#### **MYRNA CARVALHO DIAS**

# INFLUÊNCIA DA MICROESTRUTURA E DA ESPESSURA DAS CERÂMICAS PRENSADAS NA TRANSMITÂNCIA DA LUZ E NA DUREZA KNOOP DO CIMENTO RESINOSO

Tese apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para obtenção do Título de Doutor em Materiais Dentários.

Orientador: Prof. Dr. Lourenço Correr Sobrinho Co-orientador: Prof. Dr. Evandro Piva

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A Comissão Julgadora dos trabalhos de Defesa de Tese de DOUTORADO, em sessão pública realizada em 11 de Outubro de 2007, considerou a candidata MYRNA CARVALHO DIAS aprovada.

PROF. DR. LOURENCO CORRER SOBRINHO PROF. DR. MÂNIO DE CARVALHO TIBURCIO PROF. DR. MURILO BAENA LOPES PROF. DR. SIMONIDES CONSANI \_ ' 2 x. Co

PROF. DR. RAFAEL LEONARDO XEDIEK CONSANI

Eu dedico este trabalho ao MENINO JESUS, sua e nossa mãe VIRGEM MARIA e ao seu e nosso pai adotivo SÃO JOSÉ

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"A maior recompensa para o trabalho do homem não é o que ele ganha com isso, mas o que ele se torna com isso."

John Ruskin

#### RESUMO

O objetivo deste estudo foi avaliar a influência da microestrutura e espessura das cerâmicas prensadas na intensidade e transmitância da luz e na eficiência da polimerização do cimento resinoso de dupla ativação Variolink II, fotoativado através de duas cerâmicas prensadas reforçadas com cristais de leucita. Foram confeccionados amostras das cerâmicas IPS Empress (EMP) e IPS Empress Esthetic (EST) com 0,7; 1,4 e 2,0mm e IPS Empress 2 (E2) com 0,8 e 1,1mm de espessura. E2 com 0,8mm foram cobertos com IPS Eris E2 dentina (D) para a obtenção de discos com 1,2 e 1,4mm. E2+D foram cobertos com IPS Eris esmalte (E) para alcançar a espessura final de 1,5mm. O mesmo procedimento foi realizado em E2 com 1,1mm (E2+D= 1,6 e 1,9mm e E2+D+E=2,0mm). A intensidade de luz gerada pelos aparelhos de fotoativação de lâmpada halógena convencional (XL 2500/3M ESPE) e LED (Ultrablue Is/DMC) foi verificada com radiômetro (Hilux), com e sem a presença da cerâmica sobre o sensor de leitura. O percentual de transmitância de luz através das cerâmicas foi obtido com auxílio do espectofotômetro Lambda 9. O grau de polimerização do Variolink II foi realizado em corpos-de-prova com 1,0mm de altura, fotoativados diretamente e indiretamente (sob EMP e EST com 0,7; 1,4 e 2,0mm), por 40 s utilizando o aparelho de lâmpada halógena convencional. A dureza Knoop foi analisada imediatamente e após 24 horas de armazenagem. A análise da microestrutura das cerâmicas EMP e EST foi verificada pelo MEV e por difração de raio-X. Os resultados mostraram que os valores de intensidade de luz emitidos pelas unidades de fotoativação sobre o EST foram estatisticamente superiores em relação ao EMP (p<0,05) e os valores obtidos pelo E2 foram maiores do que quando cobertos com D (p<0,05). Os valores de intensidade de luz diminuíram com o aumento da espessura das cerâmicas prensadas. A lâmpada halógena convencional gerou valores de densidade maiores do que o LED. Os gráficos espectrais obtidos pela transmitância da luz por meio das cerâmicas prensadas confirmaram os resultados alcançados pela intensidade de luz. O valor de dureza

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do cimento polimerizado sob o EST foi maior do que sob EMP (p<0,05), o grau de polimerização do cimento foi maior após 24 horas de armazenagem (p<0,05) e os discos com 2,0mm de espessura promoveram dureza menor do cimento após 24 horas (p<0,05). O MEV mostrou que os cristais de leucita no EST apresentaramse mais densamente distribuídos na matriz vítrea do que no EMP. A difração de raio-X confirmou a mesma cristalinidade do EMP e EST. Concluiu-se que as alterações na microestrutura e espessura das cerâmicas prensadas influenciaram na intensidade e transmitância de luz e o EST promoveu melhor grau de polimerização do cimento resinoso Variolink II que o EMP.

Palavras-chave: Cerâmica, Materiais Dentários, Microscopia eletrônica de Varredura, Cimentos de Resina, Dureza, Difração de Raios X

### ABSTRACT

The aim of this study was to evaluate the influence of the microstruture and thickness of the pressable ceramic in the intensity and light transmittance and the polymerization efficiency of the dual resin-based cement Variolink II cured beneath two hot-pressed ceramic reinforced with leucite crystals. IPS Empress (EMP) and IPS Empress Esthetic (EST) with 0.7; 1.4 and 2.0mm and IPS Empress 2 (E2) with 0.8 and 1.1mm of thickness were made. E2 with 0.8mm were covered with IPS Eris E2 dentin (D) for obtaining disks with 1.2 and 1.4mm. E2+D were covered with IPS Eris E2 enamel (E) to reach the final thickness of 1.5mm. The same procedure was realized in E2 with 1.1mm (E2+D = 1.6 and 1.9mm and E2+D+E=2.0mm). The light intensity emitted by the light curing units guartztungsten-halogen (XL 2500/3M ESPE) and LED (Ultrablue Is/DMC) was assessed by the digital radiometer Hilux with na without the presence of the disks of ceramic on sensor. The percentage of light transmittance for the pressable ceramic was obtained by the spectrophotometer Lambda 9. The degree of polymerization of Variolink II was realized from resin cements specimens with 1.0mm of height, cured directly and indirectly (under EMP and EST with 0.7; 1.4 and 2.0mm), for 40s using the light curing units guartz-tungsten-halogen. The hardness Knoop was analyzed the immediately and 24 hours after photoactivation. The analysis of the microstructure of the pressable ceramic was realized by EDX and SEM and for the X-ray diffraction of EMP and EST. The results showed that the values of light intensity emitted for light curing units on EST were higher than of EMP (p < 0.05) and the values obtained by E2 were larger than when covered with D (p < 0.05). The values of light intensity decreased with the increase of the thickness of the pressable ceramic. The quartz-tungsten-halogen emitted light intensity higher than LED. The graphs obtained by the light transmittance through the pressable ceramic confirmed the results reached by the light intensity. The value of hardness of the cement cured under EST was higher than under EMP (p < 0.05), the degree of polimerization of the cement was higher after 24 hours of storage (p < 0.05) and

disks with 2.0mm of thickness obtained a smaller hardness of the cement after 24 hours (p < 0.05). MEV showed that the leucite crystals in EST presented more homogeneous distributed in the glassy matrix than in EMP. EDX revealed the presence of titanium oxide in composition of D. The ray-X diffraction confirmed the same cristallinity of EMP and EST. It was concluded that changes in the microstructure and thickness of the pressable ceramic influenced in the light intensity and transmittance and EST showed a better degree of polimerization of the dual resin-based cement than EMP.

Key Words: Ceramics, Dental Materials, Scanning Electron Microscope, Resin cements, Hardness, X-Ray diffraction

# **SUMÁRIO**

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## INTRODUÇÃO

As restaurações em cerâmicas apresentam excelente qualidade estética e compatibilidade biológica, embora sua friabilidade e potencial de abrasão as tornem susceptíveis à fratura (Denry, 1996; Qualtrough & Piddock, 1997). O desenvolvimento, nos últimos anos, de materiais para a confecção de restaurações livres de metal tem ocorrido na tentativa de melhorar as propriedades mecânicas dos mesmos, sem colocar em prejuízo a qualidade estética. Um dos métodos utilizados para aprimorar a resistência das cerâmicas vítreas é o aumento do conteúdo cristalino, pela inclusão de quantidades elevadas de cristais. Além disso, técnicas de processamento empregadas na fabricação de materiais cerâmicos de alta tecnologia, como prensagem a quente e infiltração de vidro, têm sido aplicadas na Odontologia para a confecção deste tipo de restauração (Denry, 1996).

Alguns materiais cerâmicos processados pela prensagem a quente são as pastilhas de vidros ceramizados IPS Empress (EMP) e IPS Empress 2 (E2) (Ivoclar Vivadent). O EMP é um vidro ceramizado reforçado com 30 a 40% de cristais de leucita (Höland *et al.*, 2000), lançado no mercado em 1990, e recomendado para confecção de inlays, onlays, veneers e coroas anteriores (Guazzato *et al.*, 2004). Por outro Iado, o E2 surgiu em 1998 (Guazzato *et al.*, 2004), apresentando 65 a 75% de cristais de dissilicato de lítio dispersos na matriz vítrea (Höland *et al.*, 2000), com alta resistência, sendo indicado para confecção de infraestrutura de coroas unitárias e prótese fixa de 3 elementos até 2º pré-molar

(Cattell *et al.*, 2002). Em 2004, um novo produto à base de leucita, intitulado IPS Empress Esthetic (EST), foi lançado no mercado, apresentando estrutura mais homogênea devido a melhor compactação dos cristais de leucita que, por sua vez, são menores comparados ao do EMP (Bülher-Zemp, 2004).

A peça protética obtida pela técnica de prensagem a quente é obtida pela confecção de um padrão de cera, que após ser incluído no revestimento é levado ao forno para sua volatilização; permitindo que a pastilha de vidro ceramizado pré-aquecida seja inserida no molde para ser pressionada por meio de um pistão de alumina em um forno de pressão pneumática (Cattell *et al.*, 2001). Os benefícios das cerâmicas prensadas em relação às cerâmicas sinterizadas, que é a técnica de processamento mais utilizada, são a diminuição de porosidade, aumento da resistência à flexão e melhor ajuste marginal (Gorman *et al.*, 2000).

Para promover a união entre a peça protética e a estrutura dental, é necessária a utilização de um agente de cimentação. Tradicionalmente, o cimento de fosfato de zinco foi o material mais popular, apesar das desvantagens como solubilidade e falta de adesão. Assim, os cimentos resinosos têm sido introduzidos como alternativas aos os cimentos de fosfato de zinco, principalmente por não apresentarem limitações, como qualidade marginal deficiente, fratura e perda de retenção (Rosenstiel *et al.*, 1998, Hofmann et al., 2001).

Os cimentos resinosos podem ser classificados em três categorias de acordo com o método de polimerização: ativados quimicamente, sendo utilizados para fixação de restaurações metálicas; fotoativados, empregados na fixação de veneers de cerâmica; e, de dupla ativação, que são utilizados para fixação de restaurações inlay e onlay, restauração de resina composta indireta e coroas de

cerâmica (el-Mowafy *et al.*, 1999). Os cimentos resinosos de dupla ativação polimerizam quimicamente por meio da mistura dos componentes base e catalisador e quando submetidos à luz ativadora de uma unidade de fotoativação (el-Badrawy & el-Mowafy, 1995; el-Mowafy *et al.*, 1999).

A recomendação da utilização de cimentos resinosos de dupla ativação para a fixação de restaurações indiretas tem como finalidade compensar a atenuação da luz proporcionada pelo material interposto e, dessa forma, tentar assegurar a completa polimerização do agente cimentante mesmo em locais em que o acesso da luz é limitado (el-Badrawy & el-Mowafy, 1995; el-Mowafy *et al.*, 1999). Para ativação do fotoiniciador canforoquinona, o mais comumente presente em materiais fotoativados (Rueggeberg, 1999), e desencadeamento da reação de polimerização, é essencial que a luz apresente comprimento de onda entre 450 e 500 nm e uma irradiância mínima por algum tempo de exposição (Davidson & de Gee, 2000; Rueggeberg, 1999).

Entretanto, o material cerâmico pode provocar a diminuição da passagem de luz, principalmente em restaurações mais espessas (Chan & Boyer, 1989; Blackman *et al.*, 1990; Warren, 1990; Linden *et al.*, 1991; Uctasli *et al.*, 1994; el-Badrawy & el-Mowafy, 1995; el-Mowafy *et al.*, 1999; Barghi & McAlister, 2003; Jung *et al.*, 2006), com cores escuras (Chan & Boyer, 1989; Cardash *et al.*, 1993; Barghi & McAlister, 2003) opacas (Linden *et al.*, 1991; Uctasli *et al.*, 1993; Barghi & McAlister, 2003) opacas (Linden *et al.*, 1991; Uctasli *et al.*, 1994), com diferentes componentes (Blackman *et al.*, 1990; Rasetto *et al.*, 2001) e com presença de cristais de reforço (quantidade, tamanho e natureza química) (Heffernan *et al.*, 2002). A atenuação da passagem de luz pode comprometer a polimerização do cimento, podendo interferir no desempenho das restaurações

(Uctasli *et al.*, 1994). O enfraquecimento da união entre a cerâmica e a estrutura dentária pode predispor à sensibilidade pós-operatória, devido à solubilidade do material de fixação, com subseqüente microinfiltração e cárie recorrente (el-Mowafy *et al.*, 1999).

Dentro destes aspectos, parece oportuno estudar a influência da microestrutura de cerâmicas prensadas na irradiância provida por aparelhos fotoativadores, transmitância da luz, eficiência da polimerização através de cerâmicas prensadas, pela dureza Knoop. O presente trabalho é apresentado no formato alternativo de tese de acordo com as normas estabelecidas pela deliberação 002/06 da Comissão Central de Pós-Graduação da Universidade Estadual de Campinas. Os artigos referentes aos Capítulos 1 e 2 serão submetidos aos periódicos Clinical Oral Investigations e Journal of Materials Science: Materials in Medicine e são apresentados conforme formatação solicitada pelos periódicos. Assim, os objetivos deste trabalho foram divididos em dois capítulos que pretendem:

- CAPÍTULO 1: Avaliar a influência da microestrutura de cerâmicas prensadas na irradiância provida por dois aparelhos fotoativadores, quando a luz é transmitida através do material indireto, e na transmitância da luz por meio de análise espectral.
- CAPÍTULO 2: Verificar a eficiência da polimerização, por meio da dureza Knoop, de um cimento resinoso dual fotoativado diretamente e indiretamente sob duas cerâmicas prensadas reforçadas com cristais de leucita.

# **CAPÍTULO 1**

# UV-Vis Spectrophotometric Analysis and Light Irradiance through Hot-Pressed and Hot-Pressed-Veneered Glass Ceramics

Artigo submetido ao periódico: Clinical Oral Investigations.

#### Abstract

The aim of this in vitro study was to evaluate the light irradiance of curing units through core and veneered hot-pressed ceramics with different thickness and also to evaluate the transmittance of these materials. Discs of 0.7, 1.4 and 2.0 mm in thickness of IPS Empress (EMP) and IPS Empress Esthetic (EST), and with 0.8 (n = 5) and 1.1mm (n = 5) of IPS Empress 2 (E2) were obtained. For E2, 2 discs with 0.8 mm were covered with dentin (1.2 and 1.4mm in thickness) and 2 with dentin + enamel (1.5 mm in thickness). The specimens with 1.1 mm in thickness were submitted to the same veneering procedures. All specimens were evaluated by UV-Vis light transmittance analysis (300-600 nm range) and the percentage of direct transmittance was recorded. Also, the light irradiance through each specimen was evaluated with a quartz-tungsten-halogen (QTH - XL2500, 3M ESPE), used in continuous or intermittent exposure mode, or a blue light-emitting diode (LED – Ultrablue Is, DMC). Data were analyzed by Dunnett's test and ANOVA followed by Tukey's (5%). For EST and EMP, exposure through ceramic significantly reduced the light intensity. Irradiance through EST was significantly higher than EMP. For E2, reduction in irradiance through ceramic, depending on the core and/or veneer thickness, was detected. In general, QTH intermittent mode showed higher irradiance than the continuous mode, but both showed higher irradiance than LED. In conclusion, the ceramic material presented a significant influence on light irradiance and transmittance, which were found to decrease with the increase in ceramic thickness.

Key Words: ceramics, dental materials, dental prosthesis, leucite, lithium.

#### 1. Introduction

New high-technology processes have led to the development of a wide range of dental ceramics [1,10,12]. These materials use different approaches in an attempt to improve the mechanical properties of the ceramic without being detrimental to their esthetic qualities [10]. Some of the most representative materials are the pressable and glass-infiltrated ones. IPS Empress (EMP) and Empress 2 (E2) are two well-known hot-pressed materials [12] and, more recently, the, IPS Empress Esthetic (EST) was introduced [5].

EMP is a leucite-reinforced glass ceramic designed for restoring single units including veneers, inlays, onlays and crowns [12,16]. E2 was developed to produce crown and bridge substructures [8,12], due to its new lithium disilicate-based microstructure, with high amounts of crystals dispersed within the glass matrix [16]. EST is also a leucite-based material, however with smaller crystals size distributed in a more homogeneous mode than EMP [5]. On the other hand, the veneering ceramic applied to E2 cores is composed of fluoroapatite crystals precipitated in a glassy matrix [16].

Resin-based cements are the materials of choice for ceramic luting procedures because of the brittle characteristic of these ceramic materials. Many of the available resin-based cements are dual-cured materials, that polymerize chemically upon mixing of a base and catalyst components and when exposed to a polymerizing light of a curing unit, due to the presence of a photo-initiator [11]. This molecule is decomposed in the presence of light with an adequate wavelength and sufficient irradiance [22,24]. In fact, in order to obtain high bond strengths after

cementation, adequate light energy reaching the luting agent is required to ensure optimal polymerization [20].

Nonetheless, several investigators have reported on the light attenuation effect promoted by ceramics [3,4,7,11,18,19,21,25]. The degree of this attenuation is primarily dependent on the characteristics of the restorative, such as its composition, thickness, opacity and shade [4,7,11,19]. Combination of scattering, reflecting and absorbing properties at the outer surface of the intervening material may explain the reduction in the incident light [14]. Indeed, several studies have evaluated the effect on irradiance for light passing through ceramic. However, few evaluations are reported in literature regarding the transmittance characteristics of ceramics and its relationship with the attenuation of the polymerizing light.

The aim of this in vitro study was to evaluate the light irradiance of curing units through core and veneered hot-pressed ceramics of different thickness and also to evaluate the transmittance of these materials.

#### 2. Materials and Methods

The glass ceramics evaluated were: IPS Empress (EMP), IPS Empress 2 (E2), IPS Empress Esthetic (EST), and IPS Eris for E2 dentin (D) and enamel (E), all from lvoclar Vivadent (AG, Schaan, Liechtenstein). For EMP e EST, 3 discs with 10 mm in diameter and 0.7, 1.4 or 2 mm in thickness were obtained. For E2, discs with 10 mm in diameter and 0.8 (n = 5) or 1.1 mm in thickness (n = 5) were obtained, and divided according to the veneering material (D or E) to be applied, as shown in Figure 1. The dimensions of each specimen were confirmed with a digital caliper (Starrett Ind. Com. Ltda., Itu, Brazil).

#### Diffuse light transmittance analysis

Diffuse light transmittance measurements were performed in the 300 to 600 nm wavelength range using a UV-Vis spectrophotometer (Lambda 9, Perkin Elmer, Shelton, USA), equipped with an integrating sphere. A black rectangular cardboard segment (4 x 4 cm), with a central orifice of 10mm in diameter, was used to position the specimens in front of the holder of the sphere. Data were recorded with a computer connected to the spectrophotometer, and a graph of light transmittance percentage x spectrum was obtained with software (Origin 6.0, Microcal Software Inc, Northampton, USA).

#### Light irradiance analysis

The irradiance through the ceramic specimens was evaluated using two light-curing units: a quartz-tungsten-halogen (XL2500, 3M ESPE; St. Paul, USA) and a blue light-emitting diode (Ultrablue Is, DMC Equip. Ltda., Sao Carlos, Brazil). The light intensity was assessed with a handheld radiometer (Hilux Dental Curing Light Meter, Benbionglu Dental Inc., Turkey), under controlled humidity (50±10%) and temperature (23±2°C) conditions [17].

The light-curing units were connected to a voltage stabilizer and the light guide was placed perpendicular to the radiometer detector (control group) or directly onto to the surface of the specimens. Ten exposures of 40 s each were sequentially carried out and the irradiance recorded every 10 s. Additionally to the continuous exposure mode, for the QTH unit, an intermittent method was tested, in which each 40 s exposure was started only after stopping the cooling fan of the curing unit.

Data for EMP and EST groups were submitted to a  $2 \times 3 \times 3$  factorial design ANOVA (ceramic x thickness x curing method), with 3 additional treatments (control for each curing method). The factors were compared with their respective control groups with Dunnett's test, and between each other with Tukey's test. Data for E2 core and veneered groups were submitted to a 11 x 3 factorial design ANOVA (ceramic x light-curing unit), followed by the Tukey's test. All statistical analyses were conducted at a significance level of p < 0.05.

#### 3. Results

#### Diffuse light transmittance

The results of light transmittance through EMP and EST specimens are shown in Figure 2, and through core and veneered E2 are shown in Figure 3. There was a reduction in percentage of light transmittance depending on the thickness of the ceramic. EST specimens showed higher percentage of light transmittance than EMP for all thicknesses. This reduction was higher for E2 specimens veneered with D compared with E2+D specimens veneered with E. Furthermore, in both Figures 2 and 3, it can be observed that the transmittance percentage increased with increased wavelength, irrespective of the ceramic material or thickness.

#### Light irradiance

The results for EMP and EST specimens are shown in Table 1. All groups irradiated through ceramic showed a significant reduction in light intensity when compared with the control group (p < 0.05), with a gradual reduction as a function

of increasing ceramic thickness. However, the irradiance through EST was significantly higher than EMP (p < 0.05), irrespective of the ceramic thickness or curing method. In addition, the QTH intermittent mode showed significantly higher irradiance than the continuous mode (p < 0.05), regardless of the ceramic material or thickness. Furthermore, the LED unit showed significantly lower irradiance than the QTH unit (p < 0.05), even when the QTH was used in the continuous mode.

The results for E2 core and veneered specimens are shown in Table 2. All groups showed a significant reduction in light irradiance, which was dependent on the core and/or veneer ceramic thickness, when compared with the control group (p < 0.05). Irrespective of the curing method, irradiance through E2 with 0.8 mm was significantly higher than irradiance through 1.1 mm (p < 0.05). Both E2 specimens veneered with D and D+E caused a significant and gradual reduction in light irradiance (p < 0.05) with increasing the veneer thickness. Moreover, the QTH intermittent mode showed higher irradiance through 1.2, 1.4 and 1.5 mm-thick veneered specimens than LED and QTH continuous modes (p < 0.05), although similar findings were detected for all exposure modes when irradiance was carried out through 1.6, 1.9 and 2 mm-thick veneered ceramics.

#### 4. Discussion

The present results show a significant attenuation of irradiance and transmittance through almost all ceramic specimens. In clinical situation, this may account for inadequate light energy reaching the cement layer during luting procedures, potentially resulting in poor polymerization. Indeed, the curing reaction is initiated and sustained when light energy of sufficient intensity and suitable

wavelength excites a great number of photo-initiator molecules, thus producing a sufficient number of free radicals [22,24]. Inadequate curing leads to poor mechanical properties and increased solubility of the cement [11,18], which may result in debonding of ceramic restoration over the course of time [3].

The present outcomes showed that the decrease in light irradiance and transmittance were dependent upon the ceramic thickness. This arises from a fact that the translucency/opacity of a ceramic is dictated by thickness. The thicker the specimen, the more opaque is the material and, as a consequence, lower light energy can be transmitted through it [2,6,14,19,25]. Besides thickness, the presence of specific elements can also contribut to the increase in opacity, as occurred in E2 veneered with D. The addition of titanium oxide to obtain an yellow-brown shade that matches that of the natural teeth [9], increases the light absorption [26].

In fact, when comparing EST and EMP specimens, the transmittance and irradiance through EST was always significantly higher than EMP, for all thicknesses. The amount of the light being absorbed, reflected and transmitted depends in large part on the amount of crystals within the glassy matrix [2,14,26] and the size of the particles compared with the incident light wavelength [14,26]. Although glass ceramics reinforced with leucite crystals present less amounts of grains - 35±5 vol% of leucite for EMP in comparison to 70±5 vol% of lithium disilicate for E2 [16] - the size of the leucite crystals dispersed within the EST glass matrix allowed a high amount of light to pass through this material. However, a maximum scattering effect occurs when the size of the crystal is approximately half [23] or similar [14] to the incident light wavelength.

Another factor that interferes with light transmission is the difference in the refractive index between the crystals and the glassy matrix. The refractive index is a measure for how much the speed of light is reduced inside a medium, and a maximum scattering effect is expected when there is a large mismatch between the index of the particles and the glassy matrix. Leucite (1.51) and lithium disilicate (1.55) have similar indexes to the glassy matrix (1.50) [14,15].

However, the presence of porous in these glass ceramics might have higher influence on the light transmission than the particles themselves. The mismatch between the refractive index of the pore (1.00) and of the glass matrix [14,15,26]. and the fact that the pore size has similar magnitude to the light wavelength, may lead to a significant light scattering effect [26].

With respect to the ceramic restoration made by the veneering, layering technique, other factors might affect the light transmission, such changes in the constituents' core material because of additional firing cycles, porosity between the layers, and reflectance at the interface between core and veneering ceramic [15]. Nonetheless, the idea that an increase in the crystalline content of E2 veneered with the D+E occurs during firing cycles is contested by Cattell *et al.* [8]. The authors showed, by means of X-ray diffraction analysis, that no significant crystalline phase change takes place for the core ceramic when this receives heat treatment for the firing cycles of the veneering materials.

Moreover, it was observed that the light transmittance increased with increasing wavelength. This is in accordance with the Rayleigh scattering equation [6], which states that higher scattering occurs at lower wavelengths. Such an observation is important when considering the spectral emission of the curing units

and the absorbance peak of the photo-initiator. For instance, camphorquinone has an absorbance peak at 468 nm [22,24], while 1-phenyl-1,2-propanedione (PPD) at 410nm, bisacylphosphine oxide (BAPO) from 320 to 390nm and triacylphosphine oxide (TPO) at 381nm [24]. Therefore, camphorquinone-containing resins would be more effective in polymerizing beneath ceramic in comparison to materials containing PPD, BAPO or TPO, because of the higher attenuation of transmittance at lower wavelengths.

In addition, the QTH intermittent mode generally showed significantly higher irradiance than the continuous mode. The large amount of infrared light emitted from QTH units produces a great amount of heat, which must be filtered out and discarded by mean of intense fan-cooling [13,22]. The absence of adequate stream of cooling air through the unit may predispose early bulb failure and cause a decrease in light intensity [22]. The present results confirm that allowing the operation of the fan cooling without interruption is essential for optimizing the irradiance level.

On the other hand, the LED unit showed significantly lower irradiance than the QTH unit, even when this was used in the continuous mode, except for E2 veneered. It has been previously reported that LED units are the most efficient ones, because are capable in converting electrical current into correct wavelength similar to the absorption wavelength of camphorquinone [13]. However, besides the spectral emission within the blue region of the visible spectrum (400 to 515nm), the irradiance level should be at least 300 mW/cm<sup>2</sup> [17]. This amount of light intensity was emitted by the LED only when EST with 0.7mm in thickness was tested. Therefore, the current outcomes suggest that cares should be taken when

polymerizing resin luting agent ceramic restorations, independent of the light curing unit used.

#### 5. Conclusion

The ceramic material presented a significant influence on light irradiance and transmittance, which were found to decrease with the increase in ceramic thickness. The QTH intermittent exposure mode produced higher irradiance than the continuous exposure mode, and both modes showed higher irradiance through ceramic compared with the LED curing unit.

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Group	Exposure mode	Thickness (mm)		
Gloup		0.7	1.4	2.0
	QTH continuous	355 (19) <sup>A,b</sup>	218 (9) <sup>B,b</sup>	149 (8) <sup>C,b</sup>
Empress	QTH intermittent	354 (4) <sup>A,a</sup>	247 (4) <sup>B,a</sup>	160 (4) <sup>C,a</sup>
	LED	262 (7) <sup>A,c</sup>	163 (3) <sup>B,c</sup>	106 (2) <sup>C,c</sup>
	QTH continuous	422 (17) <sup>A,b</sup>	263 (13) <sup>B,b</sup>	188 (8) <sup>C,b</sup>
Esthetic	QTH intermittent	457 (6) <sup>A,a</sup>	289 (3) <sup>B,a</sup>	205 (2) <sup>C,a</sup>
	LED	309 (9) <sup>A,c</sup>	189 (4) <sup>B,c</sup>	134 (3) <sup>C,c</sup>
	QTH continuous		752 (26)*	
Control	QTH intermittent		802 (10)*	
	LED		525 (13)*	

Table 1. Means (standard deviation) for irradiance (mW/cm<sup>2</sup>) through IPS Empress and IPS Empress Esthetic specimens.

Means followed by distinct capital letters in the same line, and small letters in the same column, were significantly different (Tukey's test, p < 0.05). \*All groups were significantly different from their respective control groups (Dunnett's test, p < 0.05).

	Group	Exposure mode		
	Group	QTH continuous	QTH intermittent	LED
	Control	752 (27) <sup>B,a</sup>	802 (10) <sup>A,a</sup>	525 (12) <sup>C,a</sup>
2 E E	Empress 2 (0.8mm)	224 (10) <sup>B,b</sup>	237 (3) <sup>A,b</sup>	154 (4) <sup>C,b</sup>
	Empress 2 (1.1mm)	149 (7) <sup>B,c</sup>	161 (3) <sup>A,c</sup>	104 (2) <sup>C,c</sup>
	Empress 2 + dentin (0.8 + 0.4 = 1.2mm)	69 (4) <sup>AB,d</sup>	77 (2) <sup>A,d</sup>	60 (2) <sup>B,d</sup>
	Empress 2 + dentin (0.8 + 0.6 = 1.4mm)	53 (3) <sup>AB,ef</sup>	61 (2) <sup>A,e</sup>	47 (2) <sup>B,e</sup>
	Empress 2 + dentin (1.1 + 0.5 = 1.6mm)	33 (3) <sup>A,g</sup>	36 (2) <sup>A,g</sup>	28 (2) <sup>A,f</sup>
	Empress 2 + dentin (1.1 + 0.8= 1.9mm)	17 (2) <sup>A,h</sup>	20 (1) <sup>A,h</sup>	16 (1) <sup>A,g</sup>
	Empress 2 + dentin + enamel (0.8 + 0.4 + 0.3 = 1.5mm)	61 (4) <sup>AB,de</sup>	70 (2) <sup>A,de</sup>	54 (2) <sup>B,de</sup>
	Empress 2 + dentin + enamel (0.8+ 0.6 + 0.1 = 1,5mm)	50 (4) <sup>B,f</sup>	57 (1) <sup>A,f</sup>	44 (2) <sup>B,e</sup>
	Empress 2 + dentin + enamel (1.1 + 0.5 + 0.4 = 2.0mm)	25 (2) <sup>A,g</sup>	30 (2) <sup>A,g</sup>	23 (1) <sup>A,fg</sup>
	Empress 2 + dentin + enamel (1.1 + 0.8 + 0.1 = 2.0mm)	16 (2) <sup>A,h</sup>	18 (1) <sup>A,h</sup>	14 (1) <sup>A,g</sup>

Table 2. Means (standard deviation) for irradiance (mW/cm<sup>2</sup>) through IPS Empress 2 core and veneered specimens.

Means followed by distinct capital letters in the same line, and small letters in the same column, were significantly different (Tukey's test, p < 0.05).



Figure 1 – Description of the ceramic specimens evaluated in the study.



Figure 2 – UV-Vis spectrum of IPS Empress and IPS Empress Esthetic specimens.



Figure 3 – UV-Vis spectrum of IPS Empress 2 core and veneered specimens.

# **CAPÍTULO 2**

# Polymerization efficiency of dual resin-based cements

# through heat-pressed ceramics

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#### Abstract

The aim of this in vitro study was to evaluate the polymerization efficiency of dual resin-based cements through heat-pressed ceramics. Twenty specimens dual resin-based cements Variolink II (Ivoclar/Vivadent) were made and polymerized by light-curing unit guartz-tungsten-halogen XL 2500 (3M ESPE) for 40s, directly (without ceramic) and indirectly (IPS Empress – EMP and IPS Empress Esthetic -EST with 0.7, 1.4 and 2.0mm). On 10 specimens, the Knoop hardness of resin cement was analyzed immediately and the remaining 24 hours after light-curing. Three depths (100, 500 and 900µm) were evaluated and 3 indentations were performed in each depth. Data were analyzed by Dunnett's test and ANOVA followed by Tukey's test (5%). EMP and EST specimens were made for X-ray diffraction analysis and scanning electron microscopy analysis (SEM). The highest value hardness of resin cement was obtained when polymerized under EST specimen (p<.05), after 24 hours of storage (p<.05) and when disks EMP and EST specimens of 0.7 and 1.4mm were utilized. Difference in size of leucite crystals between EST and EMP were not detected by the X-ray diffraction. The SEM showed that in EST the leucite crystals present distributed more homogeneous in the glassy matrix when comparison at EMP specimen. In conclusion, the EST permitted better polymerization than EMP, the thicker the glass ceramics (2mm) decreased the degree of hardness; dual polymerization improved the hardness of resin-based cement (24 hours).

**Key Words:** resin cements, ceramics, hardness, X-Ray diffraction, dental materials, microscopy.

#### 1. Introduction

The development of novel processing techniques along at patient demands for superior aesthetics have led to revival of all ceramic restorations [15, 29, 8, 19]. With these systems it is possible to produce single crowns, inlays, onlays and veneers similar to natural tooth [15].

Traditionally, the link between a fixed prosthesis and the supporting prepared tooth structure has been carried out with zinc phosphate and glass ionomer cements. Due its disadvantages, as solubility [31] and reduction resistance, and with the approach of the adhesion technique, the bonding of ceramic materials has been required by means of resin-based cements [28].

For cementation of ceramic restorations, dual-based resin cements are typically used [17, 23, 34, 18, 24], because conciliate favorable characteristics of self-cured and photo-activated cements, allowing an adequate polymerization of material in the presence or absence of light [16, 17, 4, 34, 18].

However, the ceramic placement between light guide and resin cement may lead insufficient adhesion [27] and increase water absorption [17, 23, 27, 24], due to some aspect of indirect material that must be carried in consideration as thickness [1, 12, 3, 36, 26, 35, 16, 17, 2, 24], shade [12, 7, 2], opacity [26, 35], and composition [3, 30]. Theses features decrease the light irradiance, provoking an inadequate curing of luting material [3, 35, 30, 2].

Beyond these features, the amount and size of the crystals dispersed glassy matrix also compromise the amount light transmitted through ceramic [21]. The ingot the glass ceramic IPS Empress and IPS Empress Esthetic although contain amount similar of leucite crystals, the size of the crystals of IPS Empress Esthetic

is smaller leucite crystals when compared with leucite crystals dispersed in glassy matrix of IPS Empress [6].

The aim of this in vitro study was to evaluate the polymerization efficiency of dual resin-based cements through heat-pressed ceramics with different microstructure and thickness. The work hypothesis was that there is no change in the curing resin cements when microstructure and thickness glass ceramics is changed.

#### 2. Materials and Methods

A total of 3 disks of IPS Empress (EMP) and 3 of IPS Empress Esthetic (EST), from Ivoclar Vivadent (AG, Schaan, Liechtenstein), were fabricated by laboratory according with manufacturer recommendation. The glass ceramics disks were milled to the 0.7, 1.4 e 2.0mm thickness and the measurement the thickness of each disk was checked with an digital caliper (Starrett Industria e Comércio Ltda, Itu, Brasil) to ensure standardized specimens.

#### **Resin cement specimens**

An standardized amount of base (shade 210/A3) and catalyst of low viscosity (transparent) dual resin-based cement Variolink II (Ivoclar Vivadent AG, Schaan, Liechtenstein) were mixed according to the manufacturer's recommendations. The cement was placed inside an addition silicone mold (Aquasil<sup>™</sup> ULV, Dentsply, Konstanz, Germany) with 5mm diameter and 1mm high, positioned on bovine dentin disk and between its was placed a polyester strip.

The resin cement was activated by a quartz-tungsten-halogen light-curing unit (XL 2500, 3M ESPE; St. Paul, USA), for 40s. The light-curing unit were connected to a voltage stabilizer (EE1500, Televolt, Sao Paulo, Brazil), and over the resin cement was placed polyester strip to avoid contact cement resin with light guide and/or ceramic and pressed flat. All procedure of resin cement specimens obtainment was carried out under controlled humidity 30% and temperature (23±1°C) conditions, according with ISO 4049 [22].

Twenty specimens of resin cement were made Knoop hardness test, 10 specimens were evaluated after polymerization (15 minutes) and remaining of specimens were evaluated after 24 hours of storage in dark bottles with distilled water at 37°C.

Resin cement specimens were prepared and tested according with following protocol: without ceramic tested immediately and after 24 hours polymerization (control group); with 0.7mm of thickness of IPS Empress and IPS Empress Esthetic immediately and after 24 hours polymerization; with 1.4mm of thickness of IPS Empress and IPS Empress Esthetic immediately and after 24 hours polymerization and after 24 hours polymerization and after 24 hours sof IPS Empress and IPS Empress Esthetic immediately and after 24 hours sof IPS Empress and IPS Empress Esthetic immediately and after 24 hours polymerization and with 2.0mm of thickness of IPS Empress and IPS Empress Esthetic immediately and after 24 hours polymerization.

#### Knoop hardness

Resin cements specimens were fixed transversally with sticky wax (Pasom, Sao Paulo, Brazil) on orificies made in acrylic resin mold (JET, Artigos Odontologicos Classico, Sao Paulo, Brazil), and were left parallel to the horizontal plane with silicon carbide sandpaper of decreasing grit (320, 400, 600 and 1200) in

automatic polisher (APL-4, Arotec Industria e Comercio Ltda., Cotia, Brazil). Knoop hardness (KHN) was measured using universal indenter tester (HMV 2, Shimadzu, Tokyo, Japan) with a load of 50g for 15s. Three indentations were performed in the depths of 100, 500 and 900 $\mu$ m on surface of resin cement specimen, resulting of 9 indentations in each specimen. Data were submitted the ANOVA in scheme of subdivided parcel represented by factor 2 x 3 x 2 (ceramic x thickness x time) with 1 additional treatment (control group) and sub parcel represented by factor depth of lecture in 3 levels. Significant differences between ceramics, thicknesses, times and depths were compared by the Tukey's test and with control group by the Dunnett's test. All statistical analyses were conducted at a significance level of p<0.05.

#### X-ray diffraction analysis

The X-ray diffraction patterns of samples were measured in a diffractometer with  $\theta$ -2 $\theta$  geometry (Rigaku Denki A-41L-Cu, Tokyo, Japan). The EMP and EST specimens were fixed in the holder of diffractometer, that emit radiation monochromatized for graphite provide of tube sealed with Cu (Cu<sub>Ka</sub> (I = 1.54187 Å). The data were collected by the scintillation detector, in the interval from 5 to 55° 2 $\theta$ , with a step size of 0.05° and a speed of count time of 1° 2 $\theta$ /min. The measurements were carried out using a current of 40 mA and 40 kV tension.

#### Scanning electron microscope

The surface of EMP and EST specimens were polished with silicon carbide sandpaper 400, 600, 1200 and 2000 grit followed with 1 and 0.05µm alumina micro polish (Arotec Industria e Comercio Ltda., Cotia, Brazil) and ultrasonic cleaning for 3min in alcohol and water. Specimens were etched using 0.2% hydrofluoric acid for 1min followed by water rinsing. Each glass ceramic etched was mounted on coded brass stub and gold coated using a sputter coater (Denton Vacuum Desk II, Bufallo, New Jersey, USA), for 100 s at 40mA. Scanning electron microscope was carried out using (JSM 5600LV, JEOL Technics, Tokyo, Japan) with an accelerating voltage of 10 or 15kV.

#### 3. Results

#### Knoop hardness

The results of the Knoop hardness of dual resin-based cements Variolink II are shown in Table 1. There was a reduction in KHN value when the ceramic glass was interposed between light guide and resin cement when compared with control group (p<0.05) in immediate time, independent of glass ceramics. In 24 hours time, all thickness of EMP specimen and EST with 2.0mm showed less KHN values significantly at control group (p<0.05). EST specimen showed higher KHN values than EMP (p<0.05), although there wasn't significant difference in thickness of 1.4mm immediate time between the glass ceramics (p>0.05). For the EMP and EST ceramics there was not significant difference in the KHN in immediate time (p>0.05). However, in 24 hours EMP and EST with 0.7 and 1.4mm showed higher KHN values in relation specimens with 2.0mm of thickness (p<0.05), with the

exception of depth 100µm, where the KHN value of EMP specimen with 2.0mm not differed significantly between the times (p>0.05); the 24 hours time showed KHN value higher significantly than immediate time (p<0.05). KHN value in depth of 100µm was higher than in 900µm in all glass ceramics and thicknesses (p<0.05). In 24 hours time, the KHN value between the depths of 100 and 900µm was similar with EMP specimen of 2.0mm and with EST specimens of 0.7 and 1.4mm (p>0.05). The EST specimens with 0.7 and 1.4mm of thickness showed KHN values similar at control group in different depths (p>0.05).

#### X-ray diffraction analysis

X-ray diffraction patterns for all the specimens analyzed are listed in Figure 1. EMP and EST specimens showed similar crystallinity (crystallites that constituted the crystal are similar) and the crystals present in glassy matrix were identified as leucite. The high half and width of peaks obtained of both glass ceramics, not revealed differences between the size of leucite crystals present in glassy matrix of EMP and EST specimens.

#### Scanning electron microscope

The results of the scanning electron photomicrographs of EMP and EST specimens are shown in Figure 2 and 3, respectively. EMP specimen showed leucite crystals distributed more heterogeneous in the glassy matrix when comparison at EST. EST specimen presented an amount greater and smaller size of leucite crystals. EMP specimen presented higher number of leucite crystal micro fracturing than EST. Leucite crystal twinning was showed both materials.

#### 4. Discussion

The null hypothesis in this study was rejected; because changes in microstructure and thickness heat-pressed glass ceramics affected the polymerization of the dual resin-based cement. The irradiance of light by means of thicker glass ceramics (2.0mm) and with heterogeneous dispersion of the crystals in the glassy matrix (EMP), may provide the attenuation of light and consequently a incompletely polymerization of resin cement.

Off all indirect methods, Knoop hardness is best predictor of conversion monomers [32] and for this reason has been used to compare the behaviour of the resin-based materials under different photo-activation conditions [4]. Typically, harder and stronger the material becomes during polymerization, greater the degree of monomer conversion [34].

When the resin cement is used for bonding etched ceramic, a complete curing is particularly important to ensure an adequate marginal seal [36] and to avoid debonding of ceramic restoration [2]. However, an adequate polymerization of resin cement is a problem beneath ceramic restoration, because the ceramic between the light source and resin cement changes the curing pattern [35]. The results showed that a satisfactory hardening of resin cement was dependent of inherent factors at ceramic material utilized to make indirect restoration.

The values obtained from EST specimens were higher than with the EMP. The ingots of IPS Empress Esthetic feature a more homogeneous microstructure, because is constituted by grains of smaller size [6]. According with Cattell [10], preferred crystal orientation and the movement of the fine leucite crystals happen

during the heat-pressing technique, which aid in uniform particle dispersion, eliminate any large glassy areas and distribute the grains in gap grading effect.

The leucite particles present in ingot of IPS Empress is grouped in cluster forms and located in large areas of the glassy phase, and in press procedure the crystals clustered disaggregate resulting in dispersion of crystals in the glass matrix [15]. However, even after heat-pressing, the IPS Empress presented agglomerated leucite crystals dispersed in glassy matrix as showed in SEM (Figure 2) and related for Guazzato *et al.* [2] and Cattell *et al.* [9]. The agglomerated leucite crystals present in IPS Empress appear to behave as larger crystals that can to avoid of light passing due the reflection, refraction and absorption [21].

The size of the particles compared to the incident light wavelength can to alter the amount of light that is absorbed, reflected and transmitted [21]. Particles similar [21] or approximately half [33] to the incident light wavelength have the greatest scattering effect and irradiance decrease [33, 21]. The size leucite crystals of EST specimens seem not promoted the light scattering, although seem be small than leucite crystals of EMP which present an average size particles of 1.8 a  $1.9\mu$ m<sup>2</sup> [9]. The result X-ray diffraction showed that not there is difference between the size grains of leucite present in EMP and EST specimens, but the decrease in size leucite crystal that is showed in SEM (Figure 3) was sufficient to improve it distribution in glassy matrix when submitted heat-pressing.

Beyond of microstructure of glass ceramics, the thickness also changed the polymerization of dual resin-based cements. After 24 hours of storage, a significant decrease of degree hardness was showed with glass ceramics of 2.0mm thickness.

Although the degree hardness of dual resin-based cement, in determined conditions, not achieve values similar at of the control group; when curing beneath EST specimens with 0.7 and 1.4mm results similar at control group were obtained after 24 hours of storage. Uctasli *et al.* [35] related that the attenuation of light by the ceramic result in lower hardness values and even the chemical component in the dual-cure cements was not sufficient to produce complete hardening. However, in this type glass ceramic the inclusion of self-curing allowed better polymerization of the dual resin-based cement, because the light transmission was high than for EMP specimen.

Independent of glass ceramic and thickness, the values of hardness were higher after 24 hours than immediately after polymerization. In according with Jung *et al.* [24], Fonseca *et al.* [18], Santos Jr *et al.* [34], Braga *et al.* [4], and Jung *et al.* [23]; the combination of light-curing with the self-curing lead the superior polymerization the dual resin-based cements. So, the delay in the obtainment of adequate curing is because the speed of chemical cure is 5-20 times lower than that of light cure [25]. The difference in speed cure between the two activation mechanisms, become the photoactivation answerable for the initial, fast hardening of the resin cement and chemical activation for the a slow, progressive hardening [1, 18]. The inefficiency of dual resin-based cements during the early stages of restoration placement, become the restoration vulnerable to loading in the first 24 hours [13].

The self-curing of dual resin-based cement also contributed for obtainment of hardness values in depth similar at control group when luting agent was polymerized beneath EST specimen with 0.7 and 1.4mm. However, when degree

of curing was analyzed between glass ceramics, it was observed that the hardness values were similar between deeper and top areas of resin cement polymerized under EST with 0.7 and 1.4mm of thickness and EMP with 2.0mm of thickness.

But, in other conditions, the decrease hardness value in deeper depth occurred because there of the characteristics of ceramic material, the light also is transmitted by mean of resin cement. The extremely small particles and dark shades as brown and yellow lead at light scattering and absorption, respectively; limited the light penetration within of resin cement and consequently it curing [11, 14]. Beside, the deeper areas of the resin cement are under-cured because light energy was partially absorbed by the top layer of the resin cement [14].

#### 5. Conclusion

Within the limitations of this study, the following conclusions were drawn:

- 1. EST specimen permitted higher curing of dual resin-based cements than EMP.
- 2. Independent of type ceramic utilized, the 2mm thickness ceramic was significant in decrease of Knoop hardness for 24 hours.
- 3. The hardness of resin cement was high after 24 hours of storage.

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Table 1 – Means (standard deviation) of value Knoop hardness (KHN) dual resin-based cement Variolink II in according with depth curing, time of storage, type and thickness of glassy ceramics.

		CERAMIC CERAMIC CERAMIC THICKNESS (mm)			nm)
DEPTH		CERAMIC -	0.7	1.4	2.0
	Control			45.00 (1.54)	
100µm	immediat	IPS Empress	40.15 (1.54)*Ab	40.05 (1.53)*Aa	39.34 (1.12)*Ab
100µm	immediat	IPS Empress Esthetic	42.19 (1.28)*Aa	41.99 (1.64)*Aa	42.02 (1.07)*Aa
	Control			51.46 (6.65)	
100µm	24 hours	IPS Empress	46.17 (3.70)*Ab	45.52 (3.79)*Ab	39.36 (1.43)*Bb
100µm	24 hours	IPS Empress Esthetic	48.81 (1.85) Aa	48.80 (4.43) Aa	46.46 (1.66)*Ba
	Control			43.88 (1.12)	
500µm	immediat	IPS Empress	38.46 (1.27)*Ab	38.88 (1.18)*Aa	37.41 (1.34)*Ab
500µm	immediat	IPS Empress Esthetic	40.97 (1.51)*Aa	40.73 (1.52)*Aa	40.04 (1.08)*Aa
	Control			51.07 (6.58)	
500µm	24 hours	IPS Empress	45.63 (3.59)*Ab	45.38 (3.01)*Ab	39.42 (1.67)*Bb
500µm	24 hours	IPS Empress Esthetic	49.54 (2.19) Aa	49.70 (4.23) Aa	45.31 (1.28)*Ba
	Control			42.56 (0.99)	
900µm	immediat	IPS Empress	36.45 (1.02)*Ab	37.77 (0.90)*Aa	35.50 (1.70)*Ab
900µm	immediat	IPS Empress Esthetic	40.44 (1.47)*Aa	39.41 (1.29)*Aa	38.89 (1.18)*Aa
	Control			49.51 (3.56)	
900µm	24 hours	IPS Empress	43.95 (3.30)*Ab	44.00 (3.19)*Ab	38.55 (1.77)*Bb
900µm	24 hours	IPS Empress Esthetic	48.11 (2.37) Aa	48.40 (4.40) Aa	44.67 (1.42)*Ba

Means followed by distinct capital letters in the same line, and small letters in the same column, were significantly different (Tukey's test, p<0.05). \*All groups were significantly different from their respective control groups (Dunnett's test, p<0.05).



Figure 1 – X-ray diffraction trace for the IPS Empress and IPS Empress Esthetic.



Figure 2 – SEM photomicrograph of etched IPS Empress.



Figure 3 – SEM photomicrograph of etched IPS Empress Esthetic.

#### **Considerações Gerais**

O objetivo dos trabalhos foi analisar a interferência dos componentes presentes nos vidros ceramizados IPS Empress (EMP), IPS Empress Esthetic (EST), IPS Empress 2 (E2) e IPS Eris para E2, dentina (D) e esmalte (E), a alteração da espessura da cerâmica na transmissão da luz e o efeito da polimerização do cimento resinoso de dupla ativação sob cerâmicas prensadas, por meio da dureza Knoop.

Na primeira parte do estudo, avaliou-se a quantidade de luz emitida e o espectro luminoso transmitido através das cerâmicas. Dois aparelhos de fotoativação foram utilizados (QTH e LED), sendo que para o QTH foram empregados dois modos de exposição: intermitente, em que a completa ventilação do aparelho foi realizada após emissão da luz, ou contínuo, em que a irradiância foi realizada sem esfriamento do equipamento. Os resultados mostraram que a quantidade de luz emitida tanto pelo QTH quanto pelo LED diminui quando transmitida por meio das cerâmicas prensadas. A dificuldade de dispersão dos cristais maiores de leucita na matriz vítrea (EMP), a presença da maior quantidade de cristais (E2) e a inclusão de determinados elementos químicos (D), tornam estes materiais cerâmicos bloqueadores da luz emitida pelos aparelhos de fotoativação. Além da microestrutura, a espessura destes materiais restauradores também reduziu a quantidade de luz transmitida através do material, pois faz com que a restauração fique ainda mais opaca interferindo mais na passagem da luz.

A análise espectral da luz transmitida pelas cerâmicas confirmou os resultados de intensidade de luz. O percentual de transmitância diminuiu quando o material cerâmico apresentou as características acima citadas. O espectro eletromagnético obtido para os vidros ceramizados também revelou que a

transmitância é maior em comprimentos de ondas maiores, pois ocorre menor dispersão da luz. Tal fato mostra que, dependendo do fotoiniciador presente na composição do cimento resinoso, o percentual de luz transmitida pelos vidros ceramizados pode dificultar o desencadeamento efetivo da reação de polimerização.

A diferença na quantidade de luz emitida pelos aparelhos de fotoativação também foi observada. O QTH, independente do modo de exposição, emitiu intensidade de luz maior que o LED quando EMP, EST e subestrutura de E2 foram os materiais interpostos. Quando o QTH foi utilizado no modo intermitente, maior intensidade de luz foi gerada em relação ao modo contínuo, mostrando que o calor gerado pelo aparelho deve ser dissipado para manter a eficiência da luz gerada. Para o LED, embora apresentando menor irradiância, o comprimento de onda emitido é semelhante ao absorvido pela canforoquinona, que é o fotoiniciador mais utilizado na Odontologia.

Na segunda parte do estudo, a eficiência da polimerização do cimento resinoso sob EMP e EST foi examinada por meio da leitura de dureza Knoop. Os resultados obtidos mostraram que o cimento resinoso fotoativado sob EST apresentou valores de dureza maiores do que quando polimerizado sob EMP. Embora, o EST e o EMP sejam vidros ceramizados reforçados com cristais de leucita, como confirmado pela análise de difração de raio-X, o tamanho do cristal gerado na matriz vítrea da pastilha do EST parece ser menor e, por esta razão, facilita sua distribuição no momento da prensagem. Este tipo de configuração da microestrutura leva a melhor transmissão da luz através do material.

Além da microestrutura do EMP e EST, a espessura destes materiais também influenciou a dureza do cimento resinoso. Uma incompleta polimerização pode ocorrer quando a restauração a ser fixada for espessa. A quantidade de luz

que atingiu o cimento resinoso quando transmitida por meio das cerâmicas com 2mm de espessura parece ter sido menor e nem mesmo a polimerização química adicional conseguiu que o cimento resinoso obtivesse um grau de polimerização semelhante às outras espessuras, sendo que somente a EST com 0,7 e 1,4 mm alcançaram valores similares ao grupo controle.

Embora as características do material cerâmico sejam responsáveis pela diminuição da transmissão da luz e, conseqüentemente, pela insatisfatória polimerização do material, também foi observado que o próprio agente de fixação pode dificultar a obtenção do grau de dureza adequado. A presença de partículas inorgânicas de tamanho muito reduzido e a adição de pigmentos tornam-se barreiras, impedindo adequada transmissão da luz dentro do material.

# Conclusão

Dentro dos limites deste estudo, concluiu-se que:

- O material cerâmico influenciou na transmitância, o qual diminuiu com o aumento da espessura da cerâmica. O modo de exposição intermitente do QTH produziu maior irradiância que o modo de exposição contínua, e ambos apresentaram maior irradiância através da cerâmica em relação o LED.
- As amostras EST permitiram maior fotoativação do cimento resinoso do que EMP.
- 3. Independente do tipo de cerâmica empregado, a espessura de 2 mm diminuiu significantemente a dureza Knoop, em 24 horas.
- 4. A dureza Knoop do cimento resinoso foi maior após armazenagem por 24 horas.

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<sup>&</sup>lt;sup>\*</sup> De acordo com a norma utilizada na FOP/Unicamp, baseada no modelo Vancouver. Abreviatura dos periódicos em conformidade com o Medline

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## ANEXOS

03-Oct-2007

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