AMÉRICO BORTOLAZZO CORRER

AVALIAÇÃO DA DUREZA KNOOP DE COMPÓSITOS RESTAURADORES ODONTOLÓGICOS FOTOATIVADOS POR DIFERENTES MÉTODOS

Dissertação apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para a obtenção do Título de Mestre em Materiais Dentários.

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“Bom mesmo é ir à luta com determinação, abraçar a vida e viver com paixão, perder com classe e vencer com ousadia, pois o triunfo pertence a quem se atreve... E a vida é muito para ser insignificante.”

(Charles Chaplin)

SUMÁRIO

RESUMO

Os compósitos odontológicos fotoativados são os materiais odontológicos restauradores estéticos mais utilizados atualmente. Dentre suas vantagens estão o controle do tempo de presa e a estética, além de ser desnecessário a confecção de preparo cavitário retentivo. Durante o processo de polimerização, o compósito sofre contração e, dependendo da velocidade da reação, essa contração pode gerar grande tensão na interface dente-restauração, promovendo o aparecimento de fendas e conseqüente falha da restauração. Além disso, para que o compósito apresente propriedades mecânicas adequadas, é necessário que ele atinja alto grau de conversão. O grau de conversão depende da quantidade de energia que é fornecida ao compósito, embora o efeito da densidade energética nas propriedades mecânicas dos compósitos ainda não seja bem evidenciado. Visando o melhor entendimento dos métodos de fotoativação sobre as propriedades finais dos compósitos restauradores odontológicos, este estudo foi dividido em dois trabalhos: O primeiro avaliou o efeito do aumento da densidade energética pelo maior tempo de exposição, mantendo-se a mesma intensidade de luz, sobre a dureza Knoop dos compósitos Z250 e Esthet-X, fotoativados por luz de lâmpada halógena (QTH) - XL2500, luz emitida por diodo (LED) – Ultrablue Is ou arco de plasma de Xenônio (PAC) – Apollo 95E. Os resultados mostraram que o aumento da densidade energética produziu maiores valores de dureza Knoop quando utilizado LED ou PAC. Para QTH não houve diferença significativa. Em relação às fontes de luz, o PAC apresentou menores valores de dureza Knoop quando comparado ao LED e QTH, que não diferiram entre si. Menores valores de dureza Knoop foram encontrados em maiores profundidades dos compósitos. O segundo trabalho verificou a influência da relação intensidade de luz *versus* tempo de exposição, utilizando a mesma densidade energética, sobre a dureza Knoop dos compósitos Z250 e Esthet-X, fotoativados por QTH, LED e PAC. Os resultados mostraram que independente do compósito e da fonte de luz, os grupos fotoativados por intensidade de luz e tempo de exposição intermediários apresentaram as maiores médias de dureza Knoop em camadas profundas. A relação dureza da base/dureza da superfície mostrou que o compósito Z250 apresentou adequada polimerização até 3 mm quando foi fotoativado por QTH e LED, e até 2 mm para PAC. O compósito Esthet-X apresentou adequada polimerização até 3 mm quando foi fotoativado por luz halógena e até 2 mm para LED e PAC.

Palavras Chave:

1- Materiais dentários; 2- Compósitos restauradores odontológicos; 3- Métodos de fotoativação; 4- Fontes de luz; 5- Propriedades mecânicas; 6- Dureza Knoop.

ABSTRACT

Nowadays, the photoactivated composites are the most used esthetic restorative dental materials in dentistry. The advantages of these materials are the control of setting time and esthetic characteristics. Besides that, it's unnecessary to prepare retentive cavity. Composites shrink during the polymerization reaction and a tension can be induced on restoration/tooth interface. Depending on composite/tooth interface bond strength, gaps can be formed in the restoration margins, leading to the failure of restorative procedure. Besides that, a high degree of conversion is necessary for composites to obtain adequate mechanical properties. The degree of conversion depends on energy density supplied to composites. The effects of energy density in mechanical properties of composites have not been satisfactory proved. In an attempt to improve the knowledge of the photo-activation methods upon the final properties of the restorative composite resins, this research was divided in two works. The first study evaluated the effect of increase on exposure time on Knoop hardness of composites Z250 and Esthet-X, photoactivated using XL2500 halogen light (QTH), Ultrablue Is Light emitting diode (LED) and Apollo 95E plasma arc curing (PAC). The results showed that higher Knoop hardness were produced by the increase on exposure time when LED and PAC were used. For QTH, the increase on exposure time did not produced significant effect. Regarding to light sources, PAC presented lower Knoop hardness than QTH and LED, which did not present significant difference from each other. Knoop hardness at deep layers was lower than superficial layers. The second study verified the influence of irradiance *versus* exposure time, using the same energy density, on Knoop hardness of Z250 and Esthet-X composites photoactivated using QTH, LED or PAC. The results showed that with intermediate light intensity and exposure times, the highest Knoop hardness were obtained, compared to others photoactivation methods. Composite Z250 presented adequate polymerization (bottom/surface relation above 0.8) up to 2 mm deep using PAC and up to 3 mm using QTH and LED. Esthet-X composite presented adequate polymerization up to 2 mm deep, using LED and PAC and up to 3 mm using QTH.

Keywords:

1- Dental materials; 2- Restorative composite resins; 3- Photo-activation methods; 4- Light sources; 5- Mechanical properties; 6- Knoop hardness.

INTRODUÇÃO GERAL

Os compósitos resinosos fotoativados são atualmente os materiais restauradores odontológicos diretos mais utilizados, tanto para dentes anteriores como posteriores. As vantagens destes materiais estão pautadas nas suas propriedades estéticas, no fácil controle do tempo de trabalho e nas suas propriedades adesivas. Mas estes materiais possuem algumas desvantagens, como o grau de conversão, que está na dependência da quantidade de energia luminosa a que são expostos. Assim, eles se polimerizam até uma certa profundidade, que varia com a penetração de luz no material. Este detrimento na polimerização tem sido chamado de profundidade de polimerização e tem influência significante nas propriedades físicas (Asmussen, 1982) e biológicas (Caughman *et al*., 1991) das restaurações. A profundidade de polimerização dos compósitos fotoativados é dependente da composição do material (Ruyter & Oysaed, 1982), de sua cor e transluscência (Ferracane *et al*., 1986), da intensidade da fonte de luz (Rueggeberg *et al*., 1994) ou da distância da ponta do aparelho fotoativador (Swartz *et al.,* 1983).

Uma outra desvantagem dos compósitos restauradores é a contração durante o processo de polimerização inerente a este tipo de material (Sakaguchi *et al*., 1991; Yap *et al*., 2000), podendo ocasionar desadaptação do material restaurador ao dente. Para minimizar a tensão gerada durante a contração de polimerização, surgiram técnicas que preconizam a utilização de baixa intensidade luminosa durante os períodos iniciais da fotoativação, diminuindo a velocidade da reação e a tensão gerada pela contração de polimerização.

A conversão dos monômeros para polímeros nunca se completa, devido a grande quantidade de ligações cruzadas entre as cadeias poliméricas, fazendo com que moléculas de monômeros fiquem aprisionadas entre as cadeias poliméricas, impedindo que estes se convertam (Ferracane, 1994). A conversão total de monômeros para polímeros ao final da reação é denominada grau de conversão, que pode ser avaliado indiretamente por meio da análise de dureza (Friedman *et al.,*1984).

Para se obter compósitos com propriedades físicas e mecânicas adequadas, é necessário que estes atinjam grau de conversão mínimo (Asmussen, 1982). Isto depende de fatores como: formulações dos monômeros (Ruyter *et al.*, 1987), temperatura da reação (Lovell *et al.*, 2001), tempo de exposição à luz dos compósitos fotoativados e eficiência do fotoiniciador, dentre outros. A irradiação pela fonte de luz e o tempo de exposição são de interesse particular, porque, na prática, estão sujeitos à manipulação pelo clínico.

Os métodos de fotoativação mais utilizados atualmente são por meio de luz de lâmpada halógena. Os aparelhos que utilizam lâmpada halógena para a fotoativação possuem grande variação espectral, de 390 a 520 nm, embora somente uma pequena parte do espectro emitido pelas lâmpadas incandescentes é apropriada para a ativação dos fotoiniciadores (Hoffmann *et al.*, 2002). Como alternativa a esse tipo de lâmpada para a fotoativação dos compósitos, foram desenvolvidos diodos que emitem luz (LED). Esses dispositivos possuem variação espectral muito pequena, o que os tornam altamente eficientes. Além disso, o calor gerado pelo LED é menor que os dispositivos que utilizam luz halógena (Hoffmann *et al.*, 2002). Outra vantagem do LED em relação às lâmpadas halógenas é sua longa vida útil (cerca de 10000 horas para LED e 40 a 100 horas para lâmpada halógena).

Outro problema dos aparelhos que emitem luz por lâmpada halógena é o tempo necessário para a fotoativação dos compósitos, principalmente quando utilizados em grandes reconstruções (Rueggeberg, 1994). Com o objetivo de reduzir o tempo de fotoativação, os fabricantes estão aumentando a potência dos aparelhos fotoativadores ou utilizando dispositivos de alta intensidade, como laser de argônio e arco de plasma. Acredita-se que estes dispositivos de alta intensidade, promovendo maior velocidade de polimerização, causem pior adaptação marginal do compósito resinoso, porque a tensão na interface dente/restauração é aumentada e, com isso, fendas podem ser formadas entre o compósito e o dente. Essa fenda propicia infiltração de microrganismos e fluidos bucais, bem como manchamento, inviabilizando a restauração. Outra desvantagem é a geração de calor destes aparelhos e, dependendo da profundidade da cavidade e da espessura de material restaurador, pode causar danos ao complexo dentina-polpa.

Outra dúvida em relação aos compósitos é se o grau de conversão está relacionado à densidade de energia aplicada ou ao método de fotoativação. Halvorson *et al*. (2002), utilizando um aparelho fotoativador com lâmpada halógena, não verificaram alterações no grau de conversão de quatro compósitos odontológicos, quando foi mantida a mesma densidade energética, obtida com diferentes intensidades e tempos de exposição. Sakagushi & Berge, (1998) verificaram que o processo de polimerização dependia mais da densidade energética do que da intensidade de luz. Entretanto existem dúvidas se somente a densidade energética seria a principal responsável pelas propriedades mecânicas dos compósitos. A intensidade da luz é um fator que deve ser considerado, e que pode exercer grande influência nas características da rede polimérica e, conseqüentemente, nas propriedades finais dos compósitos.

PROPOSIÇÃO

Em vista do questionamento a respeito do uso de densidades energéticas diferentes ou similares para se realizar a fotoativação de compósitos, o propósito deste estudo foi verificar:

1. O efeito do aumento da densidade energética na dureza Knoop dos compósitos Esthet-X e Z250, em várias profundidades, fotoativados por luz de lâmpada halógena, LED ou arco de plasma xenônio.

2. O efeito da relação intensidade de luz *versus* tempo de exposição, mantendo-se a mesma densidade energética, na dureza Knoop dos compósitos Esthet-X e Z250, em várias profundidades, fotoativados por luz de lâmpada halógena, LED ou arco de plasma de xenônio.

Este trabalho foi realizado no formato alternativo com base na deliberação da Comissão Central de Pós-Graduação (CCPG) da Universidade Estadual de Campinas (UNICAMP) nº001/98.

CAPÍTULO I

(*Brazilian Dental Journal – aceito para publicação*)

Effect of the energy density increase on Knoop hardness of dental composites light-cured by QTH, LED and xenon plasma arc

SUMMARY

The aim of this study was to evaluate the effect of the increase in exposure time on Knoop hardness of Z250 and Esthet-X composite resin. Cavities (3 mm diameter x 3 mm deep) were prepared on 144 bovine incisors. The composite was bulk inserted and light-cured by: QTH – 20s/700 mW/cm^2 (H1), 30s/700 mW/cm^2 (H2), 40s/700 mW/cm^2 (H3); LED – 20s/440 mW/cm^2 (L1), 30s/440 mW/cm^2 (L2), 40s/440 mW/cm^2 (L3); PAC – 3s/1700 mW/cm^2 (P1), 4.5s/1700 mW/cm^2 (P2), 6s/1700 mW/cm^2 (P3). The specimens were stored in incubator at 37°C for 24 hours prior to sectioning to Knoop hardness values measurement. Three measures were performed for each depth: on surface, 1 mm and 2 mm. The data were subjected to ANOVA and Tukey's test ($p<0.05$). Regardless to light source or energy density, Knoop hardness of Z250 was statistically higher than Esthet-X. PAC obtained lower hardness values than QTH and LED. Higher Knoop hardness was obtained when the energy density was increased for LED and PAC. For QTH there were no statistical differences. Knoop hardness values decrease with the increase in depth. The increase on energy density produced composites with higher Knoop hardness means using LED and PAC.

KEYWORDS: Composite resin, energy density, Knoop hardness, light curing, light sources.

INTRODUCTION

Nowadays, the resin composites are the most used esthetic restorative materials in Dentistry. Since their introduction they have been in constant modifications. One of main progresses was the introduction of the ultraviolet light activated composites, in the beginning of the 70's. Years later, light-cured resin composites activated by visible light were developed, which presented advantages as lower risk of damages to the patient's health and higher polymerization depth, when compared to ultraviolet light activated resin composites (1).

Since the introduction of the light-cured composites, there was also a constant evolution of the light sources and light-curing techniques. Recently, LEDs and high intensity light-curing devices, as the xenon plasma arc and lasers, were introduced for dental composites light-curing as an alternative to QTH curing units. LEDs and xenon plasma arc devices emit a narrow spectrum of light, at 450-490 nm, with the peak close to the peak of absorption of the camphoroquinone (468 nm). The specificity of the light emitted by LEDs is an advantage when light-curing composites activated by the amine/camphoroquinone system. For LED the use of filter to limit the width of the wavelength is unnecessary, besides them induce lower heating to the tooth during light-curing (2). But the current LEDs promote a considerable heating during the light-curing (3).

The xenon plasma arc is a high intensity light-curing unit that was introduced due to necessity of time saving in composites light-curing (4). This way, when the xenon plasma arc is used, the manufacturer recommends 3 s of exposure time to light-cure composites with camphoroquinone as photo-initiator system. But studies have shown that the mechanical properties of the composites light-cured by PAC with that short exposure time are insufficient.

The narrow wavelength spectrum of LEDs and PACs is a disadvantageous when the main photo-initiator system of the composites is not camphoroquinone. The activation of another photo-initiator system that absorbs light out of LEDs and PACs emitting wavelengths cannot efficiently be done (5). Therefore, the low degree of conversion can originate composites with deficient mechanical properties (6), besides higher citotoxity (7).

To induce the composite polymerization and obtain high degree of conversion it is necessary to supply them an appropriate energy density (8, 9). The energy density is the product of light intensity and exposure time. The energy density to obtain satisfactory mechanical properties of the composites can vary depending on the color, opacity, initiators and composition of the composite. Also, the increment volume and cavity configuration have an important role (10, 11). The light intensity should be compatible with the absorption wavelength spectrum of the photo-initiators systems. For camphoroquinone, which is the most used photo-initiator system in dental composites, the maximum absorption peak is among of the blue spectrum of visible light, about 468 nm (12).

When similar energy densities are supplied to the composite, similar degrees of conversion and depth of polymerization will be obtained, regardless the light-curing mode (9, 10). The degree of conversion increased can play an important role in the mechanical properties of the composite as decrease of the solubility, better dimensional stability, smaller color alteration and larger biocompatibility (13). Probably, composites with low mechanical properties will have short clinical life. The purpose of this study was to evaluate the influence of the increase of energy density in the Knoop hardness of two dental composites, using three types of light sources for light-curing: QTH light, LED and PAC.

MATERIAL AND METHODS

For this study 144 crowns of bovine incisors were embedded in PVC molds, with polystyrene resin (Piraglass, Piracicaba, SP 13424-550, Brazil), so that the buccal surface was exposed. After the inclusion, the buccal surface was flattened in a polishing machine APL-4 (Arotec Ind. Com., Cotia - SP, 06709-150, Brazil) using 180 grit sandpaper (Carborundum, Saint-Gobain Abrasivos Ltda, Cruz de Rebouças/Igaraçu, PE 53600-000, Brazil). Cylindrical cavities (3 mm depth and 3 mm diameter) were prepared under water-cooling with # 3018 diamond burs (Metalúrgica FAVA Ind. e Com. Ltda, Franco da Rocha, SP 02920-000, Brazil) on an air turbine (Kavo do Brasil S.A. Ind. e Com., Joinvile, SC 89221-040, Brazil). Samples were divided into two groups of 72, in agreement with the composite: Z250 (3M/ESPE Dental Products, St Paul, MN 55144, USA) or Esthet-X (Dentsply/Caulk, Milford, DE 19963-0359, USA), shade A3 (Table 1). The cavities were dried with air and composites Z250 or Esthet-X bulk inserted. A polyester strip was seated on the surface of the specimen and manually pressed to extrude composite excesses. The composites were light-cured with halogen light (XL2500, 3M/ESPE, St Paul, MN 55144, USA), LED (Ultrablue Is, D.M.C. Equipamentos Ltda. São Carlos, 13562030, São Paulo, SP, Brazil) or PAC (Apollo 95E, DMD, Westlake Village, CA 91362, USA), in agreement with the protocols showed in Table 2. Eight specimens were prepared by each group.

Light intensities of 700 mW/cm^2, 440 mW/cm^2, and 1700 mW/cm^2 were the maximum intensities of QTH, LED and PAC, respectively, checked with digital radiometer Dental Hilux Curing Light (Dental Benlioglu Inc., Binnaz SK 1-6 Kavaklidere, Ankara 06700, Turkey). In agreement with the manufacturers' recommendations, the exposure time for Z250 and Esthet-X composites for QTH and LED were 20 s, and PAC was 3 s. Therefore, the groups light-cured using QTH with 700 mW/cm^2/20 s, LED with 440 mW/cm^2/20 s and PAC with 1700 mW/cm^2/3 s were considered as control groups.

The energy densities of the QTH, LED and PAC control groups were 14 J/cm^2, 8.8 J/cm^2, and 5.1 J/cm^2, respectively (Table 2). Energy densities increased were obtained by longer exposure times.

After light curing, the specimens were stored at 37ºC during 24 h ± 1, in dark and dry container. Elapsed 24 hours of the photoactivation, the specimens were sectioned in mesio-distal direction, using the diamond-wafering blade (Extec Corp., Enfield, 06083-1258, CT, USA) mounted in a metalographic cutter (Isomet 1000, Buhler, Lake Bluff, 60044, IL, USA), under water-cooling. After sectioning, the samples were ground and polished using 320, 400, 600 and 1200 grit sandpapers (Carborundum, Saint-Gobain Abrasivos Ltda, Cruz de Rebouças/Igaraçu, PE 53600-000, Brazil) on an automated polisher under water cooling. The specimens were dried and submitted to the Knoop hardness measurements in a microhardness tester (HMV-2000, Shimadzu, Tokyo 101, Japan) with load of 50 g and time of 15 s. The Knoop hardness readings were accomplished on surface, 1, and 2 mm depth, for each depth, three readings were taken and an average was calculated.

The data were submitted to ANOVA and the means compared by Tukey´s test at the 5% significance level.

RESULTS

The Knoop hardness of Z250 composite was statistically higher ($p<0.05$) than Esthet-X composite (Table 3).

In general, the light curing with PAC presented lower Knoop hardness means than QTH and LED. Z250 composite light cured using PAC presented lower Knoop hardness means starting from 1 mm depth, for energy densities of 100 and 150%. With 200% there was significant difference among the methods solely on the surface (Table 4). For Esthet-X composite, PAC presented lower Knoop hardness means at 2 mm depth, for the three densities (Table 5). There was no statistical difference for Knoop hardness between QTH and LED (Tables 4 and 5).

Table 4 shows that, for Z250 composite, Knoop hardness on the surface was statistically higher than 1 and 2 mm depth when PAC was used with 100% energy density. For 150% energy density, the hardness in the surface was statistically higher than 2 mm. For 200% there was no statistical difference among the depths. When LED was used, the hardness on the surface was higher than 2 mm solely for 200% energy density. For QTH, there was no statistical difference among the depths for all energy densities.

The Table 5 shows that, for Esthet-X composite, the hardness on the surface was statistically higher than 2 mm depth for 100% and 150% energy densities, when QTH unit was used. For 200% energy density, there was no statistical difference among the depths. For LED, the hardness on the surface was statistically higher than 2 mm depth for all energy densities. For PAC, the hardness on the surface and 1 mm were statistically higher than 2 mm depth for all energy densities.

Regarding to energy density (Table 6), for the composite Z250, the Knoop hardness for 100% energy density was lower than other energy densities for LED and PAC, exception for LED at 1 mm and PAC on the surface. For QTH there was no statistical difference for Knoop hardness among the energy densities.

For Esthet-X (Table 7), the Knoop hardness for 100% energy density was statistically lower than 200% energy density when PAC was used, just in 2 mm depth. For QTH and LED there was no statistical difference among the energy densities, for all depths.

DISCUSSION

The hardness of the composites is influenced by several factors as composition of the organic matrix (14), type and amount of filler particles (15) and also degree of conversion (16). In this study, the Knoop hardness values of the composite Z250 was higher compared to those the Esthet-X composite. The organic matrix of the composite Z250 is mainly composed by BisGMA, UDMA and BisEMA, and the inorganic particles are zircon/silica (60% in volume). In contrast, the organic matrix of the composite Esthet-X is composed mainly by Urethane Modified BisGMA, BisEMA and TEGDMA, and by a combination of inorganic particles of a glass Aluminum Fluorine Borosilicate and Silanized Barium, colloidal and nanometric silica (60% in volume). The higher Knoop hardness values for Z250 composite may be explained by differences in filler type and organic matrix composition between Z250 and Esthet-X composites. Composites with harder filler particles exhibit higher surface hardness. However, the bonding of the filler particles to the polymeric matrix also affects their hardness values (17).

Many studies have shown that the degree of conversion depends most on the amount of energy supplied to the composite than the light-curing method (9). The hardness evaluation is an indirect method to verify the degree of conversion of resin composites (16). The hardness values show a positive correlation with degree of conversion.

Higher hardness values can be obtained by exposure time and energy density increased (18). Z250 composite light-cured using LED and PAC presented higher Knoop hardness means, using exposure time and energy density increased. For the composite Esthet-X, when PAC was used, there was an increase on Knoop hardness means using longer exposure times. The increase on energy density produced higher degree of conversion and, consequently, higher Knoop hardness means.

The comparison among the light sources showed that PAC presented the worst Knoop hardness means. Degree of conversion depends on the amount of energy supplied to the composite. Low energy density produces composites with low degree of conversion and deficient mechanical properties, like Knoop hardness (6). Due to short exposure time, even with high intensity, the low energy density supplied using PAC produced composites with lower Knoop hardness means.

There was no statistical difference of Knoop hardness means between LED and QTH up to 2 mm depth. For QTH, in spite of higher energy density than LED, many of the emitted photons are out of the spectrum of absorption of the camphoroquinone (5). Therefore, the triplex state of the camphoroquinone is not activated. For LEDs, the narrow wavelength spectrum inside of the spectrum of absorption of the camphoroquinone, the specific energy density for the camphoroquinone is higher, compensating the lower light intensity emitted by those devices. The recent progresses in LED technology made possible the obtaining of devices with higher light intensity, which allows the light-curing with shorter exposure times (20s) with enough energy densities to produce composites with sufficient mechanical properties (19).

Regarding to the polymerization depth, the hardness values decreased in direction to deeper layers. The polymerization depth of the light-cured composites depends on the composition of the restoring material, color and translucence, intensity of the light source and distance of the tip of the light-curing device from to the composite surface (20). All those factors influence the amount of light that reach deep layers of composite, then the degree of conversion in those areas is lower, and the mechanical properties, as the hardness Knoop, are influenced negatively.

It can be verified in this study that the increase of energy density influenced on the Knoop hardness values in deeper areas of the restoration, mainly for Z250 light cured with LED or PAC. Therefore, the knowledge and the understanding of the mechanisms that can cause problems to light curing composites associated with the techniques that can reduce their effects could help the clinicians to obtain the maximum benefits of the application of those materials in practice.

Based on these results, it can be concluded that the Knoop hardness means of composite Z250 was higher than Esthet-X composite. The increase on exposure time produced higher Knoop hardness values when Z250 composite was photoactivated using LED and PAC. For Esthet-X composite the increase on exposure time produced higher Knoop hardness solely when PAC was used. Besides, composites photoactivated using PAC get the lowest Knoop hardness means and there were no statistical differences on Knoop hardness when composites were photoactivated using QTH or LED.

Efeito do aumento da densidade energética sobre a dureza Knoop de compósitos odontológicos fotoativados por luz de lâmpada halógena, LED e arco de plasma de xenônio

RESUMO

O estudo teve como objetivo avaliar o efeito dos métodos de fotoativação na dureza Knoop dos compósitos Z250 e Esthet-X. Cavidades (3 mm de diâmetro x 3 mm de profundidade) foram preparadas em 144 incisivos bovinos. Os métodos de fotoativação foram: luz halógena – 20 s/700 mW/cm^2 (H1), 30 s/700 mW/cm^2 (H2), 40 s/700 mW/cm^2 (H3); LED - 20 s/440 mW/cm^2 (L1), 30 s/440 mW/cm^2 (L2), 40 s/440 mW/cm^2 (L3); arco de plasma de xenônio – 3 s/1700 mW/cm^2 (P1), 4,5 s/1700 mW/cm^2 (P2), 6 s/170 mW/cm^2 (P3). Após armazenagem a 37°C durante 24 horas, foram realizadas três leituras por profundidade: superfície, 1 mm e 2 mm. Os dados foram submetidos a ANOVA e ao teste de Tukey ($p<0,05$). A dureza do compósito Z250 foi estatisticamente superior ao do compósito Esthet-X. A dureza dos compósitos fotoativados por PAC foi estatisticamente inferior em relação aos compósitos fotoativados por luz halógena ou LED, que não diferiram entre si, independente da profundidade. O aumento da densidade energética produziu compósitos com maiores valores de dureza Knoop quando se utilizou LED ou PAC. Para luz halógena o aumento do tempo de exposição não influenciou os valores de dureza.

PALAVRAS-CHAVE: Compósito, densidade de energia, dureza Knoop, fotoativação, aparelhos fotoativadores.

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Table 1. Composition of the composites Z250 and Esthet-X (according to manufacturer's information).

Resin Composite	Composition		Batch
	Organic Matrix	**Filler**	
Z250	BisGMA, UDMA and BisEMA Camphoroquinone (initiator)	Zirconia/silica 0.19 – 3.3 μm 60% (vol.)	3CR
Esthet-X	Bis-GMA-adduct Bis-EMA TEGDMA Camphoroquinone Stabilizers	Barium-fluoro-alumino-boro-silicate < 1 μm Highly dispersed silicon dioxide 0.04 μm 60% (vol.)	030909

Table 2. Light-curing methods with different light-curing units.

Light source	Exposure time (s)	Light intensity (mW/cm^2)	Energy density (J/cm^2)	Energy density (%)
Halogen	20	700	14	100 (control)
	30	700	21	150
	40	700	28	200
LED	20	440	8.8	100 (control)
	30	440	13.2	150
	40	440	17.6	200
PAC	3	1700	5.1	100 (control)
	4.5	1700	7.65	150
	6	1700	10.2	200

Table 3: Means of Knoop hardness of composites Esthet-X and Z250, despite light-curing method, light source and depth.

Resin composite	Knoop Hardness Number (KHN)
Z250	72.9 a
Esthet-X	56.0 b

Means followed by distinct small letter represent statistical significant differences (5%).

Table 4: Knoop hardness means and standard deviation for Z250 composite in several depths, using different light sources and energy densities.

Energy density	Light source	Depth		
		Surface	1 mm	2 mm
100%	**Halogen**	74.5 (2.2) a, A	72.8 (2.2) a, A	71.0 (2.6) a, A
	LED	72.9 (3.4) a, A	71.8 (4.8) ab, A	69.7 (5.0) a, A
	PAC	72.5 (3.9) a, A	68.1 (3.7) b, B	64.7 (4.5) b, B
150%	**Halogen**	75.5 (2.5) a, A	74.1 (3.3) ab, A	73.3 (2.3) a, A
	LED	75.6 (4.1) a, A	75.3 (3.0) a, A	74.4 (2.4) a, A
	PAC	73.3 (2.8) a, A	70.7 (3.8) b, AB	67.4 (4.4) b, B
200%	**Halogen**	76.8 (3.3) ab, A	75.5 (2.9) a, A	73.6 (4.5) a, A
	LED	78.6 (2.8) a, A	75.5 (3.7) a, AB	72.9 (2.8) a, B
	PAC	74.1 (2.9) b, A	73.3 (2.9) a, A	71.0 (4.1) a, A

Means followed by different small letter in the column for each energy density and capital letter in the row represent statistical difference (5%).

Table 5: Knoop hardness means and standard deviation for Esthet-X composite, in several depths, using different light sources and energy densities.

Energy density	Light source	Depth		
		Surface	1 mm	2 mm
100%	Halogen	57.8 (2.5) a, A	55.9 (1.7) a, AB	53.4 (2.5) a, B
	LED	58.6 (1.9) a, A	56.0 (2.6) a, AB	53.0 (1.6) a, B
	PAC	56.8 (4.1) a, A	54.1 (4.0) a, A	45.8 (5.7) b, B
150%	Halogen	60.3 (3.0) a, A	57.2 (2.2) a, AB	55.7 (2.1) a, B
	LED	58.4 (2.6) a, A	56.4 (2.3) a, AB	53.8 (2.5) a, B
	PAC	58.4 (1.9) a, A	55.7 (3.3) a, A	49.4 (4.4) b, B
200%	Halogen	59.9 (4.3) a, A	57.8 (2.6) a, A	56.3 (2.5) a, A
	LED	60.6 (3.2) a, A	58.7 (2.7) a, AB	55.2 (3.2) ab, B
	PAC	60.2 (3.1) a, A	56.5 (2.2) a, A	51.5 (3.7) b, B

Means followed by different small letter in the column for each energy density and capital letter in the row represent statistical difference (5%).

Table 6: Knoop hardness means and standard deviation for Z250 composite in several depths, using different light sources and energy densities.

Light source	**Energy density**	**Depth**		
		Surface	**1 mm**	**2 mm**
Halogen	**100%**	74.5 (2.2) a	72.8 (2.2) a	71.0 (2.6) a
	150%	75.5 (2.5) a	74.1 (3.3) a	73.3 (2.3) a
	200%	76.8 (3.3) a	75.5 (2.9) a	73.6 (4.5) a
LED	**100%**	72.9 (3.4) b	71.8 (4.8) a	69.7 (5.0) b
	150%	75.6 (4.1) ab	75.3 (3.0) a	74.4 (2.4) a
	200%	78.6 (2.8) a	75.5 (3.7) a	72.9 (2.8) ab
PAC	**100%**	72.5 (3.9) a	68.1 (3.7) b	64.7 (4.5) b
	150%	73.3 (2.8) a	70.7 (3.8) ab	67.4 (4.4) ab
	200%	74.1 (2.9) a	73.3 (2.9) a	71.0 (4.1) a

Means followed by different small letter in the column for each light source represent statistical difference (5%).

Table 7: Knoop hardness means and standard deviation for Esthet-X composite, in several depths, using different light sources and energy densities.

Light source	Energy density	Depth		
		Surface	1 mm	2 mm
Halogen	100%	57.8 (2.5) a	55.9 (1.7) a	53.4 (2.5) a
	150%	60.3 (3.0) a	57.2 (2.2) a	55.7 (2.1) a
	200%	59.9 (4.3) a	57.8 (2.6) a	56.3 (2.5) a
LED	100%	58.6 (1.9) a	56.0 (2.6) a	53.0 (1.6) a
	150%	58.4 (2.6) a	56.4 (2.3) a	53.8 (2.5) a
	200%	60.6 (3.2) a	58.7 (2.7) a	55.2 (3.2) a
PAC	100%	56.8 (4.1) a	54.1 (4.0) a	45.8 (5.7) b
	150%	58.4 (1.9) a	55.7 (3.3) a	49.4 (4.4) ab
	200%	60.2 (3.1) a	56.5 (2.2) a	51.5 (3.7) a

Means followed by different small letter in the column for each light source represent statistical difference (5%).

CAPÍTULO II

(*Acta Odontologica Scandinavica – enviado para publicação*)

Effect of exposure time *versus* irradiance relationship on Knoop hardness of dental composites

Short title: **Light modulation effect on composite hardness**

ABSTRACT

The aim of this study was to evaluate the effect of many methods to attain the same light power density on Knoop hardness of Z250 and Esthet-X composites. Cavities (3 mm diameter x 6 mm deep) were prepared on 240 bovine incisors. The composite was bulk inserted and light-cured using halogen light, LED or xenon plasma arc with different combinations of light intensities and exposure times, always maintaining the same energy density. The specimens were stored in incubator at 37ºC for 24 h prior to sectioning for hardness values measurement in a dry and dark container. Three measurements were performed for each depth: on surface, 1 mm, 2 mm, 3 mm, 4 mm, and 5 mm. Data were submitted to ANOVA and Tukey's test ($p<0.05$). For light-curing method there were no statistical differences among the groups up to 2 mm for LED, up to 3 mm for PAC, and up to 5 mm for halogen light. The photo-activation with intermediate light intensity and exposure time obtained the highest Knoop hardness values.

KEY WORDS: composite resin, energy density, Knoop hardness, light curing, light sources.

INTRODUCTION

Since the introduction of resin composite in Dentistry, in the late 70's, light-curing units and light curing methods have been in constant evolution. Resin composites photo-activated by blue light have been found as the best photo-activation method (1). Quartz-Tungsten-Halogen (QTH) "bulbs" have been the most common light sources in handheld dental curing units. Halogen light-curing units used to polymerize dental composites have several drawbacks despite their popularity. Operating with a white halogen bulb filtered by a dielectric pass-band filter to remove the undesirable wavelengths, conventional composite-curing lamps operate in the deep blue region of the spectrum. However, this type of equipment still emits a considerable amount of other wavelengths. The spectral impurities of the conventional curing-lights deliver several wavelengths that are highly absorbed by dental materials, inducing heating to tooth and resin during the curing process (2). Besides that, the halogen bulbs (which have an effective lifetime limited to about 40 to 100 hours), reflector and filter degrade over time due to high operating temperatures and the large quantity of heat produced during the curing cycles (3).

Recently, the light emitting diodes (LEDs) and high light intensity devices, like plasma arc curing lights (PACs) and argon ion lasers, were introduced to resin composite photoactivation as an alternative option for QTH curing units. LEDs have lifetimes of more than 10,000 hours and undergo little degradation of light output over time. They use junctions of doped semiconductors (p-n junctions) to generate light and, hence, require no filters to produce blue light and are resistant to shock and vibration. Their relative low power consumption makes them suitable for portable use. The narrower spectral output of these blue LEDs of 440 to 490 nm fall within the camphoroquinone (CQ) absorption spectrum (2).

Long curing time is inconvenient for the patient, impractical with children, uncomfortable for the dentist, and makes the treatment more expensive because of extra-time chairside. With the objective to reduce light-curing time, PACs were introduced, which the aim a reduction on exposure time by application of high irradiation. The light is emitted from a glowing plasma, which is composed of a gaseous mixture of ionized molecules and electrons. PAC units are characterized by a very high output in a rather narrow range of wavelengths around 470 nm (4). A question that may be raised relates to the cure rate, and their influence on gap formation between toothe and restorative material. This is because high curing rates tend to result in increased wall-to-wall contraction (5). Some works showed that resin composite light-cured by PAC, with high irradiance delivered in the course of a few seconds was not enough to bring optimal properties.

The narrow wavelength emitted by LED and PAC is advantageous when the initiator system of resin composite is the CQ, because the use of optical filters to limit the wavelength is not necessary. Besides that, it induces lower overheating of the teeth and resins during the curing process (2). Despite of this, current LEDs promote a considerable heating during light curing (6). However, due to the narrow spectrum of wavelength of LED and PAC, when the main photo-initiator system of the composite is not camphoroquinone, the activation of other photo-initiator systems that absorb light out of the band of LEDs and PACs emitting wavelengths cannot efficiently occur (3, 7, 8). Thus, the low degree of conversion can produce composites with deficient mechanical properties (9, 10, 11, 12), besides higher citotoxicity (13).

For the composite polymerization initiation and to obtain a high degree of conversion, it is necessary to supply an appropriate energy density to the composite (14, 9). The energy density is a product of light intensity and exposure time. This amount of necessary energy to obtain satisfactory mechanical properties can vary depending on the color, opacity, initiators and composition of the composites. Also the increment volume and cavity configuration have an important role (15, 16, 17). The light intensity should be compatible with the absorption spectrum of the photo-initiator systems. For camphoroquinone, which is the most used in the composites, the maximum absorption peak fall inside the blue band of the visible light, about 468 nm (18).

When similar energy densities are supplied to the composite, similar degree of conversion and depth polymerization would be expected, regardless the light-curing method. Therefore, similar degree of conversion and depth polymerization can be obtained by applying low light intensity by longer exposure time or high light intensity by shorter exposure time, since that the same energy density is maintained (15,19,20).

During the light curing, the light-curing unit tip should ideally be in direct contact with the resin composite. However, this is not always clinically possible. In proximal restorations it has been demonstrated that the distance between light tip and proximal cavity bottom is of 8 mm or over (17). Light intensity diminishes as the curing tip is moved farther from the composite resin restorative material (21). Correr Sobrinho et al. (22) and Caldas et al. (23) observed that resin composite Knoop Hardness Number (KHN) decreased when the distance between the curing light tip and the resin composite increased, supporting the law of the Inverse Square (5).

Besides, several of those combinations of light modulation have been used composites light-curing with the objective of minimizing the stress of polymerization shrinkage.

However, doubts about the limits of modulation of light intensity and exposure time that can promote an appropriate composite polymerization and, consequently, better physical and mechanical properties still exist. The objective of this study was to verify the effect of exposure time *versus* light intensity, maintaining the same energy density, in the Knoop hardness of two dental composites, in several depths, using three different light-curing units.

METHODS AND MATERIALS

For this study 240 bovine incisors were embedded in PVC molds with polystyrene resin (Piraglass, Piracicaba, SP, Brazil), maintaining the buccal face exposed. The buccal surface was ground flat in polishing machine APL-4 (Arotec Ind. Com., Cotia, SP, Brazil) using 180 grit sandpaper (Carborundum, Saint-Gobain Abrasivos Ltda, Cruz de Rebouças/Igaraçu, PE, Brazil). Cylindrical cavities (6 mm depth and 3 mm diameter) were prepared under water cooling with diamond burs #3018HL (Metalúrgica FAVA Ind. e Com. Ltda, Franco da Rocha, SP, Brazil) on an air turbine (Kavo do Brasil S.A. Ind. e Com., Joinvile, SC, Brazil). Samples were divided in two groups of 120 specimens each, in agreement with the applied composite: Z250 (3M/ESPE Dental Products, St Paul, MN, USA) or Esthet-X (Dentsply/Caulk, Milford, DE, USA), shade A3 (Table 1). The cavities were air dried and composites Z250 or Esthet-X were bulk inserted. A polyester strip was seated on surface of the specimen and pressed manually to remove composite excesses. Composites were light-cured with halogen lamp (XL2500, 3M/ESPE, St Paul, MN, USA), LED (Ultrablue Is, D.M.C. Equipments Ltd. São Carlos, São Paulo. SP, Brazil) or PAC (Apollo 95E, DMD, Westlake Village, CA, USA), in agreement with the protocols proposed in Table 2. Eight specimens were prepared by group.

The light intensities of 700 mW/cm^2, 440 mW/cm^2, 1700 mW/cm^2 were the maximum intensities of the QTH, LED and PAC, respectively, checked with digital radiometer (Dental Hilux Curing Light to Put, Dental Benlioglu Inc., Binnaz SK 1-6 Kavaklidere, Ankara, Turkey). In agreement with the manufacturers' recommendations, the time of light-curing of the composites Z250 and Esthet-X for QTH and LED were of 20 s, and PAC was of 3 s. Therefore, the groups photo-activated using QTH for 20 s/700 mW/cm^2, LED for 20 s/440 mW/cm^2 and PAC for 3 s/1700 mW/cm^2 were considered the control groups.

The energy densities of the control groups for QTH, LED and PAC were 14 J/cm^2, 8.8 J/cm^2, and 5.1 J/cm^2, respectively (Table 2). For the reduction on light intensities, the tip of curing units was moved away in relation to composite surface. In order to standardize the photoactivation distance, spacers of acrylic resin (JET, Artigos Odontológicos Clássico, São Paulo, SP, Brazil) were interposed between the surface of the composite and the tip of the light curing units. For the spacers fabrication, PVC molds were filled with acrylic resin. After acrylic resin curing, a hole at the center of mold was made to permit the light pass. The acrylic resin molds were ground to obtain the exact light intensity. For photoactivation by low light intensities, the exposure times were increased to match the energy density of control groups (Table 2).

After light-curing, the specimens were stored in incubator at 37ºC for 24 h ± 1, in dark and dry container. Elapsed 24 hours of the photoactivation, the specimens were sectioned in mesio-distal direction, using a diamond wafering blade (Extec Corp., Enfield, CT, USES) on a metallographic cutter (Isomet 1000, Buheler, Lake Bluff, IL, USES), under cooling water. After the sectioning, the restorations were ground and polished using 320, 400, 600 and 1200 grit sandpapers (Carborundum, Saint-Gobain Abrasivos Ltda, Cruz de Rebouças/Igaraçu, PE, Brazil) on an automated polisher under water cooling. The specimens were dry and submitted to the Knoop hardness measurements in a microhardness tester (HMV-2000, Shimadzu, Tokyo, Japan) with load of 50 g and time of 15 s. The readings of Knoop hardness were accomplished on surface, 1, 2, 3, 4, and 5 mm deep, and, for each depth, three readings were taken and an average was calculated.

The data were submitted to analysis of variance in factorial outline with split-plot and the means compared by Tukey test at the 5% significance level.

RESULTS

The results of this study showed that the Z250 composite presented Knoop hardness means statistically higher ($p<0.05$) than Esthet-X composite (Table 3).

For photoactivation method, middle light intensity and exposure time produced highest Knoop hardness means. For QTH (Table 4), there were statistical differences for Knoop hardness only at 5 mm deep among photoactivation methods. For the composite Z250, the Knoop hardness mean to H3 group was statistically higher than H4 and H5 groups. For the composite Esthet-X, the Knoop hardness mean to H8 and H9 groups were statistically higher than H6 and H7 groups. When LED was used (Table 5), there was significant difference for the Z250 composite starting from 2 mm deep, with Knoop hardness mean to L3 group being statistically higher than L5 group. For composite Esthet-X there was statistical difference among groups at 3 mm deep, with highest

Knoop hardness means to L8 and lowest values to L9 group. For PAC (Table 6), there were statistical differences among the photoactivation methods starting from 3 mm deep. Knoop hardness mean to P3 group was statistically higher than P1 group for composite Z250. For composite Esthet-X, Knoop hardness mean to P7, P8, P9 and P10 groups were statistically higher than P6 group.

The hardness relationship bottom/surface (above 0.8 it means that the composite had an appropriate polymerization) showed that the composite Esthet-X presented good polymerization up to 2 mm depth using halogen light, LED and PAC. When Z250 was used, the hardness relationship bottom/surface was adequate up to 2 mm for PAC and up to 3 mm for QTH and LED.

DISCUSSION

In this study, the Knoop hardness of the Z250 composite was higher than composite Esthet-X. The hardness of the composites is influenced by several factors as composition of the organic matrix (24), type and amount of filler particles (25) and also by degree of conversion (26). The organic matrix composition of Z250 composite is mainly BisGMA, UDMA and BisEMA, and the inorganic filler of zirconia/silica (60%v). The organic matrix composition of Esthet-X composite is BisGMA Modified Uretane, BisEMA and TEGDMA, and the inorganic fillers are a combination of Barium fluoro alumino boro silicate glass and highly dispersed silicon dioxide (60% v). The greater Knoop hardness values for composite Z250 may be explained by differences in filler type and organic matrix composition between Z250 and Esthet-X composites. Composites with harder filler particles exhibit higher surface hardness. However, the bond of the inorganic filler to the polymeric matrix also affects their hardness values (24).

Nowadays, several methods of light modulation are used for the photoactivation of the composites with the objective of minimizing the tension generated by the polymerization shrinkage. Regardless of light modulation method, it has been recommended that energy densities similar those supplied by conventional photoactivation (continuous mode) be used. This is recommended because several studies showed that the degree of conversion depends more on energy density that is supplied to the composite than on the photoactivation method (9, 27). The hardness is a mechanical property indirectly related to degree of conversion of composites. Higher hardness means can be obtained by increase on degree of conversion, depending on light curing method (26, 28, 29).

In this study there were no statistical differences among light curing methods up to 2 mm deep, except for L5 group. This finding is in agreement with those found by Rueggeberg et al. (16) and Sakaguchi & Berge (30), who concluded that at the top surface of composites, exposure time solely is a significant factor contributing to monomer conversion. However, as light passed through the bulk of the composite, light intensity reduces greatly due to light scattering and absorption. This decreases the effectiveness of polymerization (1). Therefore, due to reduced light intensity at deeper regions, the degree of conversion and Knoop hardness are decreased. For the inner parts, only optimal cure should be considered, as pulpal tissues are affected by the leaching of unpolymerized components (13).

In this work, intermediate exposure times and light intensities produced higher Knoop hardness than groups light-cured using a short exposure time using high light intensity or long exposure time using low light intensity, in deeper layers. For groups light-cured using low light intensity and long exposure time, due to light scattering and absorption, few amount of light reach deep layers, producing composites with lower mechanical properties. The units emitting high light intensity during the first 10-15 s of the photopolymerization may cause rapid network formation at the superficial layer of the resin composite. This reduces light transmittance throughout the bulk material, due to changes in the optical properties of this zone (12). Moreover, the short irradiation time of the plasma-arc unit may be inadequate for efficient light diffusion at deep portions. Subsequently, that leads to limitation in reaction between the excited camphoroquinone molecule and the amine (31). The frequency of cross-links is unaffected by the rate of initiation, but the distance between cross-links is smaller than the predicted kinetic chain lengths (32). Therefore, this lower distance between cross-links may explain the differences in network transmittance.

Since the same energy density is maintained, light-curing units with low light intensity needs longer exposure times to produce similar degrees of conversion (9). At the top surface, only irradiation time is a significant factor contributing to monomer conversion (16, 30). However, due to light scattering and absorption through the bulk of the composite, only fewer photons of the low light intensity light sources reaches deeper layers, despite of longer exposure times (20). Therefore, the degree of conversion and the Knoop hardness at deeper layers is much decreased.

The highest Knoop Hardness means at deep layers were attained by resin composites light cured using intermediate exposure times and light intensities. Due to slower initial polymerization rate, the density crosslink is lower than resin composite light cured by high light intensity (33). The lower initial light intensity, associated with longer exposure time, permits that more photons reach deeper layers promoting better polymerization and higher Knoop hardness means.

In regard to depth of polymerization, the Knoop hardness means diminished from the surface toward deeper layers. The depth of polymerization of the photoactivated composites depends on composition of restorative material, color and translucency, light intensity and the tip distance of light curing units to the surface of composite (34). All these factors influence the amount of light that reaches deep layers of composite. Thus, the degree of conversion in these layers is lower, and the mechanical properties like Knoop hardness are negatively influenced (9, 10, 11, 12).

An ideal bottom-to-top hardness ratio of 1:1 should be achieved for effective polymerization, as the degree of polymerization should be the same throughout its depth. Light scattering and attenuation may have accounted for minor differences in hardness between the top surfaces and bottom of the light-activated composites evaluated in this study. It has been suggested that hardness gradient should not exceed 10-20% (hardness ratio should be greater than 0.8) for adequately photo-activated resin composites (35, 36). The hardness ratio of all light curing regimens at a depth of 2 mm was above 0.8. The hardness ratios of H10, L6, L7, L9, L10, P1, P4, P5, P6, P7, P8, P9 and P10 at 3 mm deep were lower than 0.8. The transmission coefficient is influenced by light wavelength, refractive indices of fillers and resin matrix, shade, opacity, filler type and size, and loading (37). Light scattering is related to filler particle size and it has been suggested that light attenuation is maximized when filler particle size is half the wavelength of the activating light (1). Smaller filler scatter more light than composites with larger and fewer glass particles (24). The mean size of fillers of Esthet-X composite is smaller than Z250 composites (see Table 1), leading to differences in scattering light and transmittance through bulk of composite. This permit that lower light intensity reaches deeper layers and polymerizes the resin composite. Besides that, depth of cure may be correlated to the composition of the monomers used in dental composites. The differences in resin matrix of composites, leading to different characteristics in light transmission properties, also determine the conversion profile and depth of cure.

The Knoop hardness for Z250 composite was higher than Esthet-X. Up to 2 mm deep, there was no statistical difference among the combinations of light intensity and exposure time for all light sources and composites, except for Z250 light-cured with LED, which was influenced since 1 mm deep. In deeper regions, intermediate light intensity and exposure time produced composites with the highest Knoop hardness values. Composites presented appropriate polymerization up to 2 mm deep for LED and PAC, and 3 mm for the QTH light. For the same energy density, the modulation methods using intermediate intensities were used promoted similar or better results than those than with high or low light intensity. Clinically, these methods could be a valid option to the traditional methods of photoactivation, even being a longer clinical time to accomplish the light-curing of the composites.

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Table 1. Composition of Z250 and Esthet-X composites (according to manufacturer's information).

Resin Composite	Composition: Organic Matrix	Composition: Filler	Batch
Z250	BisGMA, UDMA and BisEMA Camphoroquinone (initiator)	Zirconia/silica 0.19 – 3.3 µm 60% (vol.)	3CK
Esthet-X	Bis-GMA-adduct Bis-EMA TEGDMA Camphoroquinone Stabilizers	Barium-fluoro-alumino-boro-silicate < 1 µm Highly dispersed silicon dioxide 0.04 µm 60% (vol.)	0110161

Table 2: Light curing methods for QTH, LED, and PAC.

Light source	Groups		Exposure	Light intensity	Energy density
	Z250	Esthet-X	time (s)	(mW/cm^2)	(J/cm^2)
QTH	H1	H6	20	700	14
	H2	H7	28	500	14
	H3	H8	35	400	14
	H4	H9	70	200	14
	H5	H10	140	100	14
LED	L1	L6	20	440	8.8
	L2	L7	29.3	300	8.8
	L3	L8	40	220	8.8
	L4	L9	80	110	8.8
	L5	L10	160	55	8.8
PAC	P1	P6	3	1700	5.1
	P2	P7	6	850	5.1
	P3	P8	12.75	400	5.1
	P4	P9	25.5	200	5.1
	P5	P10	51	100	5.1

Table 3: Means of Knoop hardness of Esthet-X and Z250 composites, regardless light-curing method and depth.

Light source	**Resin Composite**	
	Z250	**Esthet-X**
QTH	66.2 a	43.0 b
LED	60.7 a	38.5 b
PAC	51.6 a	33.4 b

Means followed by distinct small letter represent statistical significant differences in the row by Tukey´s test (5%).

Table 4: Knoop hardness means and standard deviation for Z250 and Esthet-X composite photoactivated using QTH, in several depths.

Composite	Group	Surface (H0)	1 mm (H1)	2 mm (H2)	3 mm (H3)	4 mm (H4)	5 mm (H5)
Z250	**H1**	74.5 (2.2) a	72.8 (2.2) a	71.0 (2.6) a	67.9 (3.7) a	60.2 (4.9) a	45.3 (9.9) ab *
	H2	74.8 (2.8) a	73.6 (4.5) a	71.4 (3.6) a	69.1 (2.7) a	62.1 (3.9) a	47.5 (3.5) ab *
	H3	75.7 (3.1) a	75.1 (2.9) a	73.9 (2.1) a	72.0 (3.8) a	64.1 (2.9) a	52.0 (4.4) a *
	H4	75.0 (2.4) a	72.9 (4.6) a	70.2 (5.3) a	66.6 (6.3) a	59.2 (6.3) a *	42.8 (10.0) b *
	H5	75.0 (2.6) a	74.9 (2.6) a	73.1 (2.8) a	69.1 (3.3) a	61.6 (4.1) a	42.3 (7.8) b *
ESTHET-X	**H6**	57.8 (2.5) a	55.9 (1.7) a	53.4 (2.5) a	49.4 (4.0) a	31.3 (7.1) a *	6.8 (9.6) b *
	H7	59.3 (2.9) a	56.8 (2.2) a	53.9 (2.0) a	49.1 (0.9) a	33.3 (5.4) a *	8.3 (9.6) b *
	H8	59.5 (1.9) a	56.5 (2.0) a	53.4 (2.7) a	47.7 (3.3) a	37.5 (10.6) a *	17.3 (12.6) a *
	H9	57.0 (3.1) a	54.7 (4.2) a	50.1 (4.2) a	47.0 (4.5) a	33.0 (8.1) a *	15.7 (10.5) a *
	H10	56.6 (3.8) a	53.0 (4.0) a	50.1 (4.3) a	42.7 (4.9) a *	31.6 (9.4) a *	11.7 (13.8) ab *

Means followed by different small letter in the column for each composite represent statistical significant difference (5%). * means that the relation Hx/H0 was lower than 0.8 (x = 1, 2, 3, 4, or 5).

Table 5: Knoop hardness means and standard deviation for Z250 and Esthet-X composite photoactivated using LED, in several depths.

Composite	Group	Surface (H0)	1 mm (H1)	2 mm (H2)	3 mm (H3)	4 mm (H4)	5 mm (H5)
Z250	**L1**	72.9 (3.4) a	71.8 (4.8) a	69.7 (5.0) ab	64.3 (7.1) ab	54.8 (9.5) a *	30.4 (8.4) b *
	L2	76.2 (2.4) a	74.5 (2.6) a	73.3 (2.4) a	70.1 (3.5) a	59.7 (5.6) a *	38.0 (5.9) a *
	L3	75.2 (2.2) a	74.3 (3.4) a	72.0 (5.1) ab	69.5 (3.6) a	60.6 (4.1) a	43.1 (6.8) a *
	L4	71.9 (2.0) a	69.1 (5.2) a	67.7 (5.1) ab	63.7 (6.1) ab	53.4 (5.2) a *	29.6 (4.9) b *
	L5	68.7 (5.0) a	68.1 (4.6) a	64.7 (4.5) b	58.1 (5.7) b	42.3 (9.2) b *	13.7 (9.8) c *
ESTHET-X	**L6**	58.6 (1.9) a	56.0 (2.6) a	53.0 (1.6) a	46.4 (4.7) ab *	20.9 (7.8) bc *	2.8 (3.9) a *
	L7	58.1 (2.7) a	55.4 (1.8) a	51.8 (2.8) a	45.0 (3.3) abc *	24.0 (14.8) ab *	3.1 (8.9) a *
	L8	57.7 (2.5) a	55.0 (2.0) a	51.1 (2.3) a	47.2 (2.6) a	31.1 (10.8) a *	4.3 (6.3) a *
	L9	57.5 (1.8) a	54.2 (2.4) a	49.7 (2.9) a	38.8 (3.5) c *	15.2 (9.6) c *	0 (0.0) a *
	L10	57.2 (1.4) a	54.3 (1.9) a	49.6 (2.5) a	39.4 (6.8) bc *	17.1 (13.3) bc *	0.4 (1.3) a *

Means followed by different small letter in the column for each composite represent statistical significant difference (5%). * means that the relation Hx/H0 was lower than 0.8 (x = 1, 2, 3, 4, or 5).

Table 6: Knoop hardness means and standard deviation for Z250 and Esthet-X composite photoactivated using PAC, in several depths.

Composite	Group	Surface (H0)	1 mm (H1)	2 mm (H2)	3 mm (H3)	4 mm (H4)	5 mm (H5)
Z250	**P1**	72.5 (3.9) a	68.1 (3.8) a	64.7 (4.6) a	52.6 (5.3) b *	24.1 (4.6) c *	0.0 (0.0) b *
	P2	74.1 (3.6) a	72.3 (3.8) a	68.2 (4.2) a	59.0 (7.2) ab	40.9 (9.0) ab *	5.0 (5.9) b *
	P3	71.8 (4.1) a	70.9 (4.6) a	68.3 (3.0) a	61.3 (4.7) a	47.3 (9.1) a *	12.8 (13.9) a *
	P4	74.9 (3.0) a	72.5 (2.2) a	69.0 (3.5) a	59.5 (4.7) ab *	35.5 (4.6) b *	1.8 (5.2) b *
	P5	72.9 (4.8) a	69.9 (4.9) a	66.4 (4.0) a	56.2 (3.0) ab *	34.1 (6.5) b *	0.0 (0.0) b *
ESTHET-X	**P6**	56.8 (4.1) a	54.1 (4.0) a	45.8 (5.7) a	20.2 (8.8) b *	0.4 (1.2) b *	0.0 (0.0) a *
	P7	59.2 (1.8) a	56.1 (2.3) a	50.3 (4.4) a	33.7 (8.5) a *	11.0 (9.6) a *	0.0 (0.0) a *
	P8	58.1 (3.1) a	54.5 (1.1) a	50.4 (2.7) a	37.4 (9.8) a *	14.4 (12.2) a *	0.0 (0.0) a *
	P9	57.7 (2.2) a	54.8 (2.6) a	48.6 (5.4) a	30.1 (13.3) a *	7.8 (8.4) ab *	0.0 (0.0) a *
	P10	56.8 (3.4) a	54.2 (3.4) a	47.0 (2.7) a	33.5 (11.9) a *	9.1 (10.3) a *	0.0 (0.0) a *

Means followed by different small letter in the column for each composite represent statistical significant difference (5%). * means that the relation Hx/H0 was lower than 0.8 (x = 1, 2, 3, 4, or 5).

CONSIDERAÇÕES GERAIS

Com o surgimento dos compósitos fotoativados, o método de fotoativação passou a ser uma das principais etapas para que o procedimento restaurador obtivesse sucesso. Após a fotoativação, o compósito deve apresentar alto grau de conversão e propriedades físicas e biológicas adequadas. Como foi mostrado neste estudo, o método de fotoativação tem influência direta nas propriedades finais dos compósitos. O profissional deve se preocupar com a fonte de luz, aferindo constantemente a intensidade luminosa emitida por seus aparelhos. O primeiro trabalho mostrou que a densidade energética fornecida ao compósito tem papel fundamental na dureza Knoop após a fotoativação. Para que o compósito obtenha grau de conversão adequado, densidades energéticas mínimas devem ser fornecidas pelas fontes de luz. O aumento do tempo de exposição não afetou os valores de dureza Knoop quando foi utilizada luz de lâmpada halógena para fotoativação. Isso pode se dar devido à densidade energética de 14 J/cm^2 ter sido suficiente para que o compósito atingisse altos valores de conversão. Já para os LEDs e para o arco de plasma de xenônio, o aumento da densidade energética produziu compósitos com maiores valores de dureza Knoop, provavelmente devido a maiores graus de conversão dos compósitos.

Dentre as fontes de luz utilizadas, os aparelhos que utilizam luz de lâmpada halógena e os LEDs apresentaram os melhores resultados. Os aparelhos de luz de lâmpada halógena forneceram as maiores densidades energéticas comparadas as demais fontes de luz. Os LEDs, apesar de fornecerem aos compósitos densidade energética total inferior aos aparelhos de luz de lâmpada halógena, devido ao estreito espectro da luz emitida por esses dispositivos, possuem maior especificidade para ativar a canforoquinona, que é o fotoiniciador utilizado pelos dois compósitos deste estudo. Devido a essa maior especificidade, embora a densidade energética total fornecida pelos LEDs não ter sido alta, a densidade energética específica, ou seja, a densidade energética fornecida dentro do espectro de absorção da canforoquinona, pode ter sido semelhante à densidade energética específica fornecida pelos aparelhos de luz de lâmpada halógena. Devido a esse fator, os valores de dureza Knoop foram semelhantes. Já o arco de plasma de xenônio forneceu densidade energética muito baixa, não produzindo altos valores de conversão e propriedades mecânicas máximas.

O segundo estudo mostrou que até 2 mm de profundidade, independente da intensidade de luz, não houve diferença nos valores de dureza Knoop, quando a mesma densidade energética foi mantida para a mesma fonte de luz. Como já foi dito anteriormente, o profissional constantemente deve aferir a intensidade luminosa emitida pelos aparelhos fotoativadores. Com o tempo, há a degradação desses dispositovos, diminuindo a intensidade luminosa. Intensidades luminosas menores podem ser compensadas por tempos de exposição prolongados, desde que os incrementos de compósito não ultrapassem 2 mm.

Neste estudo, os LEDs apresentaram resultados que não diferiram daqueles apresentados pelos aparelhos de luz de lâmpada halógena. A vantagem dos LEDS é o maior tempo de vida útil desses dispositivos. Os cuidados que se deve tomar é com relação aos fotoiniciadores utilizados nos materiais odontológicos, pois devido ao estreito espectro luminoso emitido por esses aparelhos, a ativação de fotoiniciadores que absorvem luz fora do espectro dos LEDs não ocorre. Os materiais conseqüentemente apresentarão propriedades insatisfatórias. Já para o arco de plasma de xenônio, tempos de exposição prolongados são necessários para que o compósito atinja propriedades semelhantes comparados aos compósitos fotoativados por luz de lâmpada halógena ou LED.

CONCLUSÕES GERAIS

Com base nos resultados obtidos nos dois estudos, pôde-se concluir que:

1. O compósito Z250 apresentou valores de dureza Knoop estatisticamente superiores ao do compósito Esthet-X. O aumento do tempo de exposição produziu compósitos com maiores valores de dureza Knoop quando se utilizou LED ou PAC. O aumento do tempo de exposição não interferiu nos valores de dureza Knoop dos compósitos fotoativados por luz halógena. Os compósitos fotoativados por PAC apresentaram valores de dureza Knoop estatisticamente inferiores àqueles dos compósitos fotoativados por luz halógena e LED, que não diferiram entre si.
2. Não houve diferença estatisticamente significativa nos valores de dureza Knoop entre as combinações de tempo de exposição e intensidade de luz até 2 mm de profundidade, com exceção do compósito Z250 fotoativado por 160 segundos com intensidade luminosa de 55 mW/cm^2. A partir de 3 mm, os compósitos fotoativados por tempo de exposição e intensidade de luz intermediários apresentaram os maiores valores de dureza Knoop. Os compósitos apresentaram polimerização adequada até 2 mm de profundidade quando fotoativados por LED e PAC, e até 3 mm quando fotoativados por luz halógena.

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* 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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APÊNDICES

Quadro 1. Análise de Variância quatro fatores, referente ao Capítulo 1.

Causas da Variação	GL	SQ	QM	Valor F	Prob >F
Material	1	30788.4211093	30788.4211093	2647.5394	0.00001
IntXTempo	2	772.4862483	386.2431241	33.2136	0.00001
Foto	2	930.0499725	465.0249862	39.9881	0.00001
Região	2	19135859075	956.7929538	82.2760	0.00001
MatxIntxFoto	4	55.9200431	13.9800108	1.2022	0.30870
MatxIntxRegião	4	7.5695181	1.8923795	0.1627	0.95464
MatxIntxRegião	4	38.5628973	9.6407243	0.8290	0.50927
IntxfotoxRegião	8	59.5981843	7.4497730	0.6406	0.74517
MatxIntxFotoxRegião	8	40.7958428	5.0994804	0.4385	0.89784
Resíduo	396	4605.1117314	11.6290700		
Total	431	39212.1014545			

Quadro 2. Análise de Variância três fatores, fixando o fator luz halógena, referente ao Capítulo 2.

Causas da Variação	GL	SQ	QM	Valor F	Prob >F
Material	1	64454.3172992	64454.3172992	2074.1292	0.00001
IntXTempo	4	842.8980818	210.7245204	6.7811	0.0001
Região	5	81605.6777809	16321.1355562	525.2114	0.00001
MatxInt	4	153.8534906	38.4633727	1.2377	0.29337
MatxRegião	5	4321.3970047	864.2794009	27.8124	0.00001
IntxRegião	20	518.6056118	25.9302806	0.8344	0.67223
MatxIntxRegião	20	676.8034557	33.8401729	1.0890	0.35756
Resíduo	420	13051.6524831	31.0753631		
Total	479	165625.2052077			

Quadro 3. Análise de Variância três fatores, fixando o fator LED, referente ao Capítulo 2.

Causas da Variação	GL	SQ	QM	Valor F	Prob >F
Material	1	59209.6343120	59209.6343120	1830.3718	0.00001
IntXTempo	4	5096.6753434	1274.1688359	39.3889	0.00001
Região	5	144213.4866065	28842.6973213	891.6262	0.00001
MatxInt	4	1361.8607552	340.4651888	10.5249	0.00001
MatxRegião	5	4934.3066619	986.8613324	30.5072	0.00001
IntxRegião	20	1764.4674150	88.2233708	2.7273	0.00021
MatxIntxRegião	20	1104.9799724	55.2489986	1.7079	0.02920
Resíduo	420	13586.3364103	32.3484200		
Total	479	231271.7474767			

Quadro 4. Análise de Variância três fatores, fixando o fator arco de plasma de xenônio, referente ao Capítulo 2.

Causas da Variação	GL	SQ	QM	Valor F	Prob >F
Material	1	39541.5984667	39541.5984667	1154.8027	0.00001
IntXTempo	4	2962.9175417	740.7293854	21.6328	0.00001
Região	5	258114.0614533	51622.8122907	1507.6316	0.00001
MatxInt	4	145.5477182	36.3869295	1.0627	0.37490
MatxRegião	5	7623.3272862	1524.6654572	44.5275	0.00001
IntxRegião	20	2354.8278320	117.7413916	3.4386	0.00001
MatxIntxRegião	20	871.1988009	43.5599400	1.2722	0.19266
Resíduo	420	14381.2195957	34.2409990		
Total	479	325994.6986947			

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Ribeirão Preto, 05 de março de 2005.

Prezado Professor,

O trabalho BDJ 740 Effect of the energy density increase on Knoop hardness of dental composites light-cured by QTH, LED and xenon plasma arc**, Américo Bortolazzo Correr, Mário Alexandre Coelho Sinhoreti, Lourenço Correr Sobrinho, Rubens Nisie Tango, Luis Felipe Jochims Schneider, Simonides Consani**, foi aceito no mérito científico para publicação.

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Enviado em: sexta-feira, 11 de fevereiro de 2005 11:25

Para: sinhoret@fop.unicamp.br

Assunto: Acta Odontologica Scandinavica 971

Dear Dr Sinhoreti,

Today I have received your manuscript entitled "Effect of exposure time versus irradiance relationship on Knoop hardness of dental composites" to be considered for publication in *Acta Odontologica Scandinavica*.

The typescript, in accordance with the usual formalities, has been dispatched to the reviewers and a further communication will be sent to you in due course.

Yours Sincerely,

Folke Lagerlöf
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