PAULA CAROLINA DE PAIVA KOMORI

INFLUÊNCIA DO MÉTODO DE FOTOATIVAÇÃO E MATERIAIS RESTAURADORES INDIRETOS SOBRE O GRAU DE CONVERSÃO E DUREZA KNOOP DE UM CIMENTO RESINOSO DUAL

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

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A Comissão Julgadora dos trabalhos de Defesa de Tese de DOUTORADO, em sessão pública realizada em 09 de Dezembro de 2008, considerou a candidata PAULA CAROLINA DE PAIVA KOMORI aprovada.

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PROF. DR. MARIO ALEXANDRE COELHO SINHORETI

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A Estrada

Existe uma estrada

E essa estrada é a estrada que eu amo

Eu a escolhi.

Quando trilho esta estrada,

As esperanças brotam

E o sorriso se abre em meu rosto.

Dessa estrada nunca, jamais fugirei.

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RESUMO

Os cimentos resinosos têm sido amplamente utilizados devido à capacidade de união ao dente e a prótese fixa e ao sucesso estético. Os cimentos resinosos apresentam ativação química, física ou dupla ativação. Para esses cimentos, quanto maior o grau de polimerização melhores serão suas propriedades. Entretanto, em diversas situações clínicas a polimerização é afetada pela distância da ponta da unidade fotoativadora em relação ao cimento, pela presença de restaurações indiretas ou estrutura dentária. Assim, este estudo avaliou a polimerização de um cimento resinoso de dupla ativação RelyX ARC (RLX), sob diferentes condições. No primeiro estudo para simular uma restauração indireta foram confeccionados discos com 1,4mm de espessura e 8mm de diâmetro das cerâmicas Empress 2 (EMP) e Empress Esthetic (EST), Duceram Plus (DUC) e resina composta Z250 (Z250). Amostras do RLX com 0,8mm de espessura e 5mm de diâmetro, foram fotoativadas diretamente e indiretamente sob o anteparo, com os aparelhos de fotoativação com lâmpada halógena (QTH – 800mW/cm^2) e por diodo emissor de luz (LED – 1100mW/cm^2). A potência (mW) das fontes de luz foram medidas com um potenciômetro (Ophir) e o espectro da luz emitida foi registrado com espectrômetro (USB 2000), com e sem a presença do anteparo. No segundo estudo as amostras do cimento foram fotoativadas diretamente com QTH ou LED, recebendo diferentes densidades de energia (10, 20, e 30J/cm^2). O grau de polimerização do RLX foi mensurado com a porcentagem do grau de conversão pelo método FT-Raman (CD) e dureza Knoop (KHN). Os resultados obtidos CD (%) e KHN foram submetidos à Análise de Variância e teste de Tukey (5%). De acordo com os resultados destes estudos foi possível concluir que o CD e a KHN podem ser influenciadas pelo tipo de aparelho fotoativador, densidade de energia, assim como, pela presença de restaurações indiretas.

Palavras-chave: Cimento de Resina, Dureza, Grau de Conversão, Polimerização, Cerâmica, Resina Composta, Materiais Dentários.

ABSTRACT

The resin cements have been widely used because it provide link between prosthesis and tooth structure and esthetic success. Resin cements are available in chemical-cured, light-cured or dual-cured mode. For this type of cements higher polymerization reflects in improvement on properties. However in many clinical situations polymerization is affected by indirect restoration, tooth or distance of light-curing unit tip to cement. This study evaluated the polymerization of dual-cured resin cement RelyX ARC (RLX) in different simulated clinical conditions. The first study simulated indirect restoration with disks of 1.4mm in thickness and 8mm in diameter of Empress 2 (EMP), Empress Esthetic (EST), Duceram Plus (DUC) ceramics, and resin composite Z250 (Z250). Specimens of RLX with 0.8mm in thickness and 5mm in diameter were light-cured directly and indirectly through restorative materials with quartz-tungsten-halogen (QTH-800mW/cm^2) or light-emitting diode (LED - 1100mW/cm^2). The output power energy (mW) emitted by each light-curing unit was measured with the digital power meter (Ophir) and the spectral output emitted by each unit was registered using a spectrometer (USB 2000), directly and through the disks. In the second study, RLX specimens were light-cured directly with QTH or LED with 10, 20 e 30J/cm^2 energy densities. The degree of polymerization achieved by RLX was analyzed by conversion degree (CD) using a FT-Raman Spectrometer and Knoop hardness test (KHN). Data of CD (%) and Knoop hardness (KHN) were submitted to Analysis of Variance and to Tukey's test (5%). According to the results of these studies were unable to conclude that CD and KHN can be influenced by the light-curing units, light energy density and also by the presence of indirect restorations.

Key-words: Ceramics, Conversion Degree, Dental Materials, Resin Cements, Hardness, Polymerization, Resin Composite.

SUMÁRIO

INTRODUÇÃO

A Odontologia atual dispõe de diversas opções de materiais para cimentação; cimentos à base de água (fosfato de zinco, ionômero de vidro, policarboxilato de zinco) e os à base de resina (resinosos e ionômero de vidro modificado por resina) (Rosenstiel *et al.*, 1998).

Visando a eleição do melhor material para cimentação, diversos estudos comparativos avaliaram o desempenho desses cimentos e suas propriedades, tais como: biocompatibilidade, inibição de cárie, microinfiltração, tração, solubilidade, sorção de água, adesão, resistência ao desgaste, estabilidade de cor, radiopacidade, viscosidade, tempo de presa e de trabalho (Ferracane, 1985; Rosenstiel *et al.*, 1998; Rosentritt *et al.*, 2004). Entretanto, todos esses materiais apresentam algumas vantagens e desvantagens.

Os cimentos resinosos têm sido amplamente utilizados devido ao sucesso estético e a capacidade de união ao dente e a peça protética quando associados aos sistemas de união, garantindo boa adaptação marginal (Inokoshi *et al.*, 1993). São indicados para fixação de inlays, onlays, coroas totais de cerâmica pura, facetas e retentores intra-radiculares (Rosenstiel *et al.*, 1998). Os cimentos resinosos podem apresentar ativação química, física ou ainda, dupla ativação (Caughman *et al.*, 2001). Os quimicamente ativados são indicados para fixação de restaurações metálicas, os fotoativados para fixação de facetas de cerâmica e os de dupla ativação para as demais indicações, como fixação de restaurações tipo inlays e onlays de cerâmica ou resina composta e coroas de cerâmica (El-Mowafy e Rubo, 2000).

Os ativados fisicamente por luz oferecem vantagens clínicas como maior tempo de trabalho e melhor estabilidade de cor. Entretanto o seu uso tem sido considerado limitado quando envolve fixação de restaurações indiretas que atenuam a passagem de luz e dificultam a polimerização do material de fixação (Caughman *et al.*, 2001).

Devido à presença de restaurações indiretas e do substrato dentário remanescente, a fotoativação por toda extensão do agente de fixação nem sempre é possível principalmente pela atenuação da passagem de luz das unidades fotoativadoras, podendo influenciar na polimerização e conseqüentemente afetar as propriedades desses materiais (Rasetto *et al.*, 2004). Essa atenuação pode ser influenciada pela espessura, cores dos materiais restauradores e composição (El-Mowafy e Rubo, 2000; Rasetto *et al.,* 2004; Tango *et al.*, 2007a, Rueggeberg e Caughman, 1993).

Os cimentos resinosos de dupla ativação foram desenvolvidos com o propósito de combinar as propriedades desejáveis dos agentes quimicamente ativados e fotoativados (Meng *et al.*, 2006). Atualmente são os cimentos resinosos mais utilizados por apresentar polimerização adequada por toda extensão, em função dos iniciadores químicos complementar a polimerização iniciada pela luz (Peutzfeldt, 1995). Também apresentam maior tempo de trabalho quando comparado aos quimicamente ativados, estão disponíveis em diversas cores, tem rápido endurecimento a partir do momento que é fotoativado e possui polimerização tardia pela presença de catalisadores químicos (Meng *et al.*, 2006). Na composição existem aminas aromáticas e fotoinicadores como a canforoquinona para iniciar a reação de polimerização através da incidência de luz, associados ao peróxido de benzoíla e aminas terciárias responsáveis pela formação de radicais livres, independentemente da presença da luz (Middleton e Tipton, 2000; Peutzfeldt, 1995).

Diversos estudos mostram que os agentes de fixação resinosos de dupla ativação possuem melhores propriedades mecânicas quando são adequadamente fotoativados, em relação aos ativados quimicamente (El-Mowafy e Rubo, 2000; Foxton *et al.*, 2003). A energia mínima necessária para polimerização do cimento resinoso dual deveria ser incluída no rótulo dos produtos, assim como nos aparelhos fotoativadores a potência e o comprimento de onda. Entretanto, alguns fabricantes garantem que esses materiais podem polimerizar adequadamente somente pela ativação química e que o

modo de polimerização química está indicado para cimentação de restaurações onde a passagem da luz está atenuada ou impossibilitada (Lu *et al.*, 2005).

A quantidade de duplas ligações presentes nos monômeros convertidas em ligações simples para formar a cadeia polimérica durante o processo de polimerização é denominada grau de conversão (Rueggeberg e Tamareselvy, 1995). A análise do grau de conversão pode ser feita para avaliar a eficiência da polimerização através da espectroscopia Raman, que avalia de forma conservadora e rápida as alterações numa estrutura tal como, a porcentagem de conversão de um compósito (Rehman *et al.*, 1996). Alguns estudos mostraram que a variação no grau de conversão afetou diretamente as propriedades mecânicas dos agentes de fixação resinosos, como a dureza, resistência ao desgaste e à tração, solubilidade e biocompatibilidade (Peutzfeldt, 1995; Rueggeberg *et al.*, 2000). A avaliação da dureza é comumente utilizada como um método indireto para verificar o grau de conversão (Darr e Jacobsen, 1995; Ferracane, 1985).

Com a finalidade de melhorar as propriedades e o desempenho clínico dos agentes de fixação resinosos de dupla ativação é importante aperfeiçoar os métodos de fotativação. A fotoativação pode ser realizada por aparelhos de lâmpada de luz halógena emitida por filamento de tungstênio (QTH) e também, por diodo emissor de luz (LED). As QTHs emitem luz branca, que são filtradas e apenas a luz azul é selecionada para fotoativação, emitindo luz com intensidade entre 400 a 800mW/cm^2 (Rueggeberg *et al.*, 1999). Os aparelhos LEDs emitem luz azul não sendo necessário filtros para estreitar o espectro de onda emitido, são mais leves, alguns são portáteis, podem funcionar com bateria e apresentam maior tempo de vida útil quando comparados aos QTHs (Rueggeberg *et al.*, 1999). Por outro lado, estes aparelhos são modificados constantemente visando um aumento da intensidade de luz emitida e eficácia na fotoativação. Entretanto, ainda existem dúvidas quanto a sua efetividade na fotoativação (Peutzfeldt e Asmussen, 2000; Tango *et al.*, 2007b).

O processo de fotoativação depende de alguns fatores como fonte de luz, espectro de absorção do fotoiniciador, intensidade de luz emitida pelas fontes de luz e do tempo de exposição (Obici *et al.*, 2004). É possível obter valores de conversão similares utilizando-se diferentes combinações de intensidade de luz e tempo de exposição, já que o grau de conversão dos monômeros na reação de polimerização dos agentes de fixação resinosos é dependente da energia fornecida durante a fotoativação (Halvorson *et al.*, 2002). Para ativação do iniciador canforoquinona, comumente empregado nos materiais resinosos fotoativado é necessário que a luz emitida pelos aparelhos fotoativadores apresentem comprimento de onda entre 450 a 500nm e irradiância mínima por um determinado tempo de exposição (Rueggeberg *et al.*, 1999).

Assim, este estudo teve como objetivo avaliar a influência do método de fotoativação e materiais restauradores indiretos sobre o grau de conversão e dureza Knoop de um cimento resinoso de dupla ativação.

CAPÍTULO 1:

Conversion degree and hardness of dual-cured resin cement under composite resin and ceramic restorations

Artigo submetido ao periódico: Polymer Testing.

ABSTRACT

This study evaluates the conversion degree (CD) and Knoop hardness number (KHN) of RelyX ARC (RLX) under indirect restoration. Disks of Empress 2, Empress Esthetic, Duceram Plus and composite Z250 were prepared (1.4mm X 8mmØ). Rubber mold (0.8mm X 5mmØ) was used as matrix for RLX specimens. Control group was light-cured directly, with quartz-tungsten-halogen (QTH) or light-emitting diode (LED). In the experimental group, disks of esthetic restorative material were placed between RLX and the tip of QTH/LED. Light exposure time was 40 seconds. The output power energy (mW) emitted by QTH/LED was measured by a digital power meter and the spectral output with a spectrometer, directly and through the disks. The specimens were stored dry, in lightproof containers at 37°C. After 24h, the CD was analyzed by FT-Raman and, after 48h to KHN. The results obtained were submitted to 2-way ANOVA and to Tukey's test (5%). The presence of an indirect restoration had a significant influence on CD. The esthetic material used in restoration presented different effect on KHN, depending on light curing unit.

Key Words: ceramics, conversion degree, dental materials, hardness, resin cement.

INTRODUCTION

Nowadays esthetic indirect restorations has been increasingly used in dental clinic. The advantages of bonded indirect restorations include improved marginal sealing, reduced risk of postoperative sensitivity, low solubility and superior mechanical properties of resin cements when compared to zinc phosphate and glass ionomer cements [1]. Durable resin bond provides high retention, improves marginal adaptation and prevents microleakage. Adhesive bonding techniques and modern all ceramic systems offer a wide range of highly esthetic treatment options [2].

Resin cements can be classified according to their activation mode as chemical-activated, photo-activated, or dual-cured cements [3]. Dual-cured cements were developed with improved physical properties to conciliate favorable characteristics of chemical-activated and photo-activated cements [4]. These cement has extended working time and are capable of reaching a high conversion degree either in the presence or

absence of light during polymerization [5]. It is known that resin composites with high conversion degree also have good mechanical properties. For this reason, most of the research on dual-cured resin cements has focused on comparing its cure potential under different activation modes and/or photo-activation parameters [5]. Several studies have shown that dual-cured cements depend on photo-activation to achieve an optimal conversion degree [6, 7]. Hardness or conversion degree are also used to determine how the activation mode influences the mechanical properties of resin cements [8].

The composition and physical properties of esthetic indirect restorations can influence on curing of the cement, attenuating severely the light intensity emitted by the curing unit reaching the cement surface [9]. The activation of current resin based materials by blue light is well established, but a problem arises as the intensity of the activating light is reduced by passage through air, tooth and restoration [10]. Some studies demonstrated that hardness had an inverse relation with the thickness of ceramics or composites indirect restorations [11, 12].

Photoactivation light with higher intensities has potential to be used in prosthodontics. Reducing total polymerization time for luting ceramic restorations may be beneficial for the clinician and for the patient [13]. A powerful light curing unit may better cure dual-cured resin cement. The light intensity should be increased and light exposure time extended in order to improve their hardness, degree of conversion and bond strength [9]. The ultimate physical properties of dual-cured resin cement depend upon light energy delivered from the light curing unit [7].

In vitro investigations associated to controlled clinical trials should be ideal to test long term durability. The purpose of this study was to measure the conversion degree and microhardness of dual-cured resin cement under simulated indirect esthetic restoration.

MATERIAL AND METHODS

To simulate indirect restorations, disks of 1.4mm in thickness and 8mm in diameter were prepared with ceramic Empress 2 - EMP (Ivoclar-Vivadent, Schann, Liechtensein), Empress Esthetic – EST (Ivoclar-Vivadent, Schann, Liechtensein), Duceram Plus – DUC (Degussa Dental, Hanau, Germany) and resin composite Z-250 – Z250 (3M/ESPE, St. Paul, MN, USA). All specimens were prepared on shade A3 and were wet ground flat with #400, 600 and 1200 grit Silicon Carbide sandpapers (Carborundum, Saint-Gobain, Recife, Pernambuco, Brazil). Rubber molds with 5mm in diameter and 0.8mm in thickness were used as matrix for resin cement specimen obtaining - RelyX ARC – RLX (3M-ESPE, St. Paul, MN, USA), shade A3. After manipulation according to the manufacturer's instructions, the cement was inserted into the mold and was covered with a Mylar strip (Polidental Ind. and Com., São Paulo, Brazil), and gently pressed to squeeze out the excesses of material. In the experimental groups, one disk of an esthetic restorative was placed between the Mylar strip and the tip of the light curing unit. The control group was light-cured directly (DIR). Photo-activation was performed during 40 seconds only on the top surface of the specimen using a conventional quartz tungsten halogen light curing unit Degulux – QTH (Degussa Dental, Hanau, Germany), with intensity of 800mW/cm^2 and; with a light-emitting diode Smartlite PS – LED (Dentsply, Konstanz, Germany), with intensity of 1100mW/cm^2. Light intensity was measured before light curing; the output power (mW) emitted by each light curing unit was measured with a digital power meter (Ophir Optronics Inc., Danvers, MA, USA), and the diameter of the light guide tip (cm) with a digital caliper (Mitutoyo Tokyo, Japan). The irradiance (mW/cm^2) was computed as the ratio of the output power by the area of the light guide. The light spectral distribution emitted by each LCU was obtained using a computer controlled spectrometer (USB 2000, Ocean Optics, FL, USA). The specimens (n=5) were stored dry, in lightproof containers at 37°C. After 24h, for the degree of conversion measurements, the top surface of each specimen was exposed to FT-Raman. After 48h, these same specimens were submitted to Knoop hardness test.

Conversion degree (CD)

The CD was analyzed by FT-Raman. Spectra of the uncured and cured resins were obtained using a FT-Raman Spectrometer (RFS 100/S – Bruker Inc., Karlsruhe, Germany). To excite the spectra, the defocused λ 1064.1nm line of a Nd:YAG laser source was used. The maximum laser power incident on the sample surface was about 200mW and the spectrum resolution was 4^{cm-1}. The aluminum mold with the uncured resin was positioned in the sample compartment and the sample stage was mounted on an optical rail. The FT-Raman spectra of the uncured resin were obtained using 200 scans without removing the resin of the aluminum rods. For each specimen surface, three spectra were acquired in three distinct points, obtaining 150 spectra in the total. The FT-Raman spectra were analyzed by selecting a spectra region from 1590 to 1660^{cm-1}. The Raman vibrational stretching mode in 1609 and 1638^{cm-1} were fitted by Lorentzian shapes to obtain the height of the peaks using the Microcal Origin® software. To calculate CD, the height ratio of the peaks at 1609^{cm-1} and 1638^{cm-1} were used in the Equation 1. The mean value and standard deviation were calculated for each series.

Equation 1:

DC (%) = 100*[1 – R cured / R uncured]

where R = band height at 1638^{cm-1}/band height at 1609^{cm-1}

The FT-Raman results were obtained for each specimen and the values were submitted to 2-way ANOVA and to Tukey's test (α=0.05).

Knoop Hardness Number (KHN)

After 48h, the specimens were submitted to Knoop hardness test. The specimens were longitudinally sectioned in two equal parts under water cooling with diamond saw (Extec model 12205, Extec corp., Enfield, USA). The exposed surfaces were sequentially polished under water cooling with #400, 600 and 1200 grit Silicon Carbide

sandpapers for 15s, 30s and 60s, respectively, in a universal polishing machine model APL-4 (Arotec, Cotia, Brazil).

Microhardness measurements were performed in a Microhardness Tester model HMV-2 Shimadzu (Shimadzu, Tokyo, Japan). Three sequences of 3 indentations each (50g during 15s) were performed to obtain 3 hardness values for each depth: 50μm, 400μm and 750μm.

A hardness mean value was obtained for each specimen and the values were submitted to 2-way ANOVA and to Tukey's test (α=0.05).

RESULTS

Table 1 presents the 2-way ANOVA results for CD (%).There was no statistical significant difference for light curing unit (p=0.06) and for the interaction "light curing unit" X "indirect restoration" (p=0.72). There was a statistical significant difference for indirect restorations (p=0.00073).

On Table 2, it can be verified a reduction on CD value when ceramic (EMP and EST) were interposed between light curing tip and resin cement, compared to control group - directly exposed, independent of the light unit ($p<0.05$).

Table 3 presents results of 2-way ANOVA for KHN. There were significant differences for light curing unit (p=0.02), indirect restoration (p=0.06) and for the interaction "curing unit" X "indirect restoration" (p=0.00001).

According to Tukey´s test (Table 4), there was a significant decrease of resin cement KHN in EST group light-cured with QTH. With LED the highest KHN was obtained with EST, followed by EMP, Z250, DIR and DUC. Resin cement KHN was higher on EST group light-cured with LED compared to QTH, but for DUC and cirectly group the higher KHN values were obtained for QTH. For Z250 and EMP both light curing unit obtained similar results.

Table 5 presents the irradiance (mW/cm^2), which was computed as the ratio of the light curing unit output power by the area of the light guide tip. The light exposure trough indirect materials decrease the irradiance for both light curing unit, QTH and LED. For QTH the higher value was 808.52 obtained for direct group, followed for DUC=392.04, EST=391.52, EMP=374.36 and Z250=353.04. For LED all values obtained were higher compared to QTH the direct group=1103.10, EMP=662.42, DUC=621.01, EST=589.17 and Z250=550.05.

Figure 1 presents the spectral output of QTH and Figure 2 of LED, for direct transmittance (DIR) and through restorative materials (DUC, EMP, EST and Z250). The light spectral distribution emitted by each light curing unit was obtained using a computer controlled Spectrometer (USB 2000, Ocean Optics, FL, USA). Data from irradiance and spectral analyzes were tabulated in an appropriate software (Origin 6.1, OriginLab Corp., MA,USA) to obtain, by numerical integration, the specific irradiance of the light spectrum in the regions between 360-540nm.

According to figure 1 and 2, the light transmission depends on light curing unit and the indirect materials. The spectral irradiance provided by the light curing unit trough the indirect materials show significant reduction on irradiance. For QTH this reduction was followed by peak deviation.

Figure 1 shows the spectral distribution for Degulux - QTH lies between 380 and 520nm with intensity (mW/cm2): direct=808.52, DUC=621.01, EMP=662.42, EST= 589.17 and Z250=550.95.

Figure 2 shows the spectral distribution for Smartlite PS – LED lies between 420 and 520nm with light intensity (mW/cm2): direct=1103.10, DUC=392.04, EMP=374.36, EST=391.52 and Z250=353.04.

DISCUSSION

In this study a thin layer of dual-cured resin cement simulated clinical conditions involving cementation of different thicknesses of indirect restorations.

Dual-cured resin cements depends on photo-activation to achieve higher hardness values [11]. It is known that the light intensity reaching the cement is drastically reduced when light is transmitted through a resin composite or ceramic restoration [14]. The CD obtained by the cement when used under indirect restorations was lower than the control group, which was exposed directly to the light. All the indirect restoration attenuated the light from the light curing unit.

The degree of light attenuation is primarily dependent on the type, composition and thickness of the restorative material [11, 15]. The attenuation may be caused by the light absorbing characteristics of the restorative material [16]. The incident light must be accounted for by reflection, absorption or transmission [17].

Light intensity, time of exposure, direction and location of light, and light transmission characteristic of ceramic restorative materials are other factors affecting polymerization [18]. In this study the thickness and the color shade of the specimens were the same. The output energy of QTH (800mW/cm^2) when measured through a 1.4mm-thick Z250 disk-shaped specimen decreases to 350mW/cm^2 and for LED, from 1100mW/cm^2 to 550mW/cm^2. For composite resins, a light intensity of more than 400 mW/cm^2 is generally recommended [19]. The minor light intensity used in this study was 350mW/cm^2, which seem to be adequate to obtain high CD values and satisfactory KHN. It can also be supposed that this intensity could be lower for dual-cured resin cements.

Increasing crystalline content in ceramic composition to achieve greater mechanical strength generally result in greater opacity. The Empress 2 ceramic system derives its strength from hot-pressing a lithium disilicate glass ceramic framework which is veneered with a fluoride apatite ceramic [15]. The lower crystalline content and the refractive index of the lithium disilicate crystals makes it more translucent and capable of

transmitting light, what could provide the resin cement higher degree of polymerization [15, 20]. Translucent materials are more capable of transmitting light and provide higher CD values [21]. EST is a ceramic based in leucite with smaller crystals distributedin a more homogeneous mode than EMP [22] Duceram is a completely amorphous hydrothermal ceramic defined as a glass, with no crystalline phase [2], but similar values of CD and KHN were obtained with this material compared to other materials.

These conflicting results may be explained by the fact that the transmittance of light through indirect restorations is wavelength dependent. Some previous studies showed that longer wavelengths penetrate composites deeper than shorter wavelengths [23]. It is possible that high percentage of shorter wavelengths were absorbed by ceramic and result in incomplete polymerization of resin cement [18]. The photoinitiator is excited in the presence of light with an adequate wavelength and sufficient irradiance [24] [25]. In order to obtained optimal polymerization is necessary that adequate light energy reaches the luting agent [26].

A previous study that measured the intensity of light curing unit demonstrated that the CD of the resin cements decreased with a decreasing of the light intensity [21]. In this study the light intensity output of the LED unit (1100mW/cm^2) was higher than QTH unit (800mW/cm^2). The light energy density is the product between light intensity X time of exposure (40s), for LED = 44J/cm^2 and QTH = 32J/cm^2. High light intensity curing and longer polymerization times may affect the CD and KHN. The hardness test can be used as an indirect assessment of degree of conversion, because the mechanical properties of a polymer depend upon the cross-linking density and the quality of the network formed during polymerization [8]. The filler content of the composite is also responsible for its hardness [27]. Previous studies indicated that hardness was closely correlated to small change in degree of conversion, which lead to a large change in hardness [8, 28].

Most dual-cured resin cements still require photo-activation and demonstrated lower hardness values when this procedure was omitted (Hasegawa, 1991;

[11]. The ultimate hardness of dual-cured resin cements depend on the amount of time exposure to the curing light [6].

Hardness of the resin cement has an inverse relation with the thickness of ceramics or composites [9]. When dual-cured resin cement is light-cured directly or through ceramic or composite, it always achieve higher hardness value and conversion degree than chemical-cured solely [9]. The hardness increased immediately after light curing followed by a steady increase in hardness over the next 24h [10]. The reaction kinetics of dual-cured resin cements through ceramic by chemical or light activation, showed that the cure speed during light exposure was 5 to 20 times faster than by chemical curing solely [29]. Chemically induced continuation of polymerization following light curing is hard to achieve [9]. The initial light exposure causes a rapid increase in viscosity, which hinders the migration of active radical components that are responsible for further chemically induced polymerization. When light intensity is weak, a small extent of chemical curing can occur but cross-linking reaction continues even after light curing has ended [9].

Some factors have to be taken into account when selecting resin cement, such as the manipulation characteristics, esthetic demand of oral rehabilitation and the stress level the bonding interfaces will be submitted to, the conversion degree and hardness. In this study the indirect restorative material was 1.4mm in thickness and affected de CD and KHN. Previous studies suggested a minimum of 60s of light exposure for adequate polymerization through indirect restorative materials (composite/ceramic) with less than 2mm in thickness [15, 16, 21].

To compensate the decreased of light intensity longer irradiation times are recommended. The light exposure time has to be extended in order to improve the hardness, degree of conversion and other properties of resin cements [16]. The light exposure periods recommended by the manufacturers were not adequate to provide complete polymerization and to compensate the attenuation of light by the restorative

material [6, 30]. Longer light curing time would be required as the thickness of the resin composite increases, and when it exceeds the limit where the curing light could reach.

CONCLUSION

Within the limitations of this in vitro study, it was concluded that the presence of an indirect restoration had a significant influence decreasing the CD. The esthetic material used in indirect restoration presented different effect on KHN, depending on the light curing unit.

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FIGURES

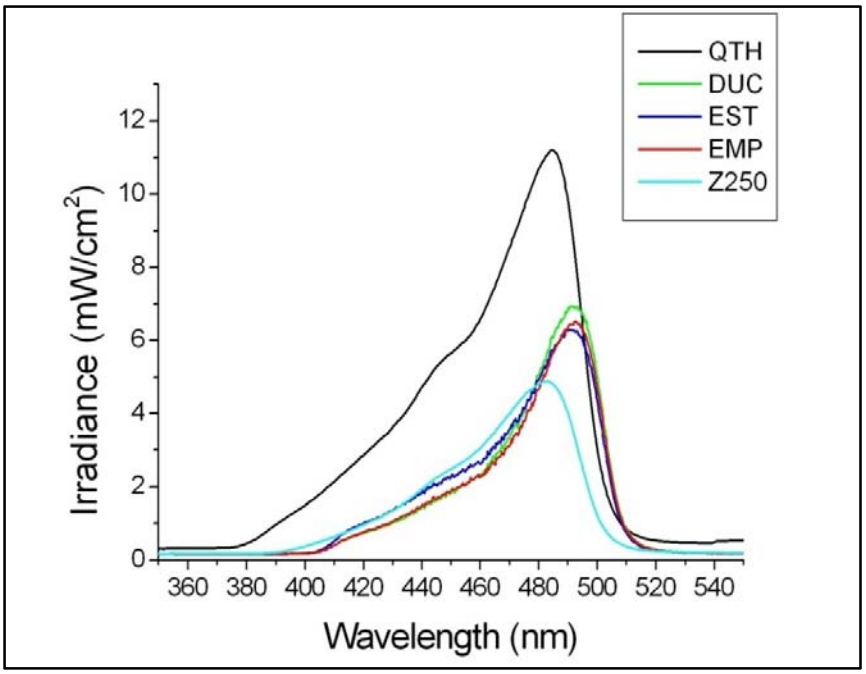

Figure 1: Spectral output-QTH

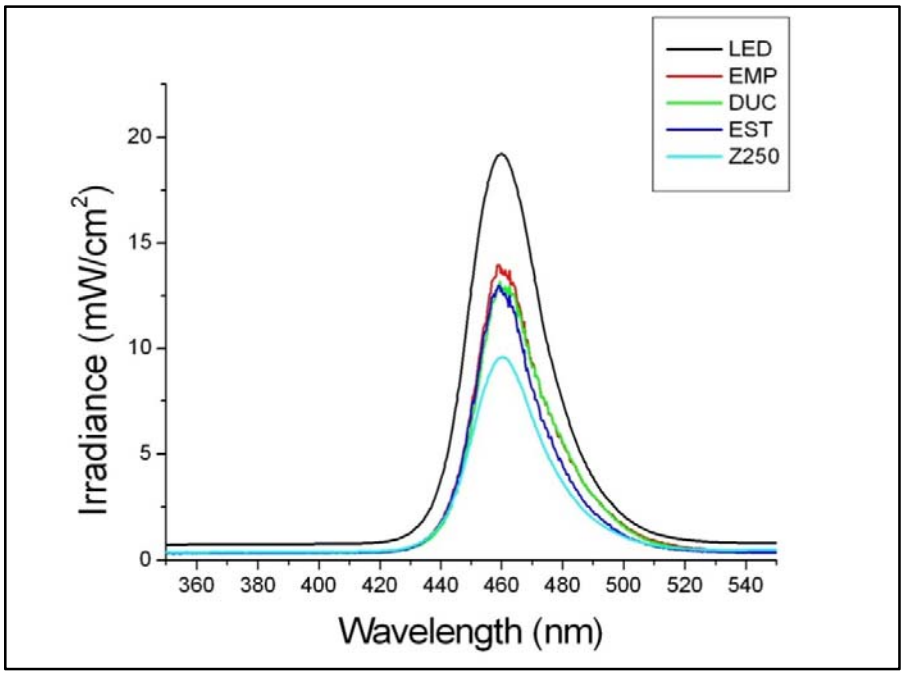

Figure 2: Spectral output-LED

TABLES

Table 1: Result of 2-way ANOVA (dependent variable: CD)

Source of variation	Df	Mean square	F	P value
Curing unit	1	8.54	3.38	0.06
Indirect restoration	4	15.86	6.29	0.00073*
A X B	4	1.31	0.52	0.72
Error	40	2.52		
Total	49	178.06		

*Significant diference

Table 2: Mean values and standard deviations of CD (%) for RLX in according with type of indirect restoration

INDIRECT RESTORATION	Direct	DUC	EMP	EST	Z250
QTH	84.19(1.01)a	82.61(0.73)ab	81.29(3.41)b	81.73(0.98)b	82.61(0.48)ab
LED	85.19(1.24)a	82.81ab(1.65)ab	82.00b(2.08)b	82.07(1.09)b	82.72(0.97)ab

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

Table 3: Result of 2-way ANOVA (dependent variable: KHN)

Source of variation	Df	Mean square	F	P value
Curing unit	1	43.54	5.4	0.02*
Indirect restoration	4	18.85	2.36	0.06
A X B	4	200.58	25.18	0.00001*
Error	40	7.96		
Total	49	1239.94		

*Significant diference

Table 4: Mean values and standard deviations of KHN for RLX in according with type of indirect restoration

Indirect Restoration	Direct	DUC	EMP	EST	Z250
QTH	50.28(3.55)a/A	46.95(2.39)a/A	46.81(2.66)a/A	37.69(0.85)b/B	45.77(2.06)a/A
LED	41.41(1.88)bc/B	38.12(2.19)c/B	44.87(1.84)b/A	50.75(5.43)a/A	43.01(2.81)bc/A

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

Table 5: Mean values for irradiance (mW/cm^2):

INDIRECT RESTORATION	Direct	DUC	EMP	EST	Z250
QTH	808.52	392.04	374.36	391.52	353.04
LED	1103.10	621.01	662.42	589.17	550.95

CAPÍTULO 2:

Effect of light energy density on conversion degree and hardness of dual-cured resin cement

Artigo submetido ao periódico: The International Journal of Prosthodontics.

ABSTRACT

The aim of this study was to evaluate the effect of different light curing units (LCU) and light energy densities on conversion degree (CD) and Knoop hardness number (KHN) of RelyX ARC (RLX) resin cement. After manipulation according to the manufacturer's instructions, RLX was inserted into a rubber mold (0.8mm X 5mm) and covered with a mylar strip. The tip of LCU was positioned in contact with the mylar surface. Quartz-tungsten-halogen (QTH) and light-emitting diode (LED) LCUs with light densities of 10, 20 and 30J/cm^2 were used to light-cure the specimens. After light curing, the specimens were stored dry, in lightproof containers at 37°C. After 24 h, CD was analyzed by FT-Raman and after additional 24h samples were submitted to KHN. Data of CD (%) and KHN were submitted to 2-way ANOVA and to Tukey's test (α=0.05). QTH and LED were effective light unit curing. For QTH, there were no differences among light energy densities for CD and KHN. For LED, there was a significant reduction of CD with light energy density set in 10J/cm^2. KHN was not influenced by the light-curing unit as well as by its light energy density.

Key Words: conversion degree, light curing, hardness, resin cement.

INTRODUCTION

Resin luting agent became popular by its high strength, low solubility and esthetic appearance (1). Nowadays, an increasing number of products are available on the market. The majority of resin luting materials employ dual-cured polymerization, which

combines desirable properties of both chemical-cured and light-cured cements – such as wide range of color shades, rapid initial hardening and further curing by the chemical catalyst (2), and extended working time providing the operator more control over the setting of the material than with chemical-cured system solely (3). The dual-cured resin luting agents have both initiation systems, on a 2-paste system, the base paste usually containing camphorquinone, both aliphatic amine and aromatic tertiary amine, and the catalyst paste containing benzoyl peroxide (4).

Several studies investigated the effect of polymerization mode on the degree of conversion, mechanical properties or on bond strength of resin luting agents (4-6). The manufacturers of dual-cured resin cements aim that these materials can be effectively polymerized by the chemical reaction solely, and also that chemical curing is recommended for cementation of restorations where light penetration is compromised (4). However, the majority of dual-cured resin cement do not reach similar conversion degree or hardness values when tested in the presence or absence of light activation (4).

The activation of current resin based materials by blue light is well established, but a problem arises as the intensity of the activating light is reduced by passage through air, tooth and restoration (7). The most commonly used light curing units are quartz tungsten halogen light curing units, which emit a wide spectrum of wavelengths (8, 9). These types of light sources usually operate at light intensities between 400 and 800mW/cm^2 (10). Newly developed light curing units operate at relatively high intensity

and are purposed to provide optimum properties to composites in a shorter exposure time (9).

The purpose of this study was that the conversion degree and hardness of dual-cured resin cement light activated is dependent of the light energy densities and the light curing unit.

MATERIAL AND METHODS

Specimen Preparation

The dual-cured resin cement used in the specimen preparation was RelyX ARC – RLX (3M-ESPE, St. Paul, MN, USA). Rubber molds with 5mm in diameter and 0.8mm in thickness were used as matrix for resin cement specimens. After manipulation according to the manufacturer's instructions, the cement was inserted into the mold and was covered with a mylar strip (Polidental Ind. and Com., São Paulo, Brazil). The tip of light unit was positioned in contact with the mylar surface during light curing with a conventional quartz-tungsten-halogen light curing unit Degulux - QTH (Degussa Dental, Hanau, Germany) with light intensity of 800mW/cm^2 and with a light emitting diode Smartlite PS – LED (Dentsply, Konstanz, Germany) with light intensity of 1100mW/cm^2. The output power energy (mW) emitted by each light curing unit was measured with a digital power meter (Ophir Optronics Inc., Danvers, MA, USA), and the diameter of the light guide tip (cm) with a digital caliper (Mitutoyo Tokyo, Japan). The irradiance (mW/cm^2) was computed as the ratio of the output power by the area of the light guide tip. Light

densities of 10, 20 and 30J/cm^2 were used to light-cure the specimens. After light curing, the specimens (n=5) were stored dry, in lightproof containers at 37°C. After 24h, the top surface of each specimen was submitted to conversion degree analysis by FT-Raman. After additional 24h, these same specimens were submitted to Knoop hardness test.

Conversion degree (CD)

The conversion degree was analyzed 24h after light curing. Spectra of uncured and cured resins were obtained using a FT-Raman Spectrometer (RFS 100/S – Bruker Inc., Karlsruhe, Germany). To excite the spectra, the defocused λ 1064.1 nm line of a Nd:YAG laser source was used. The maximum laser power incident on the sample surface was about 200mW and the spectrum resolution was 4^{cm-1}. The aluminum mold with uncured resin was positioned in the sample compartment and the sample stage was mounted on an optical rail. The FT-Raman spectra of the uncured resin were obtained using 200 scans without removing the resin of the aluminum rods. For each specimen surface, three spectra were acquired in three distinct points, obtaining 90 spectra in the total. The FT-Raman spectra were analyzed by selecting a spectra region from 1590 to 1660^{cm-1}. The Raman vibration stretching mode in 1609 and 1638^{cm-1} were fitted by Lorentzian shapes to obtain the height of the peaks using the Microcal Origin® software. To calculate the conversion degree, the height ratio of the peaks at 1609 and 1638^{cm-1} were used in the Equation 1. The mean value and standard deviation were calculated for each series.

Equation 1:

DC (%) = 100*[1 – R cured / R uncured]

where R = band height at 1638^{cm-1}/band height at 1609^{cm-1}

The FT-Raman results were obtained for each specimen and the values were submitted to 2-way ANOVA and to Tukey's test (α=0.05).

Knoop Hardness Number (KHN)

After 48h of light-curing, specimens were longitudinally sectioned in two equal parts under water cooling with diamond saw (Extec model 12205, Extec corp., Enfield, U.S.A.). The exposed surfaces were sequentially polished under water cooling with #400, 600 and 1200 grit Silicon Carbide sandpapers (Carborundum, Saint-Gobain, Recife, Pernambuco, Brazil),for 15s, 30s and 60s, respectively, in a universal polishing machine model APL-4 (Arotec, Cotia, Brazil).

Microhardness measurements were performed in a Microhardness Tester model HMV-2 Shimadzu (Shimadzu, Tokyo, Japan). Three sequences of 3 indentations each (50g during 15s) were performed to obtain 3 hardness values for each depth: 50µm, 400µm and 750µm.

A hardness mean value was obtained for each specimen and the values were submitted to 2- way ANOVA and to Tukey's (α=0.05).

RESULTS

Two-way ANOVA for CD is presented on Table 1 and shows statistical significance for light energy density (p=0.01) and for the interaction “density” X “curing unit” (p=0.003). There was no statistical significant difference for light curing unit (p=0.67).

For comparisons among groups, data of CD was submitted to Tukey´s test (Table 2). For QTH there was no significant difference among different light energy densities, but for LED there was a significant decrease in CD with densities of 10J/cm^2 ($p<0.05$).

Two-way ANOVA for KHN is presented on Table 3, which showed significance for curing unit (p=0.008) and for interaction “density” X “curing unit” (p=0.003). There was no statistical significant difference in density (p=0.12).

On Table 4, it can be verified the comparison among groups. Both light curing unit (QTH/LED) presented statistical similar KHN mean values with different light energy densities.

DISCUSSION

Several studies measured the intensity of light curing units, and demonstrated that the degree of polymerization of resin cements decreased with the decreasing of light intensity (4, 5). For composite resins, a light intensity up to 400mW/cm^2 is generally recommended (11)

The result of this study shows that QTH provides the resin cement the same values of CD and KHN when different light energy densities are compared. Cross-linking reaction and polymerization continue even after light-curing has been finished (2). The light energy density promoted by this kind of light curing unit has ability to activated polymerization and achieve reliable CD and KHN values. Under low light intensity irradiation, dual-cured resin cement still has a large amount of free radicals, mostly from chemical catalysts trapped in the hardening resin matrix. Although these trapped free radicals did not contribute significantly to overall DC, they could improve the cross-linking density of dual-cured resin luting agents through combining with a double link of methacrylate groups in local resin matrix to finish termination (12).

LED presented a significant reduction on CD with 10J/cm^2. It could result of high light intensity (1100mW/cm^2) combined to short exposure time of 9s. High intensity may produce more starter radicals and shorter polymers chains than low intensity light curing unit (13, 14). Some dual-cured resin cements exposed to initial light-curing polymerize fast, resulting in a viscous gel. The rapid increase in viscosity may hinder the migration of active radical components responsible for further chemically induced polymerization (6).

RLX received total of 11J/cm^2, but several studies showed at least 12J/cm^2 is necessary for light curable resin composite based materials achieve reliable CD and KHN values (15). The CD mean value was 81.10%, previous studies verified similar values of conversion degree in resin composites samples with different hardness values, which

were attributed to differences in the polymer cross-linking density (16). The ultimate hardness value of dual-cured resin cements depend on the amount of time exposure to the curing light (5).

Most dual-cured resin cements still require photo-activation and demonstrated inferior hardness when light activation was omitted (17). With a high-power light unit, more photons are available per second for absorption (18), more photoinitiator reacts with amine, and more free radicals are available for polymerization (19). This photoinitiator is excited in the presence of light with an adequate wavelength and sufficient irradiance (20). A rapid polymerization may also result in the formation of high cross-linked short polymers chains (21).

The result of this study shows that all light energy densities achieve high CD and KHN values. Additionally, the light curing unit tip was directly in contact with the resin cement. In dental practice, it only happens with the cement margin of the restoration. So many other factors can modify this result, such as: restoration shade, thickness, and composition for indirect restoration. Further research and controlled clinical trials are needed before clinical protocols recommendations can be given.

CONCLUSIONS

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

1) QTH and LED were effective light unit curing on polymerization;
2) LED provide a significant reduction on CD with light energy density of 10J/cm^2;
3) KHN was not influenced by the light-curing unit as well as by its light energy density.

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ILLUSTRATIONS AND TABLES

Table 1: Result of 2-way ANOVA (dependent variable: CD)

Source of variation	Df	Mean square	F	P value
Curing unit	1	0.37	0.17	0.67
Density	3	7.9	3.8	0.01*
A X B	3	11.63	5.54	0.003*
Error	32	2.09		
Total	39	126.32		

*Statistical significant difference

Table 2: Mean values and standard deviations of conversion degree (%) for RLX in according to light energy density and time of exposure [seconds].

DENSITY	10J/cm^2 [sec]	20J/cm^2 [sec]	30J/cm^2 [sec]
QTH	82.97(1.04)a/A [12s]	83.88(2.82)a/A [25s]	84.19(1.01)a/A [37s]
LED	81.10(1.32)b/A [9s]	83.60(1.35)a/A [18s]	83.89(0.97)a/A [27s]

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

Table 3: Result of 2-way ANOVA (dependent variable: KHN)

Source of variation	Df	Mean square	F	P value
Curing unit	1	94.27	7.70	0.008*
Density	3	25.29	2.06	0.12
A X B	3	71.01	5.80	0.003*
Error	32	12.23		
Total	39	774.76		

*Statistical significant difference

Table 4: Mean values and standard deviations of KHN for RLX in according to light energy density and time of exposure [seconds].

DENSITY	10J/cm^2 [sec]	20J/cm^2 [sec]	30J/cm^2[sec]
QTH	49.75(4.23)a/A [12s]	48.62(2.81)a/A [25s]	50.28(3.55)a/A [37s]
LED	49.52(3.10)a/A [9s]	50.89(2.05)a/A [18s]	45.87(4.77)a/A [27s]

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

CONSIDERAÇÕES GERAIS

Este estudo avaliou a eficiência da polimerização de um agente de fixação resinoso de dupla ativação comparando dois aparelhos de fotoativação Degulux - QTH e Smartlite PS - LED.

Alguns fabricantes indicam a utilização de cimentos resinosos de dupla ativação para fixação de restaurações indiretas em compósitos ou cerâmicos, por compensarem a atenuação da luz de fotoativação com a polimerização química mesmo em regiões onde o acesso da luz é limitado (Hofmann *et al.*, 2001). Neste estudo, foi observado que a dupla ativação não compensou a atenuação da luz que passa através dos materiais restauradores e promoveu menor grau de conversão e dureza Knoop. Diversos estudos obtiveram resultados similares e mostram que os agentes de fixação resinosos de dupla ativação apresentam melhor desempenho quando são fotoativados corretamente e por toda a sua extensão (Blackman *et al.*, 1990; Caughman *et al.*, 2001; Hasegawa *et al.*, 1991).

A intensidade da luz emitida pelos aparelhos fotoativadores é reduzida quando da passagem através de restaurações indiretas. Adicionalmente, a intensidade da luz diminui exponencialmente em função da espessura do material restaurador (Prinsloo *et al.*, 1997).

Como a densidade de energia (J/cm^2) é o produto da intensidade de luz (mW/cm^2) pelo tempo de exposição à luz, a atenuação poderia ser compensada pelo aumento do tempo de exposição (Rueggeberg e Caughman, 1993). Assim, tanto o grau de conversão como a dureza dos agentes de fixação resinosos de dupla ativação são dependentes do tempo de exposição à luz fotoativadora (Braga *et al.*, 2002;Hasegawa& Boyer e Chan, 1991).

A fotoativação indireta do cimento resinoso de dupla ativação deve ser tratada com cautela e alguns aspectos levados em consideração. Com o aumento da espessura da

restauração indireta, a dispersão da luz pode aumentar e absorção da luz reduzir ainda mais a quantidade de energia fornecida ao cimento (Watts, 2005). Pode-se recomendar, deste modo, que o tempo de fotoativação indicado pelo fabricante para o cimento deve ser analisado de forma adequada (Blackman *et al.*, 1990).

As amostras do cimento resinoso de dupla ativação foram fotoativados com diferentes aparelhos fotoativadores (Degulux - QTH e Smartlite PS – LED) e densidades de energia (10, 20 e 30J/cm^2). As amostras fotoativadas com o aparelho QTH apresentaram resultados similares em todas as condições de densidade de energia analisadas. Entretanto, com o aparelho LED foram verificados valores menores do grau de conversão e da dureza Knoop nas amostras fotoativadas com densidade de energia de 10J/cm^2.

Os aparelhos LEDs de alta potência emitem luz com intensidade superior a 1000mW/cm^2. Devido à alta intensidade, maior quantidade de fótons estará disponível, para ativar maior quantidade de moléculas do fotoiniciador (canforoquinona), formando radicais livres para reação de polimerização o que resulta em polimerização rápida com formação de polímeros com ligações cruzadas curtas e que podem dificultar a polimerização química tardia (Nalcaci *et al.*, 2005). Unidades fotoativadoras de alta potência podem ser usadas com a finalidade de promoverem polimerização adequada, em maior profundidade, obtendo-se melhores propriedades mecânicas.

O agente de fixação ideal deve ter propriedades mecânicas suficientes para resistir às forças mastigatórias, resistir à degradação do ambiente bucal e unir-se à estrutura do dente remanescente (Rosenstiel *et al.*, 1998). Para seleção de um agente de fixação diversas propriedades mecânicas devem ser avaliadas. Atualmente não existe um cimento ideal que possa ser indicado para todas as situações clínicas de cimentação.

A polimerização parcial dos agentes de fixação resulta na redução das propriedades físicas e do desempenho desses materiais (Blackman *et al.*, 1990). Embora exista relação direta entre o grau de conversão e as propriedades físicas dos agentes de

fixação, pequena redução nos valores do grau conversão pode não promover efeitos deletérios no resultado final das cimentações de restaurações indiretas (Caughman *et al.*, 2001).

Não foi comprovada correlação adequada entre o grau de conversão e a dureza Knoop, confirmando os estudos de Braga *et al*. (2002) e Chung e Greener (1990). Portanto, a medida de dureza pode não corresponder aos valores de conversão monomérica do material (Ferracane, 1985). Alguns estudos têm mostrado que o ensaio de dureza pode detectar qualquer variação na quantidade de ligações cruzadas e às áreas com menor conversão monomérica (Ferracane, 1985; Rueggeberg e Craig, 1988).

A medida do grau de conversão pelo método vibracional de espectroscopia FT-Raman, é sensível a vibração das moléculas C=C dos metacrilatos (Shin *et al.*, 1993). A média do grau de conversão para o agente de fixação resinoso RLX encontrado neste estudo (80%) foi superior às resinas compostas, que normalmente alcançam de 50-60% de conversão (Rueggeberg *et al.*, 1993). Entretanto, esses valores estão de acordo com outro estudo que avaliaram os agentes de fixação resinosos e podem estar relacionados à baixa viscosidade inicial desses materiais que permite maior difusão dos radicais livres durante a polimerização e conseqüentemente aumenta o grau de conversão (Caughman *et al.*, 2001).

Assim este estudo avaliou a dureza e o grau de conversão de um agente de fixação resinoso de dupla ativação sob algumas situações passíveis de ocorrer clinicamente. Novas pesquisas que avaliem outras propriedades são necessárias para predizer o desempenho clínico das restaurações indiretas.

CONCLUSÃO

Frente à metodologia empregada neste estudo e de acordo com os resultados obtidos, foi possível concluir que:

1. A polimerização do cimento resinoso foi influenciada pelos métodos de fotoativações, tipos de unidades fotoativadoras e material utilizado na confecção das restaurações indiretas.

2. As restaurações estéticas indiretas atenuaram a passagem de luz, reduzindo o grau de conversão e dureza do cimento resinoso dependendo do material utilizado e da unidade fotoativadora.

3. O grau de conversão do cimento resinoso foi similar quando foram comparados os aparelhos fotoativadores Degulux Soft-start e Smartlite PS.

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