0.94719644	2.83	0.0691
foto	2	6.37637157	3.18818578	9.53	0.0003
ceramica*foto	4	0.90390642	0.22597660	0.68	0.6126

Tukey's Studentized Range (HSD) Test for DC

NOTE: This test controls the Type I experimentwise error rate.

Alpha	0.05
Error Degrees of Freedom	47
Error Mean Square	0.334714
Critical Value of Studentized Range	3.42257

Comparisons significant at the 0.05 level are indicated by ***.

foto Comparison	Difference Between Means	Simultaneous 95% Confidence Limits	
3 - 2	0.4938	0.0203 0.9673	***
3 - 1	0.7913	0.3415 1.2410	***
2 - 3	-0.4938	-0.9673 -0.0203	***
2 - 1	0.2975	-0.1593 0.7543	
1 - 3	-0.7913	-1.2410 -0.3415	***
1 - 2	-0.2975	-0.7543 0.1593	

ANEXO F – Análise estatística para os resultados da taxa máxima de polimerização

Dependent Variable: rpmax3

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	8	92.5089631	11.5636204	47.07	<.0001
Error	53	13.0207143	0.2456739		
Corrected Total	61	105.5296774			

R-Square	Coeff Var	Root MSE	rpmax3 Mean
0.876616	5.981045	0.495655	8.287097

Source	DF	Type I SS	Mean Square	F Value	Pr > F
ceramica	2	72.61655837	36.30827919	147.79	<.0001
foto	2	16.85919458	8.42959729	34.31	<.0001
ceramica*foto	4	3.03321018	0.75830255	3.09	0.0234

Source	DF	Type III SS	Mean Square	F Value	Pr > F
ceramica	2	71.17561224	35.58780612	144.86	<.0001
foto	2	17.11125850	8.55562925	34.83	<.0001
ceramica*foto	4	3.03321018	0.75830255	3.09	0.0234

Least Squares Means

 Adjustment for Multiple Comparisons: Tukey-Kramer

ceramica	foto	rpmax3 LSMEAN	LSMEAN Number
1	1	10.0285714	1
1	2	9.3857143	2
1	3	9.9714286	3
2	1	7.6714286	4
2	2	6.4428571	5
2	3	8.2500000	6
3	1	7.6000000	7
3	2	6.9142857	8
3	3	8.3142857	9

Least Squares Means for effect ceramica*foto

Pr > |t| for H0: LSMean(i)=LSMean(j)

Dependent Variable: rpmax3

i/j	1	2	3	4	5	6	7	8	9
1		0.2916	1.0000	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
2	0.2916		0.4140	<.0001	<.0001	0.0040	<.0001	<.0001	0.0050
3	1.0000	0.4140		<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
4	<.0001	<.0001	<.0001		0.0007	0.4855	1.0000	0.1231	0.2916
5	<.0001	<.0001	<.0001	0.0007		<.0001	0.0018	0.6950	<.0001
6	<.0001	0.0040	<.0001	0.4855	<.0001		0.3284	0.0004	1.0000
7	<.0001	<.0001	<.0001	1.0000	0.0018	0.3284		0.2161	0.1741
8	<.0001	<.0001	<.0001	0.1231	0.6950	0.0004	0.2161		<.0001
9	<.0001	0.0050	<.0001	0.2916	<.0001	1.0000	0.1741	<.0001	