



UNIVERSIDADE ESTADUAL DE CAMPINAS
Faculdade de Odontologia de Piracicaba



MARIA DO CARMO AGUIAR JORDÃO MAINARDI

**AVALIAÇÃO DO GRAU DE CONVERSÃO E
MICRODUREZA KNOOP DE UM CIMENTO
RESINO DUAL SUBMETIDO A DIFERENTES
PROTOCOLOS DE FOTOATIVAÇÃO**

Monografia apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, como requisito para a obtenção do Título de Especialista em Dentística Restauradora

Orientador: Prof. Dr. Flávio Henrique Baggio Aguiar

FICHA CATALOGRÁFICA ELABORADA POR
MARILENE GIRELLO – CRB8/6159 - BIBLIOTECA DA
FACULDADE DE ODONTOLOGIA DE PIRACICABA DA UNICAMP

Mainardi, Maria do Carmo Aguiar Jordão, 1981-

M284a Avaliação do grau de conversão e microdureza Knoop de um cimento resino dual submetido a diferentes protocolos de fotoativação / Maria do Carmo Aguiar Jordão Mainardi. -- Piracicaba, SP : [s.n.], 2012.

Orientador: Flávio Henrique Baggio Aguiar.

Trabalho de Conclusão de Curso (especialização) –
Universidade Estadual de Campinas, Faculdade de
Odontologia de Piracicaba.

1. Dentística. 2. Canal radicular. 3. Polimerização. 4.
Dureza. I. Aguiar, Flávio Henrique Baggio, 1977- II.
Universidade Estadual de Campinas. Faculdade de
Odontologia de Piracicaba. III. Título.

RESUMO

O objetivo deste estudo foi avaliar a influência do tempo de espera, irradiância, tempo de fotoativação e terço do canal radicular no Grau de Conversão (GC) e microdureza Knoop (KHN) de um cimento resinoso dual. Para isso foram confeccionadas 78 amostras (n=6) de RelyX ARC (3M-ESPE) com o auxílio de uma matriz de teflon com 13mm de comprimento e 2mm de diâmetro. Os grupos foram submetidos a uma das seguintes densidades de energia (J/cm²): 7 (350mW/cm² e 20s), 14 (700mW/cm² e 20s), 20 (1000mW/cm² e 20s) e 28 (700mW/cm² e 40s) e tempos de espera: imediato, 1 minuto e 2 minutos. O grupo controle não recebeu fotoativação. Esta foi feita através do dispositivo LED de terceira geração Valo (Ultradent). Após 24 horas, o GC e KHN foram medidos. Os dados foram submetidos à ANOVA três fatores e Teste de Tukey. O teste de Dunnett foi utilizado para comparações com o grupo controle. Resultados de GC não mostraram interações entre os fatores. O segmento cervical obteve os maiores valores estatisticamente diferentes dos outros segmentos, e o segmento apical, os menores, sendo esses similares ao controle. Para KHN houve tripla interação entre os fatores e em todas as condições experimentais o segmento cervical foi estatisticamente diferente do controle. Conclui-se que é possível reduzir o tempo de fotoativação de um cimento resinoso dual se um dispositivo fotoativador de alta irradiância (1000mW/cm²) for utilizado com tempo de espera imediato ou de 1 minuto, chegando a resultados similares àqueles com maior tempo de fotoativação.

Palavras chave: dureza, polimerização, cimento resinoso, grau de conversão, canal radicular.

ABSTRACT

The aim of this study was to evaluate the influence of delay time, irradiance and time light-activation, and the third of the root canal on the degree of conversion (DC) and Knoop microhardness (KHN) of a dual resin cement. For this study, 78 samples (n=6) of RelyX ARC (3M-ESPE) were made with the aid of a matrix with 13 mm in length and 2mm in diameter. The groups were submitted to one of the following energy densities (J/cm²): 7 (350mW/cm² and 20s), 14 (700mW/cm² and 20s), 20 (1000mW/cm² and 20s) and 28 (700mW/cm² and 40s). Delay times were immediate, 1 minute, or 2 minutes. One group only underwent the chemical curing that was used for control. The light curing was made with a 3rd generation LED device (Valo-Ultradent). After 24 hours, the DC and KHN were measured. Data were submitted to ANOVA three factors and Tukey Test. The Dunnett Test was used for comparisons with the control. Results of DC did not show interaction among the factors. The cervical segment obtained the high value, which was significantly different from the others, and the apical obtained the lowest, being similar to the control. For KHN, there was triple interaction among the factors, and at all experimental conditions, the cervical segment was statistically different from the control. In conclusion, it is possible to reduce the light-activation of a dual resin cement, since a high irradiance light curing unit (1000 mW/cm²) is used with a short delay time (0 or 1 minute), achieving similar results to a long light-activation time.

Key words: hardness, polymerization, resin cement, degree of conversion, root canal

SUMÁRIO:

INTRODUÇÃO _____	6
CAPÍTULO ÚNICO _____	9
CONCLUSÃO _____	22
REFERÊNCIAS _____	23

INTRODUÇÃO

Os cimentos resinosos tem sido extensivamente utilizados na cimentação de restaurações indiretas e pinos de fibra de vidro¹, proporcionando uma adesão confiável, além de garantir boas propriedades mecânicas, baixa solubilidade, maior resistência ao desgaste², melhores propriedades de manuseamento e boas características estéticas³, comparado ao tradicional cimento de fosfato de zinco. Para cimentos de ativação física, a intensidade de luz durante a fotoativação pode ser fortemente atenuada pela absorção e dispersão de luz pelo material restaurador e também pela distância da fonte fotoativadora. Sendo assim, os cimentos resinosos duais foram desenvolvidos, com o objetivo de contornar os problemas relacionados à deficiência de luz⁴, combinando as propriedades desejáveis da ativação química e física⁵.

A principal vantagem da ativação química é proporcionar um adequado tempo de trabalho e garantir a polimerização em regiões nas quais a intensidade de luz é reduzida. Por outro lado há maior risco de incorporação de bolhas de ar durante a manipulação⁶. Já os cimentos com ativação física possuem um tempo de trabalho controlado⁷, e logo após a fotoativação a restauração pode ser finalizada sem a necessidade de tempo de espera⁶ (Hoffmann *et al.*, 2001).

A reação de polimerização de materiais a base de resina ocorre através da formação de radicais livres, transformando o material de um estado viscoso para um material rígido. Isto ocorre através da conversão de ligações alifáticas C=C em ligações simples C-C entre os monômeros metacrilatos⁸, formando os polímeros. O mecanismo de ativação dos cimentos resinosos duais ocorre através da canforoquinona e do peróxido de benzoíla, responsáveis pela ativação física e química, respectivamente. Quando as duas pastas são manipuladas e submetidas à fotoativação, há a formação de radicais livres através dos dois modos, sendo que em áreas com deficiência de luz, a formação de radicais pela reação entre peróxido de benzoíla e amina terciária, compensa a deficiência daqueles formados pela fotoativação, contribuindo para a manutenção do grau de conversão nessas regiões⁹.

Alto grau de conversão está relacionado a boas propriedades físicas e mecânicas do material e a baixa conversão está relacionada a cáries secundárias, sorção de água, baixa resistência de união e propriedades mecânicas comprometidas¹⁰. Tem sido pesquisado os efeitos do tempo de espera para a fotoativação no grau de conversão de cimentos resinosos duais. Seria sugerido que a fotoativação imediata poderia prejudicar a polimerização química do cimento pelo entrapamento dos radicais livres, formados pela ativação química, na matriz polimérica formada logo após a fotoativação¹¹. Faria-e-Silva¹² *et al* em 2011 analisaram o grau de conversão de três cimentos resinosos duais, incluindo o RelyX ARC, com fotoativação imediata e após cinco minutos, e encontrou que para este cimento não houve diferenças estatísticas entre os resultados.

Um método de se medir indiretamente o grau de conversão é o teste de dureza¹³. A dureza constitui uma das mais importantes propriedades dos materiais e está relacionada com a qualidade do polímero formado. Materiais a base de metacrilato podem formar uma matriz com alta densidade de ligações cruzadas, na qual um grande número de ligações covalentes entre diferentes cadeias tornam o material rígido e com alto peso¹⁴. O desenvolvimento de uma matriz polimérica com alta densidade de ligações cruzadas pode ser afetado pela velocidade com que ocorre a polimerização. Uma rápida polimerização causaria maiores quantidades de radicais livres e centros de crescimento resultando em um polímero com maior densidade de ligações cruzadas. Porém um material com alto grau de conversão pode apresentar uma estrutura polimérica linear, constituindo uma menor dureza¹⁵. Segundo Iryama³ *et al*, em 2009, valores de dureza de cimentos resinosos duais submetidos à fotoativação são maiores que os valores daqueles que sofrem somente ativação química.

Os cimentos resinosos duais com três modos de ativação (física, química e dual), podem produzir diferentes estruturas poliméricas que variam na densidade de ligações cruzadas, as quais podem afetar as propriedades mecânicas finais do polímero¹⁴. Sendo assim, o objetivo deste trabalho foi avaliar o grau de conversão e a microdureza Knoop de um cimento resinoso dual, submetido a diferentes densidades de energia e tempos de espera para a fotoativação.

CAPÍTULO ÚNICO

EVALUATION OF DEGREE OF CONVERSION AND KNOOP MICROHARDNESS OF A DUAL RESIN CEMENT SUBMITTED TO REDUCED TIME OF PHOTOACTIVATION

Abstract

The aim of this study was to evaluate the influence of delay time, irradiance and time light-activation, and the third of the root canal on the degree of conversion (DC) and Knoop microhardness (KHN) of a dual resin cement. For this study, 78 samples (n=6) of RelyX ARC (3M-ESPE) were made with the aid of a matrix with 13 mm in length and 2mm in diameter. The groups were submitted to one of the following energy densities (J/cm²): 7 (350mW/cm² and 20s), 14 (700mW/cm² and 20s), 20 (1000mW/cm² and 20s) and 28 (700mW/cm² and 40s). Delay times were immediate, 1 minute, or 2 minutes. One group only underwent the chemical curing that was used for control. The light curing was made with a 3rd generation LED device (Valo-Ultradent). After 24 hours, the DC and KHN were measured. Dates were submitted to ANOVA three factors and Tukey Test. The Dunnett Test was used for comparisons with the control. Results of DC did not show interaction among the factors. The cervical segment obtained the high value, which was significantly different from the others, and the apical obtained the lowest, being similar to the control. For KHN, there was triple interaction among the factors, and at all experimental conditions, the cervical segment was statistically different from the control. In conclusion, it is possible to reduce the light-activation of a dual resin cement, since a high irradiance light curing unit (1000 mW/cm²) is used with a short delay time (0 or 1 minute), achieving similar results to a long light-activation time.

Key words: hardness, polymerization, resin cement, degree of conversion, root canal

Introduction

The growth of demand for indirect tooth-colored restorations in endodontically treated teeth has increased the use of fiber posts (Ho et al. 2011).¹ Dual and self-cured resin cements are recommended for their cementation in intraradicular environments. Light curing resin cements are not advised for this application because there is an inadequate cure in depth regions of the root (Radovic et al. 2009).²

Dual resin cements combine the desirable characteristics of light curing and chemical curing resin cements (Ozyesil et al. 2004),³ providing proper polymerization in either the presence or absence of light (Faria-e-Silva et al. 2011)⁴. The self-curing component is useful because it favors the conversion in the absence of radiant energy, mainly in dark regions, which the light did not reach. However it has the disadvantage of requiring the mixture of two pastes, increasing the incorporation of bubbles and voids (Acquaviva et al. 2009).⁵ The light-curing component is advantageous because it provides control of working time. Although both modes of activation are present in dual resin cements, they are complementary and independent (Pedreira et al. 2009).⁶ Furthermore, some dual resin cements are dependent on light-activation, so an inadequate degree of conversion (DC) must occur if the cement was not light cured (Faria-e-Silva et al. 2007).⁷

It is being suggested that the immediate light curing of dual resin cements is associated with lower final DC (Pereira et al. 2010).⁸ The reason is that immediate light curing causes the rapid formation of cross-linked polymers chains, entrapping unreacted monomers and free radicals into the polymeric network and jeopardizing the chemical curing. Also, the curing reaction that occurs at the same time by the self- and light-polymerization may decrease the necessary time to reach the vitrification phase (Moraes 2009),⁹ perhaps making activation by light for a prolonged time unnecessary, especially if there is a delay between manipulating the resin luting agent and the light-activation. Therefore, the effects of delays in light curing on the ultimate DC of dual resin cements are being widely studied. An adequate DC is determinant of good physical and mechanical properties. Poor monomer conversion leads to low bond strength, high water sorption, and compromised physical properties (Bueno et al. 2011).¹⁰

Another method of measuring the degree of polymerization of dual resin cements is the hardness testing (Sinhoreti et al. 2007).¹¹ Hardness is one of the most important properties of dental materials (Cekic-Nagas et al. 2010)¹² and is used as an indirect measurement of the degree of conversion. But this approach can be a problem (Marchan et al. 2011)¹³ because the resulting polymer, which is dependent on polymerization mode, can differ in polymeric structure, even if the degree of conversion is the same (Aguiar et al. 2005).¹⁴

Dual resin cements, with their three modes of activation (light, chemical, and dual curing), may produce different polymers structures that vary in cross-linked density, which can affect the ultimate mechanical properties (Meng et al. 2008).¹⁵ Thus, the aim of this study is to evaluate the influence of delayed activation, energy density (varying irradiance and light-activation time), and third of root on the degree of conversion and microhardness of a dual resin cement. The null hypothesis is that the degree of conversion and microhardness are not affected by these factors.

Materials and Methods

Seventy eight samples simulating root canals with 2mm in diameter and 13mm in length were made and randomly divided into 13 groups (n=6). For this, the dual resin cement RelyX ARC, shade A1, was manipulated during 10 s and inserted in a bipartite teflon matrix. The light curing was performed immediately, either one minute or two minutes after the insertion of the resin cement in the matrix, with the LED light source Valo (Ultradent), while one group underwent only chemical curing as a control. All the samples were exposed to one of the following energy densities (J/cm²): 7 (350 mW/cm² and 20s), 14 (700mW/cm² and 20s), 20 (1000 mW/cm² and 20s) and 28 (700 mW/cm² and 40s). These irradiances were gathered through spacers previously prepared in a pilot study and were monitored by a radiometer. After 24 hours, the samples were sectioned in three segments with double-sided diamond discs, denominated cervical (C), medium (M), and apical (A), with each measuring 4 mm in length. The samples were carried to Fourier Transform Infrared Spectroscopy (FTIR) for measurements of DC on the top surface and after the Koop hardness (KHN) was recorded on the same surface.

The composition of the resin cement is listed in Table 1.

Table 1. Composition of the resin cement

Resin Cement	Composition	Shade	Filler load (%) by volume
Rely X ARC (3M ESPE, St. Paul, MN, USA)	<p>Paste A – Bis-GMA*, TEGDMA**, dimethacrylate polymer, amine and CQ***.</p> <p>Paste B – Bis-GMA, TEGDMA, dimethacrylate polymer, and BPO****.</p>	A1	67.5

*Bisfenol glycidyl dimethacrylate, **Triethyleneglycol dimethacrylate.

Camphorquinone, *Benzoyl peroxide (Faria-e-Silva et al. 2011).

Degree of Conversion Measurement

The irradiated and non-irradiated surfaces were examined through Fourier Transformed Infrared Spectroscopy (FTIR) (Spectrum 100, PerkinElmer, São Paulo, SP, Brazil) to conversion's degree evaluation. The spectrum of light cured and non-light cured resin cement were obtained through an attenuated total reflectance crystal (ART) using 16 scans at a resolution of 4cm⁻¹. The absorbance peak at 1638 cm⁻¹, corresponding to aliphatic C=C bonds, and peak at 1608 cm⁻¹, corresponding to aromatic C=C bonds, were used as internal standards for calculating the percentage of remaining double bonds of carbon in the resin cement after this polymerization (Ferracane and Greener 1984). The computation of this percentage was performed with the following formula (Noronha-Filho et al. 2010)¹⁶:

$$DC(\%) = 100 * [1 - R_{\text{polymerized}} / R_{\text{unpolymerized}}],$$

where R = peak at 1638 cm⁻¹ / peak at 1608 cm⁻¹.

Microhardness Measurement

The samples of each group were fixed in a poliesterene resin and then grifted in a polishing machine (Arotec), using sanding discs with grits #600 and #1200, in that order. After this, the microhardness at top of each sample was measured using a Knoop hardness test (Shimadzu, Japan), under 20g for 10s. Three indentations were made for each sample. The largest diagonal of the lozenge formed by the indentation was measured to gather KHN.

Results

Results of DC are presented in Table 2. ANOVA did not show either triple interactions among the factors ($p=0,4559$) or double interactions between time x energy density ($p=0,8152$) and time x segment ($p=0,28180$ and segment x energy density ($0,4935$). There were significant differences among the segments ($p<0,0001$). These differences were not found for energy densities ($p=0,1698$) and times ($p=0,3132$). Tukey test showed that the cervical segment gathered the higher values, which were statistically different from others, and the apical, the lowest for all experimental conditions. Comparisons with the control group, performed by Dunnett test, showed that the apical segment was not statistically different from the control group for all experimental conditions. The cervical segment showed significant differences from the control group, except for the density $7\text{J}/\text{cm}^2$ with time immediate and 1 minute. The medium segment showed no significant differences from the control except for the density $14\text{J}/\text{cm}^2$ with time immediate and density $20\text{J}/\text{cm}^2$ with time 1 minute.

Results of KHN are presented in Table 3. The ANOVA showed triple interaction among the factors ($p<0,001$). For the time immediate, there were no statistical differences among the segments in all densities except for the apical segment at $14\text{J}/\text{cm}^2$, which showed microhardness values that were significantly different from the others. Significant differences were found in the apical segments compared to the others at densities 7 and $14\text{J}/\text{cm}^2$, with the time of 1 minute. Comparisons among the segments at all densities and time of 2 minutes showed that the apical segment obtained the lowest value, except at the density $28\text{J}/\text{cm}^2$, at which

this segment did not show significant differences from the others. Comparing the densities for the cervical segment at the same time, the density 20J/cm² presented the higher values, which were significantly different from the others. For the apical segment, the higher values were obtained from 28J/cm², with statistical differences from the others. For the density 20J/cm² and cervical segment, the time of 2 minutes showed higher values compared to the values obtained by times of 1 minute and immediate. The same time presented the highest values from the apical segment with density 28J/cm². The Dunnett test showed significant differences comparing the cervical segments and the control, which presented the lowest values for all experimental conditions. The medium and apical segments partially differed from the control, since for some experimental conditions there were no significant differences between the groups.

Table 2: Results of DC

Time(s)	Segment	Energy density (J/cm ²) and Standard Deviation			
		7	14	20	28
0	Cervical	78,41(8,87)a	*80,07(2,35)a	*81,64(1,04)a	*80,79(1,07)a
	Medium	77,71(3,82)b	&80,20(2,85)b	79,74(2,09)b	78,72(1,63)b
	Apical	73,81(9,43)c	75,41(4,63)c	74,92(2,42)c	75,78(0,90)c
1	Cervical	78,74(3,18)a	*80,85(2,41)a	*81,55(1,68)a	*80,36(1,32)a
	Medium	77,93(2,26)b	77,87(2,88)b	*81,72(3,92)b	79,34(1,66)b
	Apical	76,42(3,44)c	72,83(9,91)c	76,60(1,62)c	76,00(1,72)c
2	Cervical	*81,55(6,47)c	*80,65(2,13)a	*84,63(5,82)a	81,06(1,23)a
	Medium	79,98(6,68)b	79,16(2,47b)	79,12(3,34)b	79,60(1,24)b
	Apical	76,36(3,64)c	74,34(2,21)c	77,01(2,15)c	76,78(0,88)c
Control	Cervical	74,36(5,85)			
	Medium	74,74(9,03)			
	Apical	75,00(5,91)			

Medium with different low case letters differ statistically. *Differ from control on Cervical segment. &Differ from control on Medium segment.

Table 3: Results of KHN

Time(s)	Segment	Energy Density (J/cm ²) and Standard Deviation			
		7	14	20	28
0	Cervical	\$45,35(1,56)Aa	\$46,19(2,13)Aa	\$46,56(1,77)Aa	\$46,42(1,49)Aa
	Medium	44,78(1,87)Aa	\$46,76(3,25)Aa	44,30(1,33)Aa	43,20(1,71)Aa
	Apical	42,06(2,01)Aa	41,31(1,77)Ab	42,35(1,88)Aa	\$44,09(2,26)Aa
1	Cervical	\$48,76(3,32)Aa	\$48,65(1,87)Aa	\$44,59(2,49)Aa	\$47,36(2,91)Aa
	Medium	\$48,94(1,68)Aa	44,10(2,41)Aab	44,57(2,53)Aa	\$47,07(1,80)Aa
	Apical	42,48(1,46)Ab	\$43,21(1,93)Ab	41,81(0,66)Aa	\$42,94(1,90)Aa
2	Cervical	\$45,94(1,74)Ba	\$49,53(0,94)Ba	\$*55,81(2,75)Aa	\$49,75(2,76)Ba
	Medium	45,85(1,34)Aa	44,37(3,42)Ab	45,25(2,27)Ab	\$46,44(2,24)Aa
	Apical	39,61(0,89)Bb	40,04(1,96)Bb	42,59(1,25)Bb	\$&48,47(4,43)Aa
Control	Cervical	39,51(1,22)			
	Medium	42,10(0,31)			
	Apical	39,48(0,50)			

Different low case letters in the same column differ statistically; Different upper case letters in the same row differ statistically; [§]Differ from control on the same segment; ^{*}Differ from time immediate and 1 minute on Cervical segment; [&]Differ from time 1 minute on Apical segment;

Discussion

The time of photoactivation recommended by the manufacturer of RelyX ARC is 40 seconds. This luting agent, like other dual resin cements, combines physical and chemical activation because these modes are complementary (Pedreira et al. 2009)⁶. Chemical activation starts as soon the two pastes are manipulated, and physical activation occurs after photoactivation (Pick et al. 2010).¹⁷ It is not entirely known if a delay time for light curing affects the final polymerization and therefore the ultimate DC and KHN of dual resin cements, since non-reactive monomers and free radicals may be entrapped on the matrix formed by the chemical activation. Because

both modes occur in this polymerization process, the present study evaluated the reduction by half of the time of photoactivation associated with some delay times on physical properties of this cement. So the question is: Does the chemical curing compensate for the low time of light exposure in maintaining of the physical properties of dual resin cement?

In this study, the null hypothesis for DC was partially accepted, since it was influenced by the third of the root but not by energy density and delay time. The cervical segment achieved the higher values at all experimental conditions and the apical the lowest, being statistically similar to the control. The dual resin cements have a complementary chemical and light curing mechanism, and an adequate DC is crucial for overall clinical success of the final restoration (Tezvergil-Mutluay 2007).¹⁸ However, at depth regions of the root, light fails to reach because of both the increase in the distance to the tip of the light source and the absorption and scattering of light by the cement (Faria-e-Silva et al. 2011).⁴ It is recognized that light transmittance in resin-based materials decreases in logarithmic proportion with the increasing depth, decreasing the DC and cross-linked densities in this region (Leprince et al. 2011).¹⁹ The results indicate that the apical segment in the present study mainly underwent the chemical curing part of the polymerization process, regardless of other factors. Another study by Faria-e-Silva et al. in 2007 that evaluated the DC of RelyX ARC in different depths of the root using translucent and opaque posts found statistical differences among the thirds of the root, with the apical segment having the lowest values. Zorba et al.²⁰ also found statistical differences with a push-out test among the thirds of the root, with the apical segment having the lowest results, suggesting that even with high doses of energy, the scattering and absorption of light by the cement makes it difficult for light to reach to the depth region of the root. In the present study, low power density associated with reduced time of photoactivation, obtaining similar values to that a high power density associated with the time of photoactivation recommended by the manufacturer for the same segments. This emphasizes the importance of chemical activation in the final polymerization of resin cements.

There were no significant differences in DC with varying delay times. This result is in accordance with a previous study conducted by Faria-e-Silva et al.⁴ that

analyzed the kinetic and degree of conversion of three dual resin cements, including RelyX ARC. The DC achieved for this cement with immediate light curing and with a delay time of 5 minutes was similar, without statistical differences. This could be attributed to dependence on light of some dual resin cements to reach a proper DC (Moraes et al. 2009).⁹ This is due to a higher ratio of photoinitiators in the composition of these cements, so the chemical polymerization that occurred in the delay period was not sufficient to form a rigid structure that would cause entrap monomers and free radicals that hinder light curing.

Other results for DC showed that the cervical segment for energy density 7J/cm² and delay times immediate and 1 minute did not differ from the control. This can be explained by the low dose of energy delivered by the light source, indicating that the light curing was not sufficient to promote an increase in DC. However, with the delay time of 2 minutes, the results showed a statistical difference from the control. With this delay time, the self polymerization had already started and the polymeric structure formed by this mode of polymerization contributed to an increase of the ultimate DC in combination with the light curing.

High DC is not always associated with high KHN in polymeric dental materials. High KHN is also related to high cross-linked density in the polymeric matrix, composition of the material such as the amount of filler, matrix composition, shade, and translucency (Vignolo et al. 2012).²¹ Rapid light curing promotes multiple growth centers, providing a larger number of covalent bonds among different chains. A slow rate of polymerization, such as the chemical curing in dual resin cements, results in fewer growth centers, forming a more linear polymeric structure and decreasing the KHN. Thus, polymers with similar DC may have different cross-linked densities and therefore different KHN (Soh et al. 2004).²² This can be correlated with the results of the present study since the decrease found in DC with the increase in the depth of the root was not always followed by a decrease in KHN under the same experimental conditions.

For the KHN, the null hypothesis was rejected because it was influenced by the third of the root, energy density, and delayed time. With no delay time, there were no significant differences between the segments and the energy densities in almost all situations. For the energy density 20J/cm² and the high power density

(1000mW/cm²), even when associated with a reduced time of photoactivation (20s), was sufficient to promote a deep light curing, providing formation of a high number of growth centers by exciting camphorquinone and forming more free radicals in the deepest region of the cement. The same can be said for the energy density 28J/cm² with a high power density (700mW/cm²) associated with 40s of photopolymerization, while for 7J/cm² and no delay time, the low dose of energy associated with a low time exposure was sufficient to promote a KHN on the cervical segment that was statistically different from the control, but statistically similar to the deepest segments. Sinhoret et al. in 2007 found different results with RelyX ARC, in which the top surface had the highest KHN value and the bottom surface had the lowest, being statistically similar to the control.

For delay time of 1 and 2 minutes, the apical segment obtained a lower KHN value than the others, except for 28J/cm² and 20J/cm² and delay time 1 minute. However, for the delay time 1 minute, this result was statistically different from the control, demonstrating that the polymeric structure formed by chemical polymerization prior to light curing was essential to help form a matrix with a higher cross-linked densities, thus increasing the KHN. Even under a low dose of energy, dual resin cements may have a larger number of free radicals formed by self curing that become entrapped in the matrix. Although these radicals did not promote an increase on the DC, they can improve the cross-linked densities by combining with double links of methacrylate groups (Meng et al. 2008).¹⁵

Taking into account the cervical segment for delay time 2 minutes, the KHN was higher for 20J/cm² than the other energy densities. This is probably due to its high intensity of light (1000mW/cm²), which causes a larger number of photons to reach the camphorquinone and form free radicals. More free radicals promote the formation of greater number of growth centers, producing a more cross-linked polymeric matrix. Furthermore, the cervical segment for all experimental conditions was statistically different from the control, reinforcing the importance of light and fast polymerization to the establishment of a harder polymer. However, the apical segment obtained the worst results for some experimental conditions. Therefore, the reduction of the time of photoactivation for 20 seconds is possible if it is done with a powerful light source and a delay time of 1 minute.

Conclusions

Based on the data gathered in the present study, it was concluded that the depth of root canals can influence the DC and KHN of dual resin cements, and KHN can be also influenced by delay time and energy density. Furthermore, it is possible to reduce the time of light activation if it is done using a high irradiance curing unit associated with a delay time of 0 or 1 minute.

References:

1. Ho YC, Lai YL, Chou IC, Yang SF, Lee SY. Effects of light attenuation by fibre posts on polymerization of a dual-cured resin cement and microleakage of post-restored teeth. **J Dent**, 2011 ;39: 309-15.
2. Radovic I, Corciolani G, Magni E, Krstanovic G, Pavlovic V, Vulicevic Z, Ferrari M. Light transmission through fiber post: The effect on adhesion, elastic modulus and hardness of dual-cured resin cement. **Dent Mater**, 2009; 25: 837-44.
3. Ozyesil AG, Usumez A, Gunduz B. The efficiency of different light sources to polymerize composite beneath a simulated ceramic restoration. **J Prosthet Dent**, 2004; 91: 151-57.
4. Faria-e-Silva A, Boaro L, Braga R, Piva E, Arias V, Martins L. Effect of immediate or delayed light activation on curing kinetics and shrinkage stress of dual-cure resin cements. **Oper Dent**, 2011; 36(2): 196-04.
5. Acquaviva PA, Cerutti F, Adami G, Gagliani M, Ferrari F, Gherlone E, Cerutti A. Degree of conversion of three composite materials employed in the adhesive cementation of indirect restorations: A micro-Raman analysis. **J Dent**, 2009; 37: 610-15.
6. Pedreira APRV, Pegoraro LF, de Góes MF, Pegoraro TA, Carvalho RM. Microhardness of resin cements in the intraradicular environment: Effect of water storage and softening treatment. **Dent Mater**, 2009; 25: 868-76.

7. Faria-e-Silva A, Arias VG, Soares LE, Martin AA, Martins LRM. Influence of fiber-post translucency on the degree of conversion of a dual-cured resin cement. **J Endod**, 2007; 33(3): 303-05.
8. Pereira SG, Fulgêncio R, Nunes TG, Toledano M, Osorio R, Carvalho RM. Effect of curing protocol on the polymerization of dual-cured resin cements. **Dent Mater**, 2010; 26: 710-18.
9. Moraes RR, Faria-e-Silva AL, Ogliari FA, Correr-Sobrinho L, Demarco FF, Piva E. Impact of immediate and delayed light activation on self-polymerization of dual-cured dental resin luting agents. **Acta Biomaterialia**, 2009; 5: 2095-100.
10. Bueno ALN, Arrais CAG, Jorge ACT, Reis AF, Amaral CM. Light-activation through indirect ceramic restorations: does the overexposure compensate for the attenuation in light intensity during resin cement polymerization?. **J Appl Oral Sci**, 2011; 19(1): 22-7.
11. Sinhorette MAC, Maneta IP, Tango RN, Iriyama NT, Consani RLX, Correr-Sobrinho L. Effect of light-curing methods on resin cement Knoop hardness at different depths. **Braz Dent J**, 2007; 18(4): 305-08.
12. Cekic-Nagas I, Ergun G. Effect of different light curing methods on mechanical and physical properties of resin-cements polymerized through ceramic discs. **J Appl Dent**, 2011; 19(4): 403-12.
13. Marchan SM, White D, Smith WA, Raman V, Coldeiro L, Dhuru V. Effect of reduced exposure times on the microhardness of nanocomposites polymerized by QTH and second-generation LED curing lights. **Oper Dent**, 2011; 36(1): 98-03.
14. Aguiar FHB, Braceiro ATB, Ambrosano GMB, Lovadino, JR. Hardness and diametral tensile strength of a hybrid composite resin polymerized with different modes and immersed in ethanol or distilled water media. **Dent Mater**, 2005; 21: 1098-103.
15. Meng X, Yoshida K, Atsuta M. Influence of ceramic thickness on mechanical properties and a polymer structure of dual-cured resin luting agents. **Dent Mater**, 2008; 24: 594-99.

16. Noronha-Filho JD, Brandão NL, Poskus LT, Guimarães JGA, da Silva EM. A critical analysis of the degree of conversion of resin-based luting agents. **J Appl Dent**, 2010; 18(5): 442-6.
17. Pick B, Gonzaga CC, Junior WS, Kawano Y, Braga RR, Cardoso PEC. Influence of curing light attenuation caused by aesthetic indirect restorative materials on resin cement polymerization. **Eur J of Dent**, 2010; 4: 314-23.
18. Tezvergil-Mutluay A, Lassila LVJ, Vallittu PK. Degree of conversion of dual-cured luting agents light-polymerized through various materials. **Acta Odontol Scand**, 2007; 65 :201-05.
19. Leprince JG, Leveque P, Nysten B, Galle B, Devaux J, Leloup G. New insight into the “depth of cure” of dimethacrylate-based dental composites. **Dent Mater**, 2012.
20. Zorba YO, Erdemir A, Turkyilmaz A, Eldeniz AU. Effects of different curing units and luting agents on push-out bond strength of translucency posts. **J Endod**, 2010; 36(9): 1521-525.
21. Vignolo V, Fuentes MV, Garrido MA, Rodríguez J, Ceballos L. Microhardness of different resin shades inside the root canal. **Med Oral Patol Cir Bucal**, 2012.
22. Soh MS, Yap AUJ. Influence of curing modes on crosslink density in polymer structures. **J Dent**, 2004; 32: 321-26.

CONCLUSÃO:

Baseado nos dados obtidos neste estudo, conclue-se que a profundidade do canal radicular pode influenciar o grau de conversão e a microdureza Knoop de cimentos resinoso duais, e a microdureza Knoop pode ser influenciada pelo tempo de espera e densidade de energia aplicada. Ainda, o tempo de fotoativação pode ser reduzido, desde que se utilize um dispositivo fotoativador com alta irradiância e com tempo de espera “imediato” ou 1 minuto.

REFERÊNCIAS:

1. Aguiar TR, Francescantonio MD, Arrais CAG, GMB Ambrosano, C Davanzo, Giannini M. Influence of curing mode and time on degree of conversion of one conventional and two self-adhesive resin cements. **Oper Dent**, 2010; 35(3): 295-299.
2. Arrais CAG, Giannini M, Rueggerberg FA. Kinetic analysis of monomer conversion in auto- and dual-polymerizing modes of commercial resin luting agents. **J Prosthet Dent**, 2009; 101(2): 128-136.
3. Iriyama NT, Tango RN, Manetta IP, Coelho MA, Sobrinho LC, Saavedra GSFA. Effect of light curing method and indirect veneering materials on the Knoop hardness of a resin cement. **Braz Oral Res**, 2009; 23(3): 108-12.
4. Arrais CAG, Rueggeberg FA, Waller JL, de Goes MF, Giannini. Effect of curing mode on the polymerization characteristics of dual-cured resin cement systems. **J Dent**, 2008; 36: 418-26;
5. Yan YL, Kim YK, Kim K-H, Kwon T-Y. Changes in degree of conversion and microhardness of dental resin cements. **Oper Dent**, 2010; 35(2): 203-10.
6. Hofmann N, Papsthart G, Hugo B, Klaiber B. Comparison of photo-activation versus chemical or dual-curing of resin-based luting cements regarding flexural strength, modulus and surface hardness. **J Oral Rehab**, 2001; 28: 1022-28.
7. Pedreira APRV, Pegoraro LF, de Góes MF, Pegoraro TA, Carvalho RM. Microhardness of resin cements in the intraradicular environment: Effects of water storage and softening treatment. **Dent Mater**, 2009; 25: 868-76.
8. Noronha-Filho JD, Brandão NL, Poskus LT, Guimarães JGA, da Silva EM. A critical analysis of the degree of conversion of resin-based luting agents. **J Appl Dent**, 2010; 18(5): 442-6.
9. Pick B, Gonzaga CC, Junior WS, Kawano Y, Braga RR, Cardoso PEC. Influence of curing light attenuation caused by aesthetic indirect restorative materials on resin cement polymerization. **Eur J Dent**, 2010; 4: 314-23.

10. de Paula AB, Tango RN, Sinhoreti MAC, Alves MC, Puppim-Rontani RM. Effect of thickness of indirect restoration and distance from the light-curing unit tip on the hardness of a dual-cured resin cement. **Braz Dent J**, 2010; 21(2): 117-122.
11. Faria-e-Silva A, Boaro L, Braga R, Piva E, Arias V, Martins L. Effect of immediate or delayed light activation on curing kinetics and shrinkage stress of dual-cure resin cements. **Oper Dent**, 2011; 36(2): 196-04.
12. Moraes RR, Brandt WC, Naves LZ, Correr-Sobrinho L, Piva E. Light- and time-dependent polymerization of dual-cured resin luting agent beneath ceramic. **Acta Odont Scand**, 2008; 66: 257-61;
13. Sinhoreti MAC, Maneta IP, Tango RN, Iriyama NT, Consani RLX, Correr-Sobrinho L. Effect of light-curing methods on resin cement Knoop hardness at different depths. **Braz Dent J**, 2007; 18(4): 305-08.
14. Meng X, Yoshida K, Atsuta M. Influence of ceramic thickness on mechanical properties and a polymer structure of dual-cured resin luting agents. **Dent Mater**, 2008; 24: 594-99.
15. Valentino TA, Borges GA, Borges LH, Vishal J, Martins LRM, Correr-Sobrinho L. Dual resin cement Knoop hardness after different activation modes through dental ceramics. **Braz Dent J**, 2010; 21(2): 104-10.