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Orientador: Prof. Dr. Paulo Vinícius Soares

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Prof. Dr. Leandro Augusto Hilgert

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torna a vida interessante.

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RESUMO

O objetivo desse estudo foi avaliar a influência de aparelhos fotoativadores em cimentos resinosos fotopolimerizáveis de diferentes cores por meio do grau de conversão (DC), dureza de Knoop (HK) e módulo de elasticidade (E). Um disco cerâmico de dissilicato de lítio (0,5 mm de espessura X 12 mm de diâmetro) foi preparado e utilizado para atenuar a luz. Quatro cimentos resinosos em diferentes cores foram testados: Variolink Esthetic LC (cores L+ e W+, Ivoclar Vivadent), RelyX Veneer (cores B0,5, A1 e OW, 3M ESPE), NX3 Nexus LC (cores Yellow, Opaque White e Bleach, Kerr) e AllCem Veneer (cores A3, E-bleach e WO, FGM). Os discos de cimento foram posicionados em uma matriz (0,7 mm de espessura X 10mm de diâmetro) e fotoativados através do disco cerâmico por 5 aparelhos fotoativadores diferentes: Valo (Ultradent), Grand Valo (Ultradent), Elipar DeepCure (3M ESPE), Bluephase N (Ivoclar Vivadent) e Radii Xpert (SDI) (n=10). Após 24h do preparo da amostra, os discos de cimento foram avaliados quando ao grau de conversão, dureza de Knoop e módulo de elasticidade. As medidas foram realizadas nas áreas central e periférica da amostra. Foi realizada análise da cor e da opacidade de cada cor de cimento, afim de caracterizar e relacionar essas propriedades com as demais testadas. As medidas da área central foram analisada por Análise de Variância a dois fatores, seguido pelo teste de Tukey. A comparação entre as medidas centrais e periféricas foram feitas com o teste T de Student. O teste de correlação de Pearson foi realizado para correlacionar opacidade, índice de brancura e grau de conversão, e para correlacionar grau de conversão e dureza de Knoop. Foi encontrada diferença estatística entre as cores do cimento e entre os aparelhos fotoativadores para todas as propriedade ($p<0,001$). A área central apresentou maiores valores do que a periférica para todos os cimentos fotoativados pelo Radii Xpert. Houve correlação positiva entre opacidade e grau de conversão ($p<0,001$; correlation coefficient = 0,453). Em conclusão, o aparelho fotoativador e a cor do cimento afetam as propriedades do cimento resinos fotopolimerizável. Apenas o aparelho Radii

Xpert apresentou resultados menores para todas as propriedades na área periférica.

Relevância Clínica: Os cimentos resinosos fotopolimerizáveis possuem diferentes composições e cores. A polimerização desses materiais é dependente do aparelho fotoativador e interfere no sucesso clínico da restauração.

PALAVRAS-CHAVE: gran de conversão, cimento resinoso, LED, dureza, análise colorimétrica.

ABSTRACT

The aim of this study was to evaluate the influence of light curing units of light-cured resin cements in different shades by degree of conversion (DC), Knoop hardness (KH) and elastic modulus (E). One lithium dissilicate ceramic disc (0.5mm thickness X 12mm diameter) was prepared and used to attenuate the light. Four resin cements were tested in different shades: Variolink Esthetic LC (shades L+ and W+, Ivoclar Vivadent), RelyX Veneer (shades B0.5, A1 and OW, 3M ESPE), NX3 Nexus LC (shades Yellow, Opaque White and Bleach, Kerr) and AllCem Veneer (shades A3, E-bleach and WO, FGM). The cement discs were made in a matrix (0.7mm tickness X 10mm diameter) and polymerizaded through a ceramic disc by five light curing units: Valo (Ultradent), Grand Valo (Ultradent), Elipar DeepCure (3M ESPE), Bluephase N (Ivoclar Vivadent) and Radii Xpert (SDI) (n=10). After 24h of preparation, the cement discs were evaluated for degree of conversion, Knoop hardness and elastic modulus. The measurements were made in the central and peripheral areas. The colorimetric analysis and opacity were measured for each cement shade to characterize and relate with the other material properties. The central measurements were analysed by ANOVA two-way and Tukey's test. The peripheral area were compare to the central with Student's T test. Pearson correlation test was performed to correlate degree of conversion and Knoop hardness, following by opacity, whiteness index and degree of conversion correlation. There was a statistic difference between the cement shades and the LCU for degree of conversion, Knoop hardness and elastic modulus ($p<0.001$). The central area presented higher properties than peripheral area for all cements polymerized by Radii Xpert. There was a positive correlation between opacity and degree of conversion ($p<0.001$; correlation coefficient = 0.453). In conclusion, the light curing unit and the cement shade affect the properties of light-cured resin cements. Only Radii Xpert showed lower values for all the properties in the peripheral area.

Clinical Relevance: Light-cured resin cements have different compositions and shades. The polymerization of this materials is dependent of the light curing unit and may affect the clinical success of restoration.

KEYWORDS: degree of conversion, resin cement, LED units, hardness, colorimetric analysis.

INTRODUÇÃO E REFERENCIAL TEÓRICO

Nos último anos a busca por tratamentos restauradores estéticos aumentou consideravelmente e devido às excelentes propriedades ópticas e estéticas, as cerâmicas odontológicas são muito utilizadas para esse tipo de tratamento restaurador. (Cekic & Ergun, 2011; Kesrak & Leevailoj, 2012; Lopes *et al.*, 2015; Moreno *et al.*, 2018) Com a evolução da Odontologia adesiva, restaurações cerâmicas conservadoras, ou também chamados de laminados cerâmicos, podem ser utilizadas para alterar formato, cor, tamanho e posicionamento dos dentes anteriores, promovendo um sorriso mais harmônico aos pacientes. (Novais *et al.*, 2017; Strazzi Sahyon *et al.*, 2018) Essas restaurações são realizadas com preparos minimamente invasivos, preferencialmente com término em esmalte, para preservar ao máximo a estrutura dentária. O sucesso dessas restaurações cerâmicas depende de uma união duradoura entre os substratos dentais, o cimento e o material restaurador. (Cekic & Ergun, 2011; Lopes *et al.*, 2015; Novais *et al.*, 2017)

Clinicamente, os cimentos resinosos têm sido muito utilizados para a cimentação não só de laminados cerâmicos, mas também de coroas, inlays, onlays e restaurações indiretas de resina composta. (Aykor & Ozel, 2009; Lopes *et al.*, 2015; Sampaio *et al.*, 2017) Esses cimentos possuem propriedades satisfatórias como baixa solubilidade, fácil manipulação, estética e adequada adesão tanto à cerâmica quanto aos tecidos dentários. (Lopes *et al*, 2015; Strazzi Sahyon *et al.*, 2018) Esse material pode ser classificado de acordo com o modo de ativação como químico (autopolimerizável), físico (fotopolimerizável) ou a combinação dos dois (dual). (Cekic & Ergun, 2011; Lopes *et al.*, 2015) A escolha de qual tipo de cimento resinoso é mais indicado para cada caso deve levar em consideração, principalmente, a espessura da restauração, que deve permitir suficiente passagem de luz para garantir a correta polimerização do material. (Martins *et al.*, 2019) Os cimentos fotopolimerizáveis são indicados para cimentação de laminados cerâmicos devido a fina espessura da restauração. Esses cimentos apresentam algumas vantagens clínicas como maior tempo de trabalho, o que permite a remoção dos excessos antes da polimerização, e maior estabilidade de cor se comparados aos cimentos cimentos químico e dual. (Sampaio *et al.*, 2017; Strazzi Sahyon *et al.*, 2018) Uma vez que a espessura da cerâmica em restaurações conservadores é extremamente fina, a cor do cimento pode interferir diretamente na cor da restauração, assim, a estabilidade de cor é uma propriedade muito importante para garantir a longevidade desses tratamentos. (Marchionatti *et al.*, 2017; Mina *et al.*, 2019) Além da espessura, a cor, o tipo e a opacidade das cerâmicas também podem influenciar na polimerização do cimento resinoso. (Bueno *et al.*, 2011; Flury *et al.*, 2013; Castellanos *et al.*, 2019; Martins *et al.*, 2019)

Além do material restaurador, características do aparelho fotoativador utilizado também podem interferir nas propriedades dos materiais resinosos. (Valentino *et al.*, 2010; Cekic & Ergun, 2011; Shimokawa *et al.*, 2016) Estudos prévios mostraram que a intensidade de luz, distância da ponta até a restauração, tamanho da ponta, modo de ativação, tempo de polimerização, espectro de luz e irradiância afetam a polimerização e podem causar um baixo grau de conversão do material. (Valentino *et al.*, 2010; Cekic & Ergun, 2011; Shimokawa *et al.*, 2016; Pereira *et al.*, 2016; Cardoso *et al.*, 2019) Existem no mercado diversas opções de aparelhos fotoativadores, sendo que os mais usados são os diodos emissores de luz (LEDs). (Cekic & Ergun, 2011; Jandt & Mills, 2013; Strazzi Sahyon *et al.*, 2018) Os LEDs podem ser classificados quando ao espectro de luz emitido entre *monowave*, que emite apenas o espectro da luz visível azul, e *poliwave*, que possui um espectro de luz amplo indo do violeta ao azul. (Rueggeberg *et al.*, 2017; Shimokawa *et al.*, 2018; Strazzi Sahyon *et al.*, 2018) Assim, a qualidade da fotoativação depende da compatibilidade entre a composição do cimento e o aparelho fotoativador utilizado. (Rueggeberg *et al.*, 2017; Strazzi Sahyon *et al.*, 2018)

Os cimentos resinosos são compostos por uma matriz polimérica, partículas de carga, pigmentos e fotoiniciadores. (Cekic & Ergun, 2011; Lopes *et al.*, 2015) Os fotoiniciadores são as moléculas responsáveis por iniciar o processo de polimerização quando ativadas por luz. Por um longo tempo, a canforoquinona foi o único fotoiniciador utilizado em materiais resinosos. Ela é ativada pelo comprimento de onda da luz azul (468 nm) e é utilizada em conjunto com aminas terciárias que atuam como coiniciador. (Meereis *et al.*, 2016; Oliveira *et al.*, 2016; Rueggeberg *et al.*, 2017; Shimokawa *et al.*, 2018; Strazzi Sahyon *et al.*, 2018) Esse sistema gera ótimos grau de conversão e propriedades mecânicas aos materiais resinosos. (Sheneider *et al.*, 2012; Meereis *et al.*, 2016; Oliveira *et al.*, 2016; Segreto *et al.*, 2016) Apesar do sistema canforoquinona associado às aminas ser efetivo para a polimerização, ele apresenta algumas desvantagens clínicas. A canforoquinona é um componente de cor amarelada, que, portanto, pode interferir na cor do material resinoso e dificultar a obtenção de cores mais claras que são muito utilizadas em tratamentos estéticos. (Sheneider *et al.*, 2012; Meereis *et al.*, 2016; Oliveira *et al.*, 2016; Segreto *et al.*, 2016) Além disso, as aminas terciárias não-reagidas diminuem a estabilidade de cor do material e aumentam o risco de irritação pulpar, diminuindo assim a biocompatibilidade do material. (Sheneider *et al.*, 2012; Meereis *et al.*, 2016; Oliveira *et al.*, 2016; Segreto *et al.*, 2016) Dessa forma, novos sistemas fotoiniciadores foram desenvolvidos com o objetivo de diminuir ou substituir a quantidade de canforoquinona nos materiais. Esses sistemas não necessitam de coiniciadores, porém, para serem polimerizados adequadamente, necessitam de aparelhos fotoativadores de grande espectro, como os LEDs poliwave. (Oliveira, 2016 *et al.*; Segreto *et al.*, 2016; Rueggebert *et al.*, 2017) Assim, tanto a cor quanto a composição dos cimentos fototopolimerizáveis podem influenciar nas suas propriedades mecânicas do material. (Cekic & Ergun, 2011; Lopes *et al.*, 2015)

As propriedades químicas, mecânicas e ópticas dos cimentos resinosos interferem diretamente no resultado final da restauração. A polimerização é dependente de muitos fatores e quando é inadequada gera um baixo grau de conversão que, por sua vez, prejudica essas propriedades. (Silami *et al.*, 2013; Lopes *et al.*, 2015; Novais *et al.*, 2017) O baixo grau de conversão diminui a adesão da restauração, as propriedades mecânicas e a estabilidade de cor, e aumenta a sorção e solubilidade do cimento resinoso. (Silami *et al.*, 2013; Lopes *et al.*, 2015; Novais *et al.*, 2017;) Além disso, a presença de monômeros residuais resultantes do processo de polimerização está relacionada com inflamações pulpares. (Lopes *et al.*, 2015; Novais *et al.*, 2017) Por esses motivos, avaliar maneiras para otimizar a polimerização dos cimentos resinosos é importante para estabelecer protocolos que melhorem o desempenho clínico do material e, consequentemente, aumentem a longevidade e o sucesso do tratamento.

CAPÍTULO 1

Influence of light curing unit on the mechanical and chemical properties of light-cured resin cements in different shades

Influence of light curing unit on the properties of light-cured resin cements

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ABSTRACT

The aim of this study was to evaluate the influence of light curing units of light-cured resin cements in different shades by degree of conversion (DC), Knoop hardness (KH) and elastic modulus (E). One lithium dissilicate ceramic disc (0.5mm thickness X 12mm diameter) was prepared and used to attenuate the light. Four resin cements were tested in different shades: Variolink Esthetic LC (shades L+ and W+, Ivoclar Vivadent), RelyX Veneer (shades B0.5, A1 and OW, 3M ESPE), NX3 Nexus LC (shades Yellow, Opaque White and Bleach, Kerr) and AllCem Veneer (shades A3, E-bleach and WO, FGM). The cement discs were made in a matrix (0.7mm tickness X 10mm diameter) and polymerizaded through a ceramic disc by five light curing units: Valo (Ultradent), Grand Valo (Ultradent), Elipar DeepCure (3M ESPE), Bluephase N (Ivoclar Vivadent) and Radii Xpert (SDI) (n=10). After 24h of preparation, the cement discs were evaluated for degree of conversion, Knoop hardness and elastic modulus. The measurements were made in both the central and peripheral areas of each disc. The colorimetric analysis and opacity were measured for each cement shade to characterize and relate with the other material properties. The central measurements were analysed by ANOVA two-way and Tukey's test. The peripheral area was compare to the central with Student's T test. A Pearson correlation test was performed to correlate degree of conversion and Knoop hardness, following by opacity, whiteness index and degree of conversion correlation. There was a statistical difference between the cement shades and the LCU for degree of conversion, Knoop hardness and elastic modulus ($p<0.001$). The central area presented higher properties than peripheral area for all cements polymerized by Radii Xpert. There was a positive correlation between opacity and degree of conversion ($p<0.001$; correlation coefficient = 0.453). In conclusion, the light curing unit and the cement shade affect the

properties of light-cured resin cements. Only Radii Xpert showed lower values for all the properties in the peripheral area.

Clinical Relevance: Light-cured resin cements have different compositions and shades. The polymerization of this materials is dependent of the light curing unit and may affect the clinical success of restoration.

KEYWORDS: degree of conversion, resin cement, LED units, hardness, colorimetric analysis.

INTRODUTION

With the evolution of adhesive dentistry, conservative ceramic restorations, like ceramic laminates veneers, can be used to change color, shape and position of anterior teeth and promote a natural and symmetrical smile with minimal tooth preparation.[1-5]. The laminate veneers have been popular since the beginning of the 1980's[6] and clinical studies show 90% survival rate in 5 years of follow up.[6, 7] These restorations are considered as minimally invasive treatments[8] and the success depends significantly on a durable bond between the dental substrates, the luting agent and the ceramic restoration material. [1, 2, 4, 8]

The bond strength of laminate veneers can be affect by both the dental substrate and luting agent.[7, 9] As a minimal invasive treatment, the preparation should be mostly in enamel and this characteristic increases the bond strength of the restoration.[7] In relation to the luting agent, light-cured resin cements have been the most widely used for ceramic veneers because of their improved color stability and that their longer working time allows for the removal of excess cement.[5, 9] Due the thickness (0.1 - 0.7mm) and the higher translucency of ceramic veneers, the resin cement shade may affect the final color and the long-term success of the restoration.[3, 10, 11]

The resin cements are composed of a polymeric matrix dimethacrylate base, pigments, a photoinitiator system and filler particles.[1, 2] The photoinitiator system most commonly used in resin cements is the camphorquinone.[12-14] This photoinitiator is a Norrish type II, which requires a co-initiator, such as tertiary amines, to react and initiate the polymerization.[12-14] Although in higher concentration this system increases degree of conversion and mechanical properties,[14-17] it can compromise aesthetics procedures since the camphorquinone is a yellow colored compound.[13, 18] Also non-reactive tertiary amines tending to cause discoloration over time and decrease the biocompatibility.[13, 14] Thus, alternative photoinitiator systems were developed to reduce the amount of camphorquinone and improve the esthetic of resin-based materials.[13, 18] (Lima, 2019; Castellanos, 2019) These Norrish type I systems (alternative photoinitiators) are based on diphenyl(2,4,6-trimethylbenzoyl)phosphin oxide (TPO), phenylpropanedione (PPD), bisacylphosphine oxide (BAPO) and a germanium-based photoinitiator (commercially known as Ivocerin).[12, 13, 15, 17-19]

To activate the alternative photoinitiators properly it is necessary to use light curing units (LCU) that emit a large spectrum of light (320 to 410nm).[19, 20] There are different types of LCUs and the most used are light emittance diode (LED) lights. The LEDs are either monowave (first and second generation) or poliwave (third generation).[1, 5, 21] The monowave have the emission spectrum close to camphorquinone absorption peak (467 nm) and the poliwave have a large spectrum range that can activate TPO, BAPO and PPD.[5, 19, 20] Moreover, the intensity of the light, distance of the tip from the material, tip size, activation mode, polymerization time, wavelength and irradiance of the LCU also affect the resin cement polymerization and can interfere on the clinical performance of the restoration.[1, 20, 22-25]

Considering each manufacture has developed distinct compositions to increase the clinical performance and esthetic of resin cements, the aim of this study was evaluate the influence of LCU in light-cured resin cements with different composition and shades by degree of conversion, Knoop hardness and elastic modulus. In addition, the colorimetric analysis and opacity were measured for each cement shade to characterize and relate with the other material properties. The tested null hypothesis was that different LCU do not interfere with the properties of different shades of light-cured resin cements.

METHODS AND MATERIALS

Preparation of ceramic specimen

One lithium dissilicate ceramic disc of high translucency (IPS e.max Press HT, A1, Ivoclar Vivadent, Schaan, Liechtenstein), 12 mm diameter with 0.5 tickness, was used to attenuate the light and simulate a ceramic veneer. One autopolymerizing polymethylmethacrylate (Dencrilay, Dencril, Pirassununga, SP, Brazil) resin pattern was made and used to produce the ceramic disc. The pressing procedures were prepared following the manufacture's instruction. After the pressing procedure, the ceramic disc was disinvested using 100 μm glass microspheres at 4 bar pressure, followed by cleaning in running water and drying with air. After disinvestment, the disc was finished using #600 grit silicon carbide papers and ultrasonic cleaning for 10min.

Preparation of resin cement samples

In this study, four light-cured resin cements were evaluated with the following shades: Variolink Esthetic LC (L+ and W+, Ivoclar Vivadent), RelyX Veneer (B0.5, A1 and OW, 3M ESPE, St. Paul, MN, USA), NX3 Nexus LC (Yellow, OW and Bleach, Kerr, Orange, CA, USA) and AllCem Veneer (A3, E-bleach and WO, FGM, Joinville, SC, Brazil). The material information are describe on Table 1. The resin cements were inserted into a stainless-steel matrix (10mm in diameter and 0.7 mm in thickness) with mylar strips on the bottom and top surfaces of the samples to obtain smooth surfaces (Fig. 1A). The diameter of the specimen simulated an upper central incisor size. The ceramic disc was placed on the top surface of a mylar strip. The same ceramic disc was used for all the samples. The light activation was performed with 5 different LEDs equipments (Table 2): Valo (Ultradent, Salt Lake City, UT, USA), Grand Valo (Ultradent), Elipar DeepCure (3M ESPE), Bluephase N (Ivoclar Vivadent) and Radii Xpert (SDI, Bayswater, Australia). All the light curing units were fully charged and the tip was positioned in the center of the sample in contact with the ceramic disc (Fig. 1A). The light curing process was performed in a dark room with neutral yellow light for 20 seconds for each sample. All the samples were stored at 37 °C under dry and dark conditions.

Degree of conversion

After at least 24h of the specimen preparation, the degree of conversion (DC) was evaluated by FTIR (Fourier transform infrared spectroscopy unit, Tensor 27, Bruker, Ettlingen, Germany) (n=10). The measurement was taken on the top surface in central and four equidistant peripheral areas of the same sample with the aim to compare the DC in both areas (Fig. 1B). The mean of peripheral areas were calculated by obtaining a single value. The percentage of aliphatic C=C (1638 cm^{-1}) and aromatic C=C (1608 cm^{-1}) for uncured and cured samples were measured to obtain the number of remaining carbon double bonds. The spectra of both cured and uncured specimens were obtained by scanning the samples 32 times at a resolution of 4cm^{-1} within the range from 1000 to 6000 cm^{-1}. In the software (OMINIC 6.1, Nicolet Instrument Corp, Madison, WI, USA) the spectra were subtracted from the background spectra, expanded and analysed in the interest area (from 1560 to 1670 cm^{-1}). The DC was calculated using a comparison between the peak area at 1639 cm^{-1} and the internal standard peak at 1609 cm^{-1}. The DC was calculated according to the following equation:

$$DC\ (\%) = \frac{1 - (\text{Cured aliphatic / Aromatic ratio})}{(\text{Uncured aliphatic / Aromatic ratio})} \times 100$$

Knoop microhardness and Elastic Modulus

After the DC analysis, the Knoop hardness (KH) were performed using a Microhardness tester (FM-700, Future Tech, Tokyo, Japan) under a load of 50g for 15s (n=10). Three indentations were made on the top surface in central and four equidistant peripheral areas of the same sample (Fig. 1B), resulting in fifteen indentations per sample. The mean of the peripheral areas were calculated in order to obtain a single value. For each indentation, the length of the large diagonal was measured, then the Knoop hardness was calculated based on this value. The average of the three measurements was calculated and used for each area. The elastic modulus was calculated according to the method described by Marshall *et al*. in 1982 that is based on elastic recovery of the material in the indentation walls after the removal of an applied load.[26] During the load application, the ratio between the longer (D) and shorter (d) diagonals is constant (D/d = 7.11). However, after the removal of the load, elastic recovery of the shorter diagonal occurs without affect the longer diagonal.[26, 27] The elastic recovery extend is dependent of the relation between Knoop Microhardness and the elastic modulus. The E was calculated with the following equation:

$$E = \frac{0.45 \times KH}{(0.140647 - d/D) \times 100}$$

Colorimetric Analysis and Opacity

The colorimetric analysis was performed with the aim of describing numerically color characteristics of each shade of cement. The color was measured with CIELab system, which describes the color in three axes (L*: luminosity; a*: green-red; b*: yellow-blue), using a handheld spectrophotometer (Ci6X X-Rite, Pantone, Grand Rapids, MI, USA) calibrated with the standard illuminant D65. The black-and-white standard plaque were used to calibrate the spectrophotometer and also served as standard backgrounds for the specimens during the measurements. For the color measurement, a white background was used. The samples were placed in a device to standardize the reading position. The color was measured three times in the center of each sample and the average was calculated. Based on CIELab system chroma (c), hue (h) and whiteness index (WI) were also calculated according to the following equations:[28, 29]

$$c = (a^{*2} + b^{*2})^{1/2}$$

$$h = \tan^{-1}(b^*/a^*)$$

$$WI = (0.511 \times L^*) - (2.24 \times a^*) - (1.100 \times b^*)$$

The opacity was measured using the spectrophotometer with black and white backgrounds. The relation between L*a*b* values in both backgrounds were calculated and the opacity was expressed in a percentage (%).

Statistical Analysis

All the data was tested to normality (Shapiro-Wilk). Degree of conversion, Knoop hardness and elastic modulus were individually analysed by analysis of variance (ANOVA) two-way using the central measures of each sample. The post hoc multiple comparisons were performed using Tukey's test. The comparison between the central and peripheral area for DC, KH and E were analysed by Student T test to each shade of cement and light curing unit. Pearson correlation test was used to analyse the correlation between degree of conversion and Knoop hardness, and between WI, opacity and degree of conversion. The software SigmaPlot 12.0 (Systat Software, San, Jose, CA, USA) was used for all analysis and the confidence interval was 95%.

RESULTS

The results of DC, KH and E of the central measure are shown in Tables 3, 4 and 5, respectively. The LCU and the resin cement shade affected the three evaluated properties ($p<0.001$). The Variolink cement shade L+ showed the highest degree of conversion results for all the LCUs ($p<0.001$) with values between 82.91 and 85.07%. All the shades of AllCem presented inferior results independent of the LCU used when compared to other cements. However, Elipar and Radii Xpert presented the lowest values for all shades of this cement and when there is a statistic difference between the shades, the shade OW has a lower degree of conversion (62.71 - 76.87%). For the NX3 cement, the shade Bleach presented the highest degree of conversion independent of the LCU used (76.02 - 77.37%).

There is no correlation between Knoop hardness and degree of conversion ($p=0.411$). The Variolink cement in both shades (L+ and W+) demonstrated lower hardness and no statistical difference between the two shades for all the LCUs tested (15.74 - 21.65). Valo light curing unit showed better hardness results for NBl (33.83) and Valo Grand showed highest values of hardness to all the shades of the RelyX cement. Radii Xpert and Bluephase LCUs presented higher hardness for RWO, 34.25 and 33.84 respectively. For the Variolink cement, in both shades, the highest values were found using Bluephase (L+: 21.10; W+: 21.65). In the resin cement AllCem, the lowest values were showed for Radii Xpert LCU (Bl: 22.41; OW: 23.26; A3: 24.84).

As in hardness, the Variolink in both shades also demonstrated lower elastic modulus values ($p<0.001$) for all the LCUs tested. The RelyX cement showed the highest elastic modulus results for the three shades independent of the LCU. The elastic modulus modulus results were similar to hardness results. For the RelyX, Variolink and NX3 resin cements, the light curing units Grand Valo, Bluephase and Elipar demonstrated highest values of elastic modulus, respectively. For all shades of AllCem cement, the lowest values were presented for Radii Xpert LCU.

The results of DC, KH and E comparing the central and peripheral areas are shown in Figure 2. The Radii Xpert light curing unit (Fig. 2.D) presented significant differences between the peripheral and central areas for all the shades and properties, wherein the central area showed better results than the peripheral. All the light curing units showed lowest degree of conversion results in the peripheral area in all the shades of AllCem cement. The peripheral area of NX3 cement also presented the lowest values of degree of conversion for the LCUs Valo Grand, Elipar, Radii Xpert and Bluephase in all shades, and for Valo only the shades Yellow and Bleach influenced the degree of conversion of the peripheral area.

The Table 6 describes the color data for all cements. NYe presented the lowest opacity value (46.30%) and the RWO the highest opacity value (86.05%). VW+ and AOW showed the lowest and highest WI values, respectively. There was a positive correlation between opacity and degree of conversion ($p<0.001$; correlation coefficient = 0.453) and no correlation was found between WI and DC ($p=0.429$). RelyX and Variolink cements, independent of the LCU used, presented higher conversion degree values for the most opaque shade, WO and L+, respectivaly. For the NX3 the lowest values were found with the most translucent color (NYe) for all light curing unit.

DISCUSSION

The null hypothesis of this study was reject because different light curing units affect the three evaluated properties of light-cured resin cements in different shades. The polymerization of resin cements can be influence for different factors related to the ceramic, cement, and light curing unit used.

Previous studies showed that characteristics of the ceramic, such as shade, thickness, opacity and type, can attenuate the light of the LCU and interfere with the polymerization of the resin cement.[30-32] A systematic review with meta-analysis showed that ceramics with more than 1.0mm of thickness drastically decrease the degree of conversion of light-cured resin cement.[31] So, the small thickness of the laminate ceramics do not significantly interfere on the light transmission, but allows the influence of the resin cement shade on the final color of restoration.[3, 10, 11, 33]

For the clinical success of the restoration, it is essential color match between the ceramic restoration and the adjacent dentition.[34, 35] Therefore, each brand developed a distinct strategy to obtain different shades of the light-cured resin cements.[35] It is known that the manufacture change the pigments in the cement and maintain the polymeric matrix and the filler characteristics to obtain different colors.[35] This study showed different results on the properties of the same cement in different shades, which corroborate with previous studies that evaluated the influence of the resin cement shade on the mechanical properties of the material.[32, 35] The Pearson correlation test found a correlation between degree of conversion and opacity, wherein high opacity resin cements have a higher degree of conversion. Some studies presented opposite results, that cements with higher translucency have higher degree of conversion. These studies explain that the translucency allows the light penetration through the resin cement disc which promotes higher degree of conversion.[32, 35] However, as it is known, the cement layer in ceramic veneers is very thin (0.15 - 0.32mm),[9, 36] so the properties of the top surface is more important than the depth of cure. The samples in the present study were not sanded prior to test, so the surface analysed was that of intimate contact with the ceramic, being closer to the clinical situation.

Other than pigment alterations to obtain different shades, it is possible that changes in the photoinitiator systems are also performed since camphorquinone is a yellow coloured compound and may interfere with the final color of resin cements.[14, 19] This may explain why in different shades of NX3 and AllCem resin cements presented better results with different light curing units. The photoinitiators used as an alternative to camphorquinone have presented advantages such as white coloration of the compound and a high reactivity which increases the polymerization without using an amine as a co-initiator.[12, 14, 19] These photoinitiators Norrish type II need a broad-spectrum light curing unit to be correctly polymerized,[19]. The polywave LEDs, such as Valo, Valo Grand and Bluephase used in this study, have a large spectrum and are better designed for these types of cements.[21] However, Elipar and Radii Xpert, monowave LEDs that emit only blue light spectrum, showed high degree of conversion values with the Variolink Esthetic cements, in both shades, which have only an alternative photoinitiator (Ivocerin) in composition. Previous studies showed that Ivocerin absorbs light in both the blue and violet spectrum[18, 19] and resin-based materials associated with this type of photoinitiator are related with higher degree of conversion.[12, 18, 37] In the study of Alkhudhairy *et al.,*[37] the cements Variolink Esthetic and NX3 Nexus presented 87.19 and 75.92% of conversion degree, respectively, values closer to those found in this study.

The present study demonstrated lower degree of conversion for all the shades of AllCem when polymerized by Elipar and Radii Xpert LCUs. These results are probably related with the photoinitiator system used, because both light curing units are monowave LEDs and may not properly activate some alternative photoinitiators, such as BAPO and TPO.[5, 19, 20] Another study using a monowave LED also showed lower results for the properties of AllCem Veneer.[38] Therefore, to increase the clinical performance of light-cured resin cements, it is important to determine different protocols for each shade and brand. However the information about the photoinitiator system is not available in the most technical product profiles, which makes it difficult to choose the correct light curing unit for each material.

The lowest degree of conversion value was observed in the AllCem shade OW with Radii Expert (62.71%). The literature describes that for resin-based materials, a degree of conversion of 60% is adequate to ensure good properties of the material.[39, 40] When these results are compared to other studies, the degree of conversion can be considered acceptable.[39, 40, 41] In 1986, two formulations of light-cured resin cements presented more than 70% of degree of conversion[42]. So with the evolution of dental materials and the development of new technologies, such as Ivocerin, it is important to reevaluate this information because it is possible to obtain higher degree of conversion and increase the clinical performance of resin-based materials, specially for the resin cements.

Other important properties of resin-based materials are hardness and elastic modulus.[2, 4, 43] These properties are related with the polymerization of the material, however, in the present study, a correlation between hardness and degree of conversion was not observed. In addition to monomer conversion, the hardness also may be affected by the resin cement composition.[2, 32] The monomer composition of the resin-based materials are related with the degree of conversion, since cements with Bis-GMA, TEGDMA and UDMA promote higher conversion of monomers than HEMA and Bis-EMA.[37, 38, 40] However, hardness and elastic modulus are also related with the particle size and filler amount.[9, 38, 44] . The cement Variolink presented higher degree of conversion (76.43 - 85.07) and lower hardness (15.74 - 21.65) and elastic modulus (4.54 - 6.44), while RelyX showed satisfactory values for the three properties (GC: 73.17 - 78.48; KH: 30.72 - 37.00; E: 8.34 - 10.04). The composition of both cements can explain these results (Table 1). The monomeric composition of Variolink and RelyX can promote higher degree of conversion, but in relation to inorganic composition, RelyX has a higher filler amount and particle size (66%; 0.6mm) while Variolink has only a filler amount of 38% and mean particle size of 0.1μm. So the Variolink inorganic particles characteristics explain why this cement presented lower hardness and elastic modulus results. Similar hardness results for all the cements were found by Nascimento *et al.*[38] (RelyX: 31.20 - 39.18; AllCem: 24.3 - 33.18), Oztuk *et al.*[35] (Variolink: 8.9 - 16-0) and Kesrak *et al.*[44] (NX3: 30.16).

The properties of the material are important to ensure clinical success, but it is also necessary to perform all clinical steps correctly. The clinical cementation of ceramic veneers is a long process with multiple steps.[3, 9] These type of restorations are performed on anterior teeth that usually have a large size, close to 10mm.[45] Previous studies showed that some LCU tips are not large enough to properly polymerize large restorations.[20, 46] The present study evaluated the influence of different LCU on the properties of resin cements in different areas of the same sample (central compare to peripheral area). The only LCU used with less than 9 mm of tip was the Radii Xpert (Table 2), which explain the results showed in this study. The Radii Xpert presented significant decrease in all three properties in the peripheral area of all cements and shades. Clinically, it is possible to perform more than one cycle of polymerization in different areas of the same restoration to compensate this disadvantage (at least 2 cycles of 20s).

The light-cured resin cements are the material most commonly used for ceramic veneers cementation.[1, 2, 9] However, since 2001 another options of materials was suggested for this type of clinical procedure.[7] The proposed materials are flowable and preheated composite resin restorative materials because they have shown color stability, better marginal adaptation and higher mechanical properties than light-cured resin cements.[9, 47, 48] In the present study, the resin cements presented satisfactory values for all the properties and comparable conversion degree results to flowable and preheated composites, that have a degree fo conversion range between 76.23 and 82.99%.[48] However, the hardness of the proposed materials are higher than the resin cements.[47] Therefore, considering the disadvantages, such as difficult color matching, due to the absence of try-in paste and higher volumetric shrinkage of preheated composite resins,[9, 47] these techniques are an option to cement laminate veneers, but the light-cured resin cements can promote the same clinical performance.

The aging of resin-based materials is constant in the oral environment[4, 8] and may lead to leaching of internal components, in addition to swelling and degradation of the polymeric matrix.[8] The degradation of the finish line of the light-cured resin cements at the ceramic veneer restoration margin may be accelerated by inadequate polymerization and lower mechanical properties that increase sorption and solubility.[4, 39] The exposure to food components, acid beverages, changes of temperature, chewing, saliva and biofilm also influence the cement line degradation[4, 8, 39]. These factors can be responsible for the failure of ceramic veneers restorations, because it increase the margin pigmentation and reduces the bond strength, causing debond of the restoration and secondary caries.[9, 39]

This is an *in vitro* study and considering the methodological limitations, the results should be interpreted with caution because it may not be significant to clinical performance of ceramic veneers restorations. In addition, the present study evaluated only light curing units with higher irradiance, so future studies should test light curing units in different conditions. In the future, it is also important evaluate bond strength and color stability over time of light-cured resin cements in different shades and compositions. Clinical studies are necessary to determine the influence of the materials and techniques used on the longevity of these types of restorations.

CONCLUSION

In conclusion, the light curing unit does affect the properties (degree of conversion, Knoop hardness and elastic modulus) of light-cured resin cements and the resin cement shade does influence the polymerization of the material. Also, the peripheral area can present lower properties than the central area when light curing units with a tip smaller than the restoration is used.

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TABLES AND FIGURES

Figure 1. Schematic diagram of the experimental setup. (A) Resin cement was inserted into the stainless-steel matrix between two mylar strips. The light curing unit was placed onto the center of the ceramic disc. The red line simulates a location marking. (B) Division of the central and peripheral (1,2,3 and 4) areas in the resin cement disc.

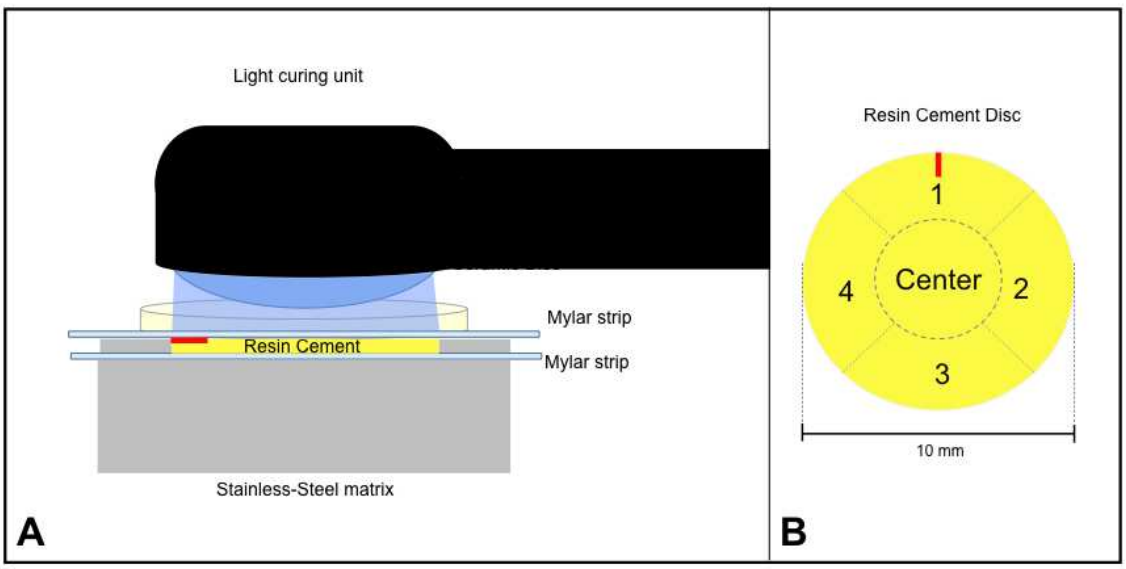

Table 1. Material used in this study.

Product	Manufacture	Shade	Sigle	Phoinitiator	Composition	Filler amount and particle size
RelyX Veneer	3M ESPE, St. Paul, Minessota, USA.	WO	RWO	Blue visible light activated system	Bis-GMA and TEGDMA polymer, zirconia/silica and fumed silica filler, pigments	66% 0.6mm
		B0.5	RB0.5			
		A1	RA1			
Variolink Esthetic LC	Ivoclar Vivadent, Shaan, Liechtenstein.	Light +	VL+	Ivocerin	UDMA and further methacrylate monomers, ytterbium trifluoride and spheroid mixed oxide, stabilizers, pigments.	38% 0.1µm
		Warm +	VW+			
NX3 Nexus LC	Kerr, Orange, California, USA.	WO	NWO	Proprietory redox initiator system	HEMA, uncured methacrylate, titanium dioxide pigments	Not found
		Bleach	NBl			
		Yellow	NYe			
AllCem Veneer	FGM, Joinville, Sta. Catarina,	OW	AOW	Advanced Polymerization System (APS),	Bis-EMA, TEGDMA and Bis-GMA, silanized barium-aluminum-silicate	Not found
		OW	ABl			

Product	Manufacture	Shade	Sigle	Phoinitiator	Composition	Filler amount and particle size
[illegible]	Brazil.	A3	AA3	camphorquino ne	filler particles and silicon dioxide.	

Abbreviations: Bis-GMA, bisphenol-A-diglycidylether dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; HEMA, hydroxyethylmethacrylate. Bis-EMA, ethoxylated bisphenol-A dimethacrylate.

Table 2. Light curing units (LCUs) used in this study.

LCU	Manufacture	Irradiance (mW/cm^2)	Diameter of tip (mm)	Emission Spectrum (nm)
Valo Cordless[22]	Ultradent, Salt Lake City, Utah, USA.	1000	9,5	395 - 480
Valo Grand[20]	Ultradent, Salt Lake City, Utah, USA.	1000	11,6	395 - 480
Elipar DeepCure[20]	3M ESPE, St. Paul, Minessota, USA.	1470	9,0	430 - 480
Radii Xpert[22]	SDI, Bayswater, Australia.	1500	7,8	440 - 480
Bluephase N[22]	Ivoclar Vivadent, Shaan, Liechtenstein.	1200	9,3	385 - 515

Table 3. Means (%) and standard deviation (SD) for degree of conversion of light-cured resin cements in different shades with different light curing units.

	Valo	Grand Valo	Elipar	Radii Xpert	Bluephase
RelyX WO	78.48 (0.68) Ab	77.82 (0.56) Bb	76.50 (0.60) Cbc	78.52 (0.49) Ab	76.55 (0.49) Cc
RelyX B0,5	76.88 (0.37) Bcd	75.22 (0.39) Be	75.67 (0.48) Bd	78.42 (0.62) Ab	75.45 (0.58) Bde
Rely X A1	75.48 (0.53) Ae	73.87 (0.47) Cf	75.19 (0.59) Abe	74.78 (0.43) Be	73.17 (0.55) Df
Variolink L+	83.57 (0.49) Ba	83.56 (0.64) Ba	82.91 (0.60) Ca	83.93 (0.65) Ba	85.07 (0.64) Aa
Variolink W+	78.31 (0.47) Bb	76.72 (0.45) Ccd	76.61 (0.39) Cb	76.43 (0.57) Ccd	79.18 (0.43) Ab
NX3 Bleach	77.37 (0.53) Ac	77.22 (0.45) Abc	76.38 (0.48) BCbd	76.94 (0.59) ABc	76.02 (0.57) Ccd
NX3 WO	76.28 (0.58) Ad	76.33 (0.57) Ad	75.87 (0.50) ABc	75.81 (0.57) ABd	75.37 (0.53) Bde
NX3 Yellow	74.99 (0.50) Be	75.02 (0.40) Be	75.91 (0.34) Abe	74.90 (0.46) Be	74.88 (0.54) Be
AllCem E-bleach	72.34 (0.52) BCf	72.74 (0.54) ABg	71.80 (0.51) Cf	69.02 (0.49) Df	73.17 (0.52) Af
AllCem OW	72.68 (0.54) Af	76.87 (0.42) Bi	65.72 (0.46) Ch	62.71 (0.43) Dg	67.49 (0.23) Bg
AllCem A3	71.98 (0.54) Af	71.01 (0.48) Bh	69.85 (0.43) Cg	69.83 (0.49) Df	72.47 (0.32) Af

Different letters represent significant differences ($p<0.005$). Capital letters compare LCU (row) and lowercase letters compare resin cements (column).

Table 4. Means (N/mm^2) and standard deviation (SD) for Knoop microhardness of light-cured resin cements in different shades with different light curing units.

	Valo	Grand Valo	Elipar	Radii Xpert	Bluephase
RelyX WO	31.72 (1.29) Cb	35.99 (1.30) Aab	30.91 (1.23) Cd	34.25 (0.91) Ba	33.84 (1.17) Ba
RelyX B0,5	30.72 (0.93) Cb	37.00 (1.08) Aa	33.86 (1.28) Bab	31.17 (1.12) Ccd	31.91 (1.44) Cb
Rely X A1	30.81 (0.65) Dbc	35.14 (1.51) Ab	34,77 (1.56) ABa	33.40 (1.23) Cab	33.75 (0.82) BCa
Variolink L+	15.74 (0.65) Ce	20.79 (0.76) Ae	17.52 (0.98) Bg	20.44 (0,75) Ag	21.10 (0.83) Ae
Variolink W+	16.98 (0.53) Ce	21.39 (0.75) Ae	16.66 (0.85) Cg	19.13 (1.06) Bg	21.65 (0.78) Ae
NX3 Bleach	33.83 (1.34) ABa	34.42 (0.98) Ab	32.18 (0.98) Ccd	29.91 (1.26) Dd	32.94 (1.23) BCab
NX3 WO	29.33 (1.30) Bc	30.99 (0.99) Ac	31.71 (1.29) Ac	31.83 (1.38) Abc	30.73 (1.06) Ac
NX3 Yellow	30.53 (1.43) Cb	30.66 (0.92) Cc	32.59 (1.40) ABbc	33.46 (1.45) Aa	32.08 (1.56) Bbc
AllCem E-bleach	26.29 (0.93) Bd	26.55 (1.08) Bd	25.98 (1.30) Be	22.41 (0.89) Cf	28.78 (0.39) Ad
AllCem OW	31.65 (1.28) Ab	27.86 (1.11) Bd	27.22 (1.23) Be	23.26 (0.99) Cef	27.60 (1.25) Bd
AllCem A3	30.75 (1.08) Ab	30.73 (1.29) Ac	24.05 (1.00) Cf	24.84 (0.78) Ce	27.71 (0.91) Bd

Different letters represent significant differences ($p<0.005$). Capital letters compare LCU (row) and lowercase letters compare resin cements (column).

Table 5. Means (MPa) and standard deviation (SD) for elastic modulus of light-cured resin cements in different shades with different light curing units.

	Valo	Grand Valo	Elipar	Radii Xpert	Bluephase
RelyX WO	9.03 (0.58) Aab	9.36 (0.60) Ab	8.34 (0.33) Ca	8.46 (0.51) BCa	8.99 (0.44) ABa
RelyX B0,5	9,55 (0.28) Aa	10.04 (0.58) Aa	8.96 (0.68) Ba	8.38 (0.64) Ca	8.72 (0.58) BCa
Rely X A1	9.35 (0.39) Aa	9.39 (0.57) Ab	8.98 (0.69) Aa	8.72 (0.51) Bab	9.00 (0.57) ABa
Variolink L+	4.61 (0.32) Bg	6.17 (0.23) Af	4.54 (0.26) Be	5.79 (0.36) Ae	6.18 (0.34) Ad
Variolink W+	4.86 (0.28) Bg	6.43 (0.48) Af	4.66 (0.34) Be	6.12 (0.34) Ade	6.44 (0.31) Ad
NX3 Bleach	8.48 (0.57) BCb	9.17 (0.40) Abc	8.37 (0.39) Ba	8.24 (0.28) Cb	8.81 (0.25) ABa
NX3 WO	7.30 (0.30) Be	8.49 (0.37) Ad	8.15 (0.64) Ab	8.60 (0.39) Aa	7.64 (0.51) Bc
NX3 Yellow	7.55 (0.40) Bde	8.52 (0.60) Acd	8.52 (0.37) Aab	8.93 (0.37) Aa	8.52 (0.54) Aab
AllCem E-bleach	6.87 (0.38) BCf	7.72 (0.34) Ae	7.35 (0.49) ABcd	6.55 (0.43) Cc	7.68 (0.48) Ac
AllCem OW	8.51 (0,47) Abc	8.01 (0.44) Abd	7.99 (0.26) Abc	6.69 (0.33) Ccd	7.90 (0.43) Bbc
AllCem A3	8.13 (0.53) ABcd	8.34 (0.53) Ade	6.94 (0.36) Cd	6.88 (0.38) Cc	7.58 (0.47) Bc

Different letters represent significant differences (p<0.005). Capital letters compare LCU (row) and lowercase letters compare resin cements (column).

Figure 2. Means for degree of conversion (DC), Knoop hardness (KH) and elastic modulus (E) of light-cured resin cements in different shades with different light curing units. The symbol * represents significant differences (p<0.005) between the peripheral and central areas.

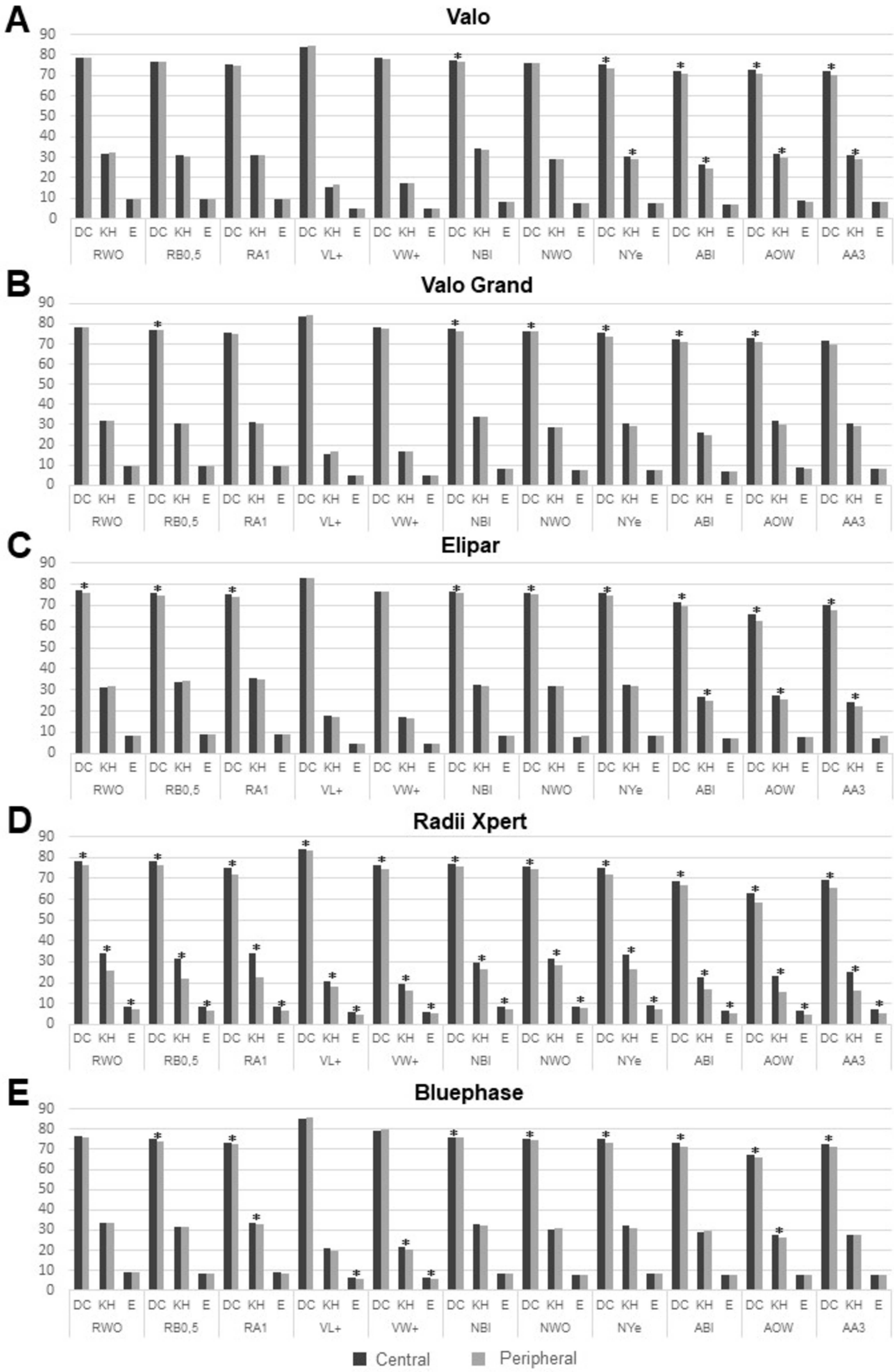

Table 6. Means for colorimetric values of light-cured resin cements in different shades.

Resin Cement	L	a	b	C	h	WI	Opacity (%)
RA1	77,65	-1,01	11,33	11,38	95,30	29,56	55,00
RB0.5	83,08	-0,30	8,13	8,13	92,13	34,21	67,80
RWO	87,45	-0,58	4,35	4,39	97,62	41,25	86,05
VL+	89,03	-0,74	4,11	4,18	100,23	42,69	78,31
VW+	70,11	5,33	25,85	26,39	78,36	-5,00	53,48
NYe	76,88	0,68	27,25	27,26	88,58	7,73	46,30
NBl	86,94	-3,82	10,65	11,32	109,74	41,59	72,59
NWO	88,65	-4,43	12,43	13,20	109,62	41,92	83,24
AA3	77,85	1,05	13,93	13,97	85,70	22,02	57,20
ABl	82,51	-1,78	4,28	4,64	112,60	41,59	48,46
AOW	85,50	-2,21	4,36	4,89	116,89	44,03	62,43

Abbreviations: C, chroma; h, hue; WI, whiteness index

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