Universidade Estadual de Campinas
Faculdade de Odontologia de Piracicaba

ROGÉRIO VIEIRA REGES

Cirurgião-Dentista

INFLUÊNCIA DA COR DE CIMENTOS RESINOSOS ATIVADOS POR DIFERENTES FONTES DE LUZ NA DUREZA KNOOP

Tese apresentada a Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas para obtenção do título de Doutor em Materiais Dentários.

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Orientador: Prof. Dr. Lourenço Correr Sobrinho – FOP/UNICAMP

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A Deus, por ser o guia e protegedor de cada passo dado, proporcionando a concretização dos objetivos da vida e das amizades realizadas.

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“Atrás de todas as coisas que julgamos impossíveis sempre existe uma luz que nos oferece esperança. E é através dessa luz que lutamos para conseguir o que sempre desejamos”

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RESUMO

O objetivo deste estudo foi avaliar a influência de duas fontes de luz sobre a dureza Knoop dos cimentos resinosos Variolink II, nas cores XL transparente – (A1), A3, A2 e Opaco e Enforce B1, A2 e Branco Opaco, nos período imediato e 24 horas após a polimerização. Corpos-de-prova com 5mm de diâmetro por 1mm de espessura dos cimentos resinosos Variolink II (Ivoclar-Vivadent) e Enforce (Denstisply) foram preparados num molde de teflon, cobertos com uma tira de poliéster e disco de cerâmica feldspática Duceram na espessura de 2,0 mm e fotoativados por 40 segundos com aparelho XL 2500 (3M), com 615 mW/cm^2 e 40 segundos com o aparelho LED – Ultrablue Is (D.M.C.) com 610 mW/cm^2 . A dureza Knoop foi efetuada no aparelho HMV-2, com carga de 50 gramas por 15 segundos, imediatamente e após armazenagem por 24 horas a 37^o C. Doze penetrações foram feitas em cada corpo-de-prova para o cimento resinoso Enforce, sendo que cinco corpos-de-prova foram confeccionados para cada cor do cimento e cinco para o grupo controle (sem cerâmica) em cada tempo de armazenagem, totalizando 60 corpos-de-prova. Para o cimento Variolink II, quatro penetrações foram feitas em cada corpo-de-prova, sendo que cinco corpos-de-prova foram confeccionados para cada cor do cimento em cada condição experimental, totalizando 80 corpos-de-prova. Os dados foram submetidos à análise de variância e ao teste de Tukey (5%) e mostraram que o cimento resinoso Variolink II opaco apresentou o menor valor de dureza Knoop em relação as outras cores do cimento, para as duas fontes de luz e dois períodos de ativação (mediato e 24 horas). O aparelho fotoativador de lâmpada halógena apresentou valores de dureza Knoop estatisticamente superior em relação ao aparelho LED para as cores do cimento A2, A3 e opaco para a condição mediato e XL, A2 e A3 após 24 horas. Quando os dois métodos de fotoativação foram comparados para a mesma cor do cimento, os aparelhos fotoativadores com lâmpada halógena e LED (24 horas) apresentaram valores de dureza Knoop estatisticamente superiores aos valores fotoativados na condição mediato. A regressão linear positiva da dureza Knoop foi estabelecida em função da profundidade de fotoativação da amostra do cimento resinoso (Enforce) pela fotoativação direta e indireta pela interposição da cerâmica. O cimento resinoso Enforce na cor opaca mostrou valores de dureza Knoop estatisticamente inferior às outras cores do cimento. A dureza Knoop mediata mostrou valores de dureza Knoop na região de topo (100 µm) estatisticamente superior em relação a dureza na região de fundo (700 µm).

Palavra-chave: Cimento resinoso, aparelhos fotoativadores, dureza Knoop, cor, cerâmica.

ABSTRACT

The aim of this study was to evaluate the influence of two light sources in Knoop hardness values of resin cement Variolink II in the shade XL transparent (A1), A3, A2, and Opaque and Enforce B1, A2 and Opaque White, in the immediate time and 24 hours after polymerization. The specimens with 5.0 mm in diameter and 1.0 mm in thickness of resin cements Variolink II (Ivoclar-Vivadent) and Enforce (Dentisply) were made in the Teflon mould, covered with a Mylar strip and fledspatic ceramic Duceram Plus (DeguDent) disc 2.0 mm thick and photoactivated for 40 seconds with halogen light unit XL 2500 (3M), with intensity of 615 mW/cm^2 and 40 seconds with LED –Ultrablue Is (D.M.C.) with power density of 610 mW/cm^2. Knoop hardness was measured using HMV-2 (Shimadzu), with a 50g load for 15 seconds, immediately stored at 37^o C for 24 hours. Twelve indentations were made in each specimen for the resin cement (Enforce). Five specimens were made for each color and five specimens for the control group (without ceramic) in each stored time, totaling sixty specimens. For the resin cement Variolink II, four indentations were made in each specimen, and five specimens were made for each one in each stored time, totaling eighty specimens. The data were submitted to variance analysis and Tukey's test (5%) and showed that the opaque shade of the resin cement had lower Knoop hardness value in relation to the other shades for both light sources and both activation periods (mediate and 24 hours). The halogen units showed Knoop hardness values higher than LED's for A2, A3 and opaque for mediate and XL, A2 and A3 after 24 hours. When the photoactivation methods were compared for the same shade halogen and LED (24 hours) showed higher Knoop hardness values than mediate photoactivation. Knoop hardness positive linear regression is a function of depth of specimen of resin cement direct and indirectly photoactivated. Opaque shade showed KHN statistically inferior to the others resin cement groups. Mediate KHN reading showed that Knoop hardness near top (100 µm) is statistically different from bottom surface (700 µm).

Key-words: Resin cement, light source, Knoop hardness, shade, ceramic.

INTRODUÇÃO GERAL

Cimentos resinosos são materiais de primeira escolha para cimentação ou fixação de restaurações cerâmicas.[13] O sucesso de restaurações cerâmicas depende em grande parte do cimento resinoso utilizado, para garantir união efetiva entre o material restaurador e estrutura dentária, proporcionando boa adaptação marginal.[15,17]

De acordo com a normatização da ISO 4049 (The International Organization for Standardization), todos os cimentos resinosos para fixação ou cimentação de peças protéticas pertencem ao Tipo 2 de materiais restauradores à base de polímeros. Ainda, os cimentos resinosos são divididos em classes, de acordo com o tipo de ativação; Classe 1 – Cimentos autopolimerizávies (self-cured) quando a polimerização é iniciada pela mistura de um iniciador e um ativador; Classe 2, cimentos fotoativados, ou seja, quando a energia é fornecida a partir de fonte foto ativadora para uso intra-bucal (photo-cured) e Classe 3 para cimentos de dupla ativação, química e foto (dual-cured).

Cimentos resinosos de dupla ativação possuem os co-iniciadores, peróxido e amina, encontrados também nos compósitos restauradores quimicamente ativados, e adicionalmente um fotoiniciador (canforoquinona), que também está presente nos compósitos restauradores fotoativados.[16]

Apesar da variedade de cimentos disponíveis atualmente, não há um cimento que seja ideal para todas as situações clínicas.[18] A escolha do agente de cimentação, para cada condição clínica, deve ser baseada nas características físicas, biológicas e de manipulação do agente para cimentação frente aos fatores relacionados ao remanescente dentário preparado e a peça protética a ser cimentada.

Inlays, onlays, facetas laminadas e coroas de cerâmicas livres de metal são comumente fixadas com cimentos resinosos de dupla ativação. A vantagem da escolha de cimento ativado quimicamente está baseada no processo de polimerização, que acontecerá mesmo em áreas onde a exposição à luz é crítica. Neste contexto, o período de ativação químico irá, teoricamente, garantir reação de presa satisfatória em locais de difícil acesso à luz. Entretanto, a fotoativação desses materiais é um fator essencial na reação de polimerização, melhorando de modo geral as propriedades mecânicas e físicas dos cimentos resinosos de dupla ativação.[22]

Quando a fotoativação do cimento resinoso é realizada indiretamente, alguns aspectos devem ser levados em consideração. À medida que há aumento na espessura do material restaurador, a dispersão e a absorção da luz aumentam, reduzindo dessa maneira a quantidade de energia fornecida pelo dispositivo de fotoativação, que atinge a camada de cimento.[22] Existem fatores extrínsecos e intrínsecos que relacionam com a dureza do material, tais como: efeito da cor, opacidade[15] e espessura da estrutura cerâmica sobre o grau de conversão de cimentos resinosos, medida indiretamente através da microdureza.[2,21] As evidências comprovam que há um efeito atenuador proporcional à espessura da cerâmica e da opacidade do material podendo acarretar menor grau de conversão para cimentos resinosos.[21]

O grau de conversão de monômeros da reação de polimerização dos compósitos é dependente da energia fornecida, caracterizada como sendo o produto da intensidade luminosa e do tempo de exposição.[10,19]

Em trabalho recente, Braga *et. al*, 2002[3], após avaliar diferentes cimentos resinosos em função do método de polimerização, não encontraram correlação entre microdureza e resistência flexural. De acordo com esses autores, fatores como tipo e tamanho de partículas também podem interferir nos resultados, bem como os tipos de monômeros que constituem a cadeia polimérica[1] e a concentração dos iniciadores.[8]

Os cimentos de ativação dupla demonstram melhores propriedades mecânicas quando for empregada fotoativação adequada, do que quando polimerizados somente pelo modo autopolimerizável.[4,6,7,9,11,21] Menor microdureza pode ser sinônimo de polimerização incompleta dos compósitos resinosos para cimentação, causando alteração nas propriedades mecânicas do cimento, além de aumentar a sorpção de água.[21] Além disso, moléculas não polimerizadas podem ser desprendidas do material, causando inflamação tecidual.[5] Deste modo, torna-se importante otimizar os métodos de fotoativação dos cimentos resinosos para melhorar o desempenho clínico desses materiais.

Geralmente, a fotoativação de compósitos é realizada por dispositivos fotoativadores com lâmpada halógena, onde a luz é emitida por filamento de tungstênio. Entretanto, outras tecnologias estão disponíveis, e dentre elas está o aparelho em que a luz é emitida por diodos – LED.[12,14,20] Esse sistema está se tornando cada vez mais difundido e utilizado, contudo, muitas dúvidas permanecem a respeito da efetividade da fotoativação indireta desses dispositivos e sobre o efeito nas propriedades finais dos cimentos resinosos irradiados de diferentes formas através da estrutura dentária e diferentes materiais restauradores.

Como existem diferenças entre os métodos de fotoativação, incluindo a distribuição espectral da luz emitida pelo aparelho fotoativador, torna-se importante o entendimento do processo de fotoativação do cimento resinoso dual. Existem poucas informações sobre o melhor método para fotoativação de cimentos resinoso dual através da interposição de estrutura cerâmica.

PROPOSIÇÃO

O presente estudo teve como objetivos:

1 – Avaliar a eficiência de dois aparelhos fotoativadores na fotoativação indireta pela interposição da cerâmica feldspática na espessura de 2 mm na dureza Knoop, do cimento de dupla ativação Variolink II, em diferentes cores, após dois intervalos de tempo pós-ativação.

2 – Verificar, através da dureza Knoop, a eficiência de dois aparelhos fotoativadores nas ativações direta e indireta do cimento resinoso de dupla ativação Enforce, em diferentes cores e profundidades.

Chapter 1

(Article handed in at the Brazilian Dental Journal)

Immediate and 24-hour Knoop hardness of different shades of dual-cured resin-based cement after indirect photoactivation

The aim of this study was to evaluate the immediate and 24-hour knoop hardness of different shades of dual-cured resin-based cement after indirect photoactivation. Resin cement Variolink II shade transparent XL, A2, A3, opaque (Ivoclar/vivadent) was used to form disk shaped specimens. Feldspatic ceramic; Duceram Plus cor A3 (Degudent) was used to produce disc of thickness: 2.0 mm. The disk was used for the indirect photoactivation mode, through ceramic (Ptc). A halogen light unit, XL 2500 (3M ESPE) was used with power density of 615 mW/cm^2 during 40 s and 40 s with LED – Ultrablue IS (D.M.C.) with power density of 610 mW/cm^2. Koop hardness was measured using HMV-2 (Shimadzu), with a 50g load for 15 seconds, immediately and stored at 37^o C for 24 hours. Four indentations were made in each specimens for the resin cement (Variolink II). Four specimens were made for each color, in each stored time and light source, totaling eighty specimens. The data were submitted to variance analysis and Tukey's test (5%) and showed that the opaque shade of the resin cement demonstrated lower Knoop hardness value in relation to the other shades for both light sources and both activation periods (mediate and 24 hours). The halogen units showed Knoop hardness values higher than LED for A2, A3 and opaque for mediate and XL, A2 and A3 after 24 hours. When the photoactivation methods were compared for the same shade halogen and LED (24 hours) showed higher Knoop hardness values than photoactivation mediate. Opaque shades showed lower knoop hardness than other shades. Halogen units were more efficient than LED units.

Key Words: Knoop hardness, dental ceramic, resin cement.

INTRODUCTION

Ceramic restorations with excellent biocompatibility are widely used to achieve highly esthetic characteristics in fixed prosthodontics. Ceramic or resin-based composite offer an alternative to amalgam or gold for the restoration of medium to large sized posterior cavities.[1] An increasing number of all-ceramic materials and systems are currently available for clinical use.[2] The ceramic inlays lute using zinc phosphate or glass-ionomer cements suffer from poor margin quality, fracture and loss of retention.[3] Therefore, resin-based luting cements have been recommended for the placement of ceramic inlays.[4] On the other hand, ceramic or resin-based composite inlays reduce the amount of light reaching the bottom of the cavity and therefore compromise photo-activation of the luting material [5].

Several clinical studies report excellent long-term success of resin-bonded restorations, such as porcelain laminate veneers, ceramic inlays and onlays, resin-bonded fixed partial dentures and all-ceramic crowns.[6,7,8,9]

Clinical application is simplified through long handling times before and rapid hardening after exposure to light. Shade, thickness, and transmission coefficients of the bonded ceramic restoration and the composite itself influence the conversion rate of the photo-activated material and limit its application to thin silica-based ceramics.[8] Polymerization of cement resin depends upon the light absorption and dispersion within the cement, the shade and opacity of the composite, the filler type and filler load, the concentration of the photoinitiator, the power density delivered by the curing unit, and the irradiation time.[11,,12,13] Thus, adequate polymerization of resin composites is considered to be a factor in obtaining optimal physical and mechanical properties and clinical performance.[14] Inadequate polymerization is associated with a low monomer–polymer conversion rate with a higher residual quantity of double bonds. This alterations physical properties, water absorption and degradation, and leads to discoloration of the cement resin.[15] Moreover, the degree of cure may influence the gap formation in a cavity. High curing rates tend to result in increased wall-to-wall contraction.[16] Since, the degree of cure correlates to the product of the logarithms of light intensity and curing time improving light intensity may allow shorter irradiation times while maintaining the same degree of cure.[17,18,19]

Ceramic onlay and inlay veneer restorations are commonly luted with light-activated composite resin cements.[20,21,22] The degree of polymerization of these cements may be influenced by the ceramic material placed between the curing unit and the resin. Setting times of resin cements used for luting etched porcelain resin-bonded veneers, but their study did not include thickness porcelain samples (3 to 4 mm) as may occur in posterior resin-bonded restorations.[23,24] The porcelain absorbs 40% to 50% of the curing light and that increased porcelain thickness required increased exposure time for resin curing.[25]

The effect of ceramic shade and opacity was investigated by [13] and thickness on the polymerization of resin cements measured by microhardness.[26,27] There is an light attenuation effect according to increasing and to thickness and capacity.[28] It has been showed that thickness of porcelain instead of the opacity or color.[29,30]

However, an adequate cure of the resin-based cement is an important pre-requisite for the mechanical stability and biocompatibility of the restoration.[31] Generally, the thicker the restoration or the darker its shade, the more critical the intensity of the incident light to achieve optimal photopolymerization of the material.[32] The polymerization of light cured resin composites is affected by the chemical composition, filler particle size, shade, light intensity, duration of exposure, and the thickness of the overlying resin or porcelain.[32,33]

The light source/photosensitizer combination most typically used to photopolymerize composite resin is the tungsten-halogen incandescent lamp and camphorquinone. The emission of tungsten-halogen lamps designed for dental use is filtered to pass radiation of wavelengths between 400 and 500 nm, corresponding to the carbonyl absorbance of camphorquinone. More recently, a new method light emitting diodes (LEDs) option of halogen bulbs have been commercially introduced. Previous studies have shown advantages of LED LCUs (light curing units), such as lower temperature or a higher efficiency and disadvantages, such as the irradiance of some LED LCUs or their inability to cure certain types of composites properly compared with conventional halogen LCUs.[32] Halogen LCUs emit a broad light spectrum from approximately 400-525 nm whereas LED LCUs emit a peak like spectrum ranging from approximately 425-500.[33] The higher efficiency of LED compared to halogen LCUs has been explained by the better match of the light emission of LED LCUs with the absorption spectrum of the photoinitiator camphorquinone.[33]

Then, the aim of this study was to evaluate resin cement microhardness after different simulated clinical situations. Two null hypotheses were tested is this study: 1) different activation period will not influence the microhardness of a dual-cured resin cement at different shade; 2) two post-activation times (immediately and 24-hours post-irradiation) will exhibit similar performances in relation to the microhardness of the tested resin cement at different shade.; 3) KHN of resin based cement are similar for into shade.

MATERIALS AND METHODS

Feldspatic ceramic material, Duceram Plus (Degudent), shade dentin A3, were condensed in a metallic die to form a cylindrical specimen that was fired in ceramic furnace (Austromat M, Dekema Austromat-Keramiköfen, Freilassing, Germany), according to manufacture's instructions. This specimen was sectioned with diamond disc at low speed, under water refrigeration. After finishing and glaze firing, a disk with 8.0 (±0.01) mm diameter and 2.0 (±0.01) mm thickness was obtained.

The resin cement Variolink II (Ivoclar-Vivadent, Schaan/Liechtensien), was used in dual cure mode by mixing catalyst and base paste. Paste A shade XL, A2, A3 and Opaque was mixed with Paste Catalyst according to manufacturers' directions and inserted in nylon die with a centered hole with 5.0 (±0.01) mm diameter and 1.0 (±0.01) mm deep. The nylon die was previously coated with black paint (Colorgin Spray, Sherwin-Wiliams do Brazil Ind, São Bernardo do Campo, SP). The aim of this procedure was to limit light transmission through ceramic and resin cement only.[6] A polyester film (±25 µm thickness) was placed above the mold and resin cement. The cement was mixed under controlled temperature (23°C ±1) and relative humidity (higher than 30%), according to ISO 4049 (The International Organization for Standardization).

The resin cement was photo-activated by (Hal) quartz tungsten halogen light unit XL 2500, (3M ESPE, St. Paul, MN, USA) with power density 615 mW/cm^2, for 40 s or using (LED) Light emitting diode photocuring unit (Ultrablue Is, D.M.C. Equipamentos Ltda) with power density irradiance of 610 mW/cm^2 for 40 seconds. The power density irradiance of the curing units was measured with a radiometer (Curing Radiometer, model 100, Demetron/Kerr, Danbury, CT, USA).

Two post-cured modes were investigated: 1- immediate, specimens were evaluated 15 minutes after photo-activation. 2– 24 hours post-cure, specimens were stored in dry and dark conditions at 37°C for approximately 24 hours.

Groups were tested according to activation mode and for different shades of resin cement. Four groups (n=20) for indirect photoactivation were made.

The ceramic disc with 2.0 mm thickness was interposed between resin cement and light source (Indirect Photoactivation). Four groups (n=20) were created with this mode for Knoop Hardness (KH) readings: immediately, 15 minutes post-activation by light irradiation (DPa-immed) or after 24 hours post-activation (DPa-24h). Then indirect photo activation trough ceramic was performed.

Preliminary procedures before indentation were completed until 10 minutes after catalyst and base paste mixture. Specimens were fixed in a premolded acrylic resin die and sticky wax was previously used to improve fixation and fill eventual space or lack of adaptation. Resin cement disks were sectioned longitudinally and the surface was ground and finished in waterproof sandpaper (Norton SA, São

Paulo, SP, Brazil) in decreasing sequence (180, 320, 400, 600 and 1200 grid sandpaper).

A universal indenter tester HMV-2 (Shimadzu, Tokyo, Japan) was used for knoop hardness test (KH). Tester was set for automatic mode of 50 grams-force for 15 seconds. Knoop hardness values were obtained accordingly at 100 µm from surface irradiated. Four indentations were made with 1 mm distance between them.

Measurements were made within 40X magnification of crosssectional area of resin cement specimens. KH number was provided automatically by tester's software, based in indentation measurement performed for operator. The testing was conducted by one experienced operator, to maximize standardization.

Data treated with Variance Analysis (ANOVA) with split plot factorial on parcel (Post-cure time X activation mode X shade) and sub parcel represented by factor shade of lecture. Overall changes in Knoop hardness between groups were made for Tukey's test (All pairwise multiple comparison procedure). All tests were performed at $p < 0.05$.

RESULTS

Table 1 showed that when halogen was compared with Led (mediate photoactivation) difference statistically significant was found for the shades A2, A3 and opaque ($p<0.05$). No difference was found for XL shade ($p>0.05$). After 24 hours, difference statistically significant was found between halogen and LED for

XL, A2 and A3 shades ($p<0.05$). No difference was found for opaque shade ($p>0.05$).

When the Knoop hardness was analysed among the shades for each light source (mediate) opaque shade showed lower knoop hardness with difference statistically significant in relation XL, A2 and A3 shade for halogen and LED source ($p<0.05$). No difference was found among XL, A2 and A3 for halogen and between A2 and A3 for LED ($p>0.05$).

After 24 hours, the results showed that there were significant statistically differences among opaque shade than XL, A2 and A3 shade using the halogen light photoactivation ($p<0.05$). No difference was found among XL, A2 and A3 ($p>0.05$). For LED opaque showed lower Knoop hardness with difference statistically significant in relation to XL ($p<0.05$). No difference was found between opaque, A2 and A3 ($p>0.05$).

When the photoactivation methods were compared for the same shade photoactivation with halogen and LED (24 hours) was statistically significant in relation to photoactivation mediate for halogen and LED in all shades ($p<0.05$). No difference was found between halogen and LED for photoactivation 24 hours and mediate ($p>0.05$).

DISCUSSION

The two null hypotheses tested in this study were rejected. There were statistical differences in microhardness means according to different conditions tested (time post-irradiation, activation mode and shade of test on resin cement) (Table 1).

Bonded all-ceramic restorations is popular with clinicians and patients because of their superior esthetics: notably the diffuse transmission combined with diffuse and spectral reflectance of light that mimics the depth of translucency and color of natural teeth.[17,19] However, clinical success with these restorations requires that they are bonded to teeth with high-strength, low solubility, and thin-film adhesives commonly light polymerized resin luting agents.[15,18] The requirement that the resin luting agents is completely polymerized within an acceptable period of time.[14,36]

In addition, for ceramic veneers of comparable thickness and composition, the light polymerization of the underlying resin luting agent will be markedly affected by the light source, notably the wavelength and intensity of emitted light.[20] While the wavelength of the light is determined by the generation source. The polymerization method, the use of catalyst may be a decisive factor for hardness and specially for the depth of polymerization of dual – polymerization composite.[20] Attenuation of light is also an important factor. Light polymerization of all portions of the luting agent is not always possible because, for example, the substantial thickness of the inlay restoration.[24]

There are several factors that can influence the degree of polymerization of a resin cement, for instance, the porcelain shade and thickness, the light unit, and the curing time.[27,28] Polymerization of cement resin depends upon the light absorption and dispersion within the cement, the shade and opacity of the composite resin.[9]

McLean pointed out that curing units produced sufficient hardness of a composite resin luting material when cured through ceramic restorations of 2 mm thickness in a shorter time than conventional curing. However, the necessary curing time was 50% higher than that recommended by the manufacturer.[25]

When a ceramic restoration is cemented with dual curing or light curing, the light from the curing unit should penetrate the restoration to ensure bonding and optimal properties of resin cement.[32,33] Linear regression analyses were used to predict the feasibility of obtaining a shade match of dental porcelain with shade tabs at porcelain thickness that are clinically achievable. Considering that thickness of 2,0 mm would require excessive tooth reduction that would likely violate pulpal integrity of an anterior tooth or adversely weaken tooth structure.[7]

Dual-cure resin based cements have been recommended for luting ceramic or resin composite inlays to compensate for the attenuation of the curing light effected by the inlay and to allow complete polymerization of the luting material even at the bottom of the cavity where access for the curing light is limited. [8,9]

The ceramic material under which a resin cement is cured seems to exert a considerable influence on the degree of resin polymerization achievable much the same as thickness does with direct composite resin restorations cured by light.[2]

According to ISO 4049 the setting time for dual cure resin cements should be no more than 10 min. The present study shows that deep of polymerization of resin cement layer is sometimes not properly to receive immediate loads. At this time resin cement should have good mechanical properties, such as satisfactory elastic modulus and microhardness, which will protect bonding and increasing fracture toughness of some ceramic restorations, due to inherent brittleness and limited flexural strength of silica-based ceramics.[1,4]

The irradiance of light source, the exposure time and light transmission of irradiated material are significant variables that affect the hardness or conversion profile[1,5] Additionally, the light cure resin composites is affected by the chemical composition, filler particle size, shade, light intensity, duration of exposure, and the thickness of the overlying resin or porcelain.[3,8] Thus, adequate polymerization of resin cement is considered to be factor for obtaining optimal physical and biological properties and clinical performance[6,14,18,23,32].

CONCLUSIONS

1 - The opaque shade of the resin cement demonstrated lower Knoop hardness value in relation to the other shades for both light sources and both activation periods (mediate and 24 hours), except LED, shade A2 e A3.

2 – The QTH light source showed Knoop hardness values higher than LED for A2, A3 and opaque for mediate and XL, A2 and A3 after 24 hours.

3 - When the photoactivation methods were compared for the same shade halogen and LED (24 hours) showed higher Knoop hardness values than photoactivation mediate.

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Table 1 - Means and Standard Deviation of Knoop Hardness Number with ceramic interposition at 100 µm depth of different shades of resin cement photoactivated with XL 2500 and LED – Ultrablue Is.

Light photo-activation unit		Shade of resin cement			
		XL	A2	A3	Opaque
Photoactivated (Mediate)	Hal	41.77 (3.2) Aa	39.48 (3.9) Aa	38.34 (4.1) Aa	34.78 (5.4) Ab
Photoactivated (Mediate)	LED	40.18 (3.3) Aa	35.57 (3.8) Bb	34.33 (3.1) Bb	27,77 (2.5) Bc
Photoactivated (24 hours)	Hal	*59.46 (3.0) Aa	*55,58 (2,5) Aa	*58.29 (3.5) Aa	*45.52 (5.9) Ab
Photoactivated (24 hours)	LED	*53.34 (4.7)Ba	*49.57 (5.4) Bab	*48.48 (3.9) Bb	*46.48 (4.9) Ab

Means followed by the different small case in the row and capital letter in the columns indicate statistical difference at the 95% confidence level (Tukey's test, $p<0.05$).

* The values are statistically higher from that on columns (comparisons for the same shade).

Chapter 2

Article handed in at Journal Oral Rehabilitation

Linear regression analyses of different shades and depth hardness of the dual cure resin based cement

SUMMARY: The aim of this study was to evaluate hardness of different shades of dual-cured resin-based cement after indirect photoactivation. Resin based cement Enforce (Dentsply) shade A2(A), B1(B) and opaque(OP) was used to form disk shaped specimens that was photo activated directly or through 2.0 mm Feldspatic ceramic piece (Duceram Plus, Degudent). A tungsten halogen light curing unit (XL 2500, 3M ESPE light) was used with power density 600 mW/cm^2, for 40s. Twelve groups were tested (n=15). Knoop Hardness numbers (KHN) were taken in-depth from cross sectional areas at 100, 300, 500, and 700 µm depth of the irradiated surfaces of the specimens after storage for 24 hours at 37°C. Three-Way ANOVA and Tukey´s test was used to investigate interactions of Shade (OP, A, B), mode (direct and through ceramic) and post activation time (mediate and 24h after). Opaque shade showed KHN statistically inferior to others groups ($p<0.05$). Mediate KHN readings showed that hardness near top (100µm) is statistically different from bottom hardness (700µm). Changes in KHN as a function of depth of hardness reading was well predicted by using linear regression model ($r^2=0.97$). Linear regression is a good model to predict the KHN variation as a function of depth of specimen of resin cement direct and indirectly photoactivated.

Key Words: Hardness, dental ceramic, resin cement.

Introduction

The ceramics exhibit mechanical properties which include high strength, excellent esthetics, marginal integrity, and etchability. (Rasetto *et al*., 2004). Ceramic etchability permits adhesive bonding to the underlying tooth structure by permitting micro-mechanical interlocking of dual-curing resins within surface features (Luo *et al*., 2003).

An increasing number of all-ceramic materials and systems are currently available for clinical use (Jung *et al*., 2001). Several clinical studies document excellent long-term success of resin-bonded restorations, such as porcelain laminate veneers, ceramic inlays and onlays, resin-bonded fixed partial dentures, (Mills, 1995; Harrington *et al*., 2003; Uhl *et al*., 2003) and all-ceramic crowns. (Park *et al*., 2002).

Resin-based composites are the material of choice for the adhesive luting of ceramic restorations (Jung *et al*., 2001). Composite cements have composition and characteristics similar to conventional restorative composites and consist of inorganic fillers embedded in an organic matrix (for example: Bis-GMA, TEGDMA, UDMA) (Kramer *et al*., 2000). Composite cements can be classified according to their initiation mode as autopolymerizing (chemically activated), photoactivated, or dual-activated materials (Rueggeberg *et al*., 2000). Photoactivated composites offer wide varieties of shades, consistencies, and compositions (Rueggeberg *et al*., 1993). Clinical application is simplified through long handling times before and rapid hardening after exposure to light. Shade, thickness, and transmission coefficient of the bonded ceramic restoration and the composite itself influence the conversion rate of the photo-activated material and limit its application to thin silica-based ceramics (Blatz *et al*., 2003). Thus, adequate polymerization of resin composites is considered to be a important factor in obtaining optimal physical and biological properties and a satisfying clinical performance (Luo *et al*., 2003).

Inadequate polymerization is associated with a low monomer–polymer conversion rate with a higher residual quantity of double bonds, which causes inferior physical properties, raises water absorption and solubility, and leads to discoloration of the cement resin (Danesh *et al*., 2004). High curing rates tend to result in increased wall-to-wall contraction (Feilzer *et al*., 1988) whereas, if the restoration does not receive sufficient total energy, other problems may arise (Nomoto *et al*., 2004) the bonding resin at the bottom of the restoration may not polymerize resulting in a weak bond between the tooth and restoration. Since, the degree of cure correlates to the product of the logarithms of light intensity and curing time (Nomoto *et al*., 2004) improving light intensity may allow shorter irradiation times while maintaining the same degree of cure (Danesh *et al*., 2004). However, an adequate cure of the resin-based cement is an important pre-requisite for the stability and biocompatibility of the restoration (Hofmann *et al*., 2001). Generally, the thicker the restoration or the darker its shade, the more critical the intensity of the incident light to achieve optimal photopolymerization of the material (Rasetto *et al*., 2004). The polymerization of light cured resin composites is affected by the chemical composition, filler particle size, shade, light intensity, duration of exposure, thickness of the overlying resin or porcelain and the depth of cure (Feilzer *et al*., 1988; Rueggeberg, 1993; Lee *et al*., 2001). Depth of cure can be defined as the extent of quality resin polymerization deep from the surface of composite restoratives (Rueggeberg, 1993).

Ceramic onlay and inlay veneer restorations are commonly luted with light-activated composite resin cements. The degree of polymerization of these cements may be influenced by the ceramic material placed between the curing and the resin (Strang *et al*., 1987), reported setting times of resin cements used for luting etched porcelain resin-bonded veneers, but their study did not include thick porcelain samples (3 to 4 mm) as may occur in posterior resin-bonded restorations. They showed that porcelain absorbs 40% to 50% of the curing light and that increased porcelain thickness required increased exposure times for resin curing (Blackman *et al*., 1990).

Light exposure of the resin through the porcelain samples markedly increased the conversion degree. Exposure times recommended by the manufacturers were insufficient to compensate for the attenuation of light by the tooth and the restorative material (Blatz *et al*., 2003). The microhardness of a light cured composite decreased when cured through varying thickness of ceramic, and increasing the cure time from 40 to 60 s produced a significant increase in microhardness (Della Bona *et al*., 1998). The thickness of the ceramic, not opacity, was the primary factor affecting light transmission. The polymerization of light – activated composite resin luting agents cannot predictably be accomplished through a restoration exceeding 2 mm in thickness using light exposure of 90 s or less (Lee *et al*., 2001).

The effect of ceramic shade and opacity was investigated by Foxton *et al*. (2003) and thickness on the polymerization of resin cements measured by microhardness (Hofmann *et al;* Jung *et al*). There is an light attenuation effect according to increasing an to thickness and capacity.[16] It has been showed that thickness of porcelain instead of the opacity or color.[13,15]

Then, the aim of this study was to test the following null hypothesis; 1- KHN of resin based cement at different depths is similar to the same shade. 2- Indirect photoactivation through ceramic shows KHN similar to direct photoactivation. (mediate and 24h after). 3 – There is no relationship between KHN and cross-sectional depth of reading for simulations.

Materials and Methods

Feldspatic ceramic material, Duceram Plus (Degudent), shade dentin A3, were condensed into a metallic die to form a cylindrical specimen that was fired in ceramic furnace (Austromat M, Dekema Austromat-Keramiköfen, Freilassing, Germany), according to manufacture's instructions. The disc 2.0 (±0.01) mm thick and 8.0 (±0.01) mm in diameter. One specimen was obtained and submitted to finishing and glaze firing.

A resin cement Enforce with fluoride (Dentsply Indústria e comércio Ltda. Petrópolis-RJ, Brazil) was mixed according to manufacturers' instructions at 23^{o} C ± 1^{o} C and a controlled relative humidity (higher than 30%), according to ISO 4049 (The International Organization for Standardization). Paste base shade B1, A2 and Opaque) were mixed in Paste catalyst according to manufacturers' directions and inserted in nylon die with a centered hole with 5.0 (±0.01) mm diameter and 1.0 (±0.01) mm deep. The nylon die was previous coated with black paint (Colorgin Spray, Sherwin-Wiliams do Brasil Ind Com Ltda, São Bernardo do Campo, SP). The aim of this procedure was to limit light transmission through ceramic and resin cement only.[6] A polyester film (±25 µm thickness) was placed above the mold and resin cement.

The resin cement was photo-activated by (Hal) tungsten halogen light unit XL 2500, (3MESPE, St. Paul, MN, USA) with power density 615 mW/cm^2, for 40 s. The irradiance (light intensity) of the curing units was measured with a hand held radiometer (Curing Radiometer, model 100, Demetron/Kerr, Danbury, CT, USA).

Two post-cured modes were investigated: 1- immediate, specimens were evaluated 15 minutes after photo-activation. 2 - 24 hours post-cure, specimens were stored in bottles in dry and dark conditions at 37°C for approximately 24 hours.

Each group was tested with direct photo activation (without ceramic interposed) for mediate (control, C-m) and 24h post-photoactivation (control, C-24h). Indirect Photoactivation Mode (Test groups). The ceramic disc with 2.0 mm thickness was interposed between resin cement and the light tip of LCU. Then indirect photo activation trough ceramic was performed. Direct Photoactivation Mode (Controls groups); when direct photoactivation was performed, without ceramic interposed.

Groups were tested according to activation mode direct and indirect for different shades of resin cement. Eight groups (n=20) were created and for each group a respective control (Direct photo-activation) was used.

Mode DPa (Direct Photo-activation controls); when direct photoactivation was carried out, without ceramic interposed. Two groups (n= 40) were created with this mode for Knoop Hardness (KH) readings: immediately, 15 minutes post-activation by light irradiation (DPa-immed) or approximately 24 hours post-activation (DPa-24h).

Mode IPa (Indirect Photoactivation): The ceramic disc with 2.0 mm thickness was interposed between resin cement and light tip of LCU. Two groups (n=40) were created with this mode for Knoop Hardness (KH) readings: immediately, between 15 minutes post-activation by light irradiation (DPa-immed) or approximately after 24 hours post-activation (DPa-24h). Then indirect photo-activation trough ceramic was performed.

Preliminary procedures before indentation were completed until 10 minutes after catalyst and base paste mixture. Specimens were fixed in a premolded acrylic resin die and sticky wax was previously used to improve fixation and fill eventual space or lack of adaptation. Resin cement disks were sectioned longitudinally and the surface was ground and finished in waterproof sandpaper (Norton SA, São Paulo, SP, Brazil) in decreasing sequence (180, 320, 400, 600 and 1200 grid).

A universal indenter tester HMV-2 (Shimadzu, Tokyo, Japan) was used for knoop hardness test (KH). Tester was set for automatic mode of 50 grams-force for 15 seconds. Knoop hardness values were obtained according to different depths at 100, 300, 500 and 700µm from surface irradiated. Three indentations were made for each depth with 1mm distance between them, totaling 12 indentations for specimens

Measurements were made within 40X magnification of cross-sectional area of resin cement specimens. KH number was provided automatically by tester's software, based in indentation measurement performed for one operator. The testing was conducted by one experienced operator, to maximize standardization.

Data were analyzed in Statistical software (SigmaStat 3.01, SPSS Inc,. Chicago, Ilinois). Overall changes in KHN between different depths were evaluated by One-way Analysis of Variance (ANOVA) and Tukey's Test. Overall changes in Knoop hardness between groups were made for Tukey's post-hoc (All pairwise multiple comparison procedure). Three-Way ANOVA was used to investigate interactions of Shade (O, A, B), mode (direct and through ceramic) and post activation time (mediate and 24h after). A predictive period of microhardness (dependent variable) as a function of depth (independent variable) were investigated using linear regression mode. All tests were performed at 0.05 level of significance.

Results

Three way ANOVA revealed significant interaction between Shade, radiation mode and post activation time ($p<0.002$). Different KHN means were found for shades (A2>B1>Op), although these results were dependent of radiation mode and post cure time. Comparisons of shades inside mediate post activation time reveal that A>B>O, and inside 24h post activation time indicate that A=B>O. Only for shade O, KHN obtained after 24h post activation readings were statistically different among irradiation modes (Direct > trough ceramic). These comparisons are represented in Figure 1.

Group C-Om was statistically different after 24 hours of storage from other groups of same shade. Comparisons among KHN of each test group and respective control group showed only one difference. C-O24h was statistically superior to Om ($p<0.05$). When groups indirectly photoactivated were compared, inside same shade, KHN means was similar. There are no differences among mediate and 24h post photoactivation time. When comparisons of KHN means between each layer were performed it was possible to observe that only B24h showed similar means of depth, for other groups always KHN at 100µm (near top surface) was statistically superior to 700µm (near bottom surface).

Linear regression lines for each group investigated were plotted in Figures 2, 3 and 4. For all situations data were well fit in linear regression model ($r^2=0.97$).

For different scenarios investigated KHN obtained in cross-sectional area of resin based cement disks (in depth) decrease from top to base. KHN at 100µm was statistically superior to 700µm, except for group B24h. Table 1 summarizes and compares KHN means at different depths for each group.

Discussion

Each shade requires a specific activation strategy in order to maximize hardness. Manufactures should provide additional data for some resin cements shades, (i.e. opaque) whether thicker layers or short photoactivation time must be avoided.

All-ceramic restorations are because of their superior esthetics: notably the diffuse transmission combined with diffuse and spectral reflectance of light that mimics the depth of translucency and color of natural teeth. (Hondrum, 1992; Jung, Friedl *et al.*, 2001). However, clinical success with these restorations requires that they are bonded to teeth with high-strength, low solubility, and thin-film adhesives commonly light polymerized resin luting agents (Hofmann, Hugo *et al*., 2000; Jandt, Mills *et al*., 2000). The requirement that the resin luting agents is completely polymerized within an acceptable mode of time (Hasegawa, Boyer *et al*., 1991).

There are several factors that can influence the degree of polymerization of a resin cement, for instance, the porcelain shade and thickness, the light unit, and the curing time (Myers, Caughman *et al*., 1994; Nomoto, 1997). Polymerization of cement depends upon the light absorption and dispersion within the cement, the shade and opacity of the composite resin. (Feilzer and De Gee *et al*.,1988)

McLean pointed out that units curing produced sufficient hardness of a composite resin luting material when cured through ceramic restorations of 2 mm thickness in a shorter time than conventional curing. However, the necessary curing time was 50% higher than that recommended by the manufacturer (McLean, Nicholson *et al*., 1994).

When a ceramic restoration is cemented with dual curing or light curing, the light from the curing unit should penetrate the restoration to ensure bonding and optimal properties of resin cement (Rueggeberg and Caughman, 1993; Rosenstiel, Land *et al*., 1998). Linear regression analyses were used to relation of obtaining a shade of dental porcelain with shade tabs at porcelain thickness that are clinically achievable. It is acknowledged that thickness of 2,0 mm would require excessive tooth reduction that would likely damage pulpal integrity of an anterior tooth or adversely weaken tooth structure (Danesh, Davids *et al*., 2004).

While the wavelength of the light is determined by the generation source, from the polymerization method, the use of catalyst may be a decisive factor for hardness and specially for the depth of polymerization of dual – polymerization composite (Knezevic, Tarle *et al*., 2002). Attenuation of light is also an important factor, and light polymerization of all portions of the luting agent is not always possible because, for example, the substantial thickness of the inlay restoration may shade or block the light (Marais, Dannheimer *et al*., 1997)

Dual-cure resin based cements have been recommended for luting ceramic or resin composite inlays to compensate for the attenuation of the curing light effected by the inlay and to allow complete polymerization of the luting material even at the bottom of the cavity where access to the curing light is limited (Feilzer, De Gee *et al*., 1988; el-Badrawy and el-Mowafy,1995).

The ceramic material under which resin cement is cured seems to exert a considerable influence on the degree of resin polymerization achievable much the same as thickness does with direct composite resin restorations cured by light (Hofmann, 2001).

The present study shows that deep of polymerization of resin cement layer is sometimes not properly with maximum hardening to receive immediate loads. At this time resin cement should have good mechanical properties, such as satisfactory elastic modulus and microhardness, which will protect bonding and increasing fracture toughness of some ceramic restorations, due to inherent brittleness and limited flexural strength of silica-based ceramics (Breeding, Dixon *et al*., 1991; Barghi and McAlister, 2003).

The exposure time and light transmission of irradiated material are significant variables that affect the hardness or conversion profile (Brodbelt, O'Brien *et al*., 1980; Barghi and McAlister, 2003). Additionally, the light cure resin composite is affected by the chemical composition, filler particle size, shade, light intensity, duration of exposure, and the thickness of the overlying resin or porcelain (el-Badrawy and el-Mowafy, 1995; Blatz, Sadan *et al*., 2003). Thus, adequate polymerization of resin cement is considered to be factor for obtaining optimal physical and biological properties and clinical performance.

According to linear regression a decreasing of KHN can be well predicted by increasing of depth of at cross-sectional areas. Linear regression is a good model to predict the KHN variation as a function of depth of specimen of composite resin.

Conclusions

1 – Knoop hardness positive linear regression was determined in depth of specimen of resin cement direct and indirectly photoactivated;

2 - Opaque shade was KHN statistically inferior to other resin cement groups; and,

3 – Mediate KHN showed that Knoop hardness near top (100 µm) is statistically superior different from bottom surface (700 µm).

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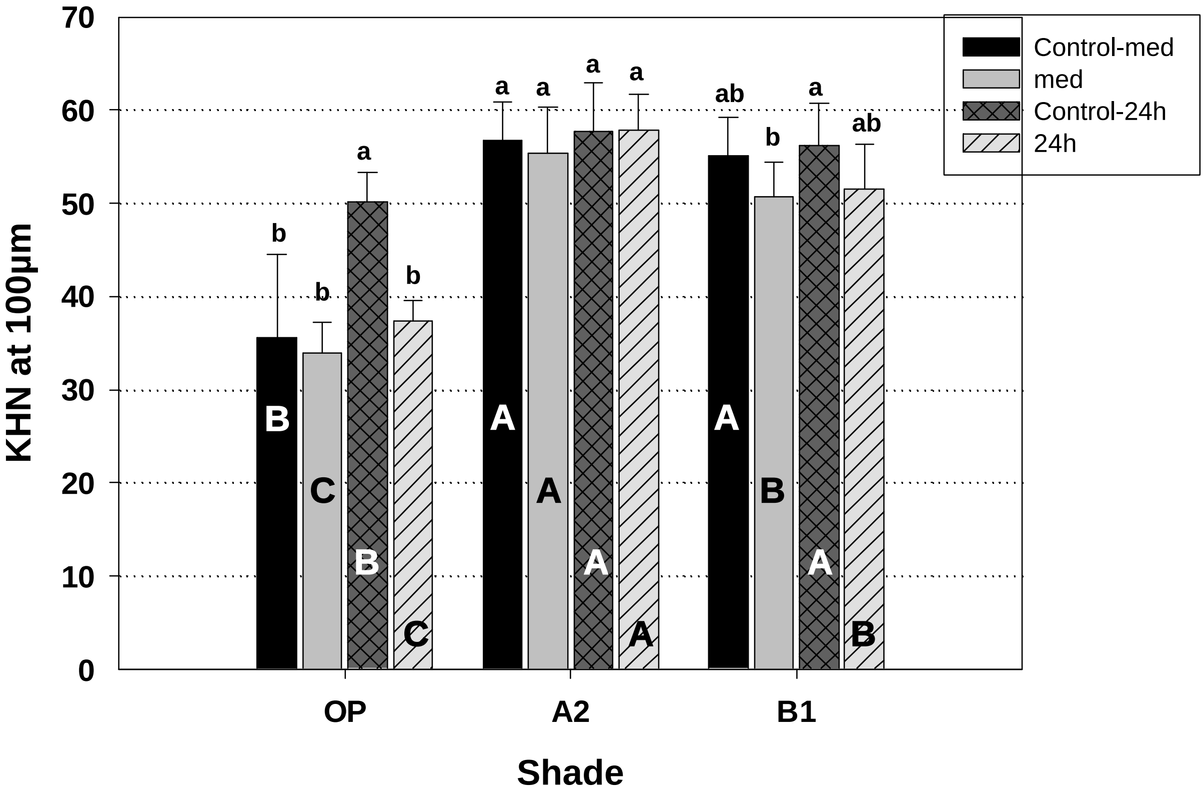


Figure 1 - Bar Graph representation of means (SD) for different photo-activation simulations according to shades tested. Different small letters represents statistically significant differences between activation mode same shade ($p<0.05$). Different capital letters aligned on horizontal represents statistically significant differences between groups.

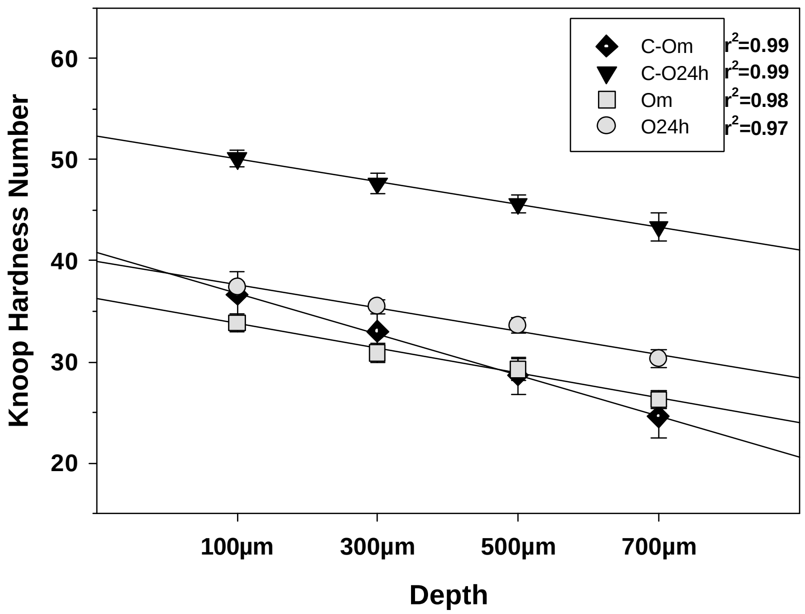

OPAQUE (OP)

Figure 2 - Linear regression of KHN as a function of depth of cross-sectional area of specimens for shade Opaque (OP).

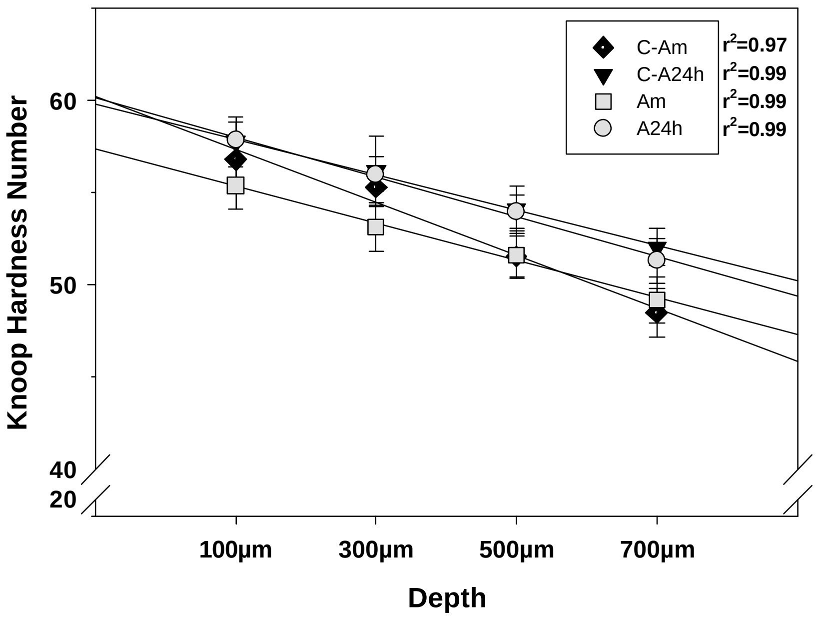
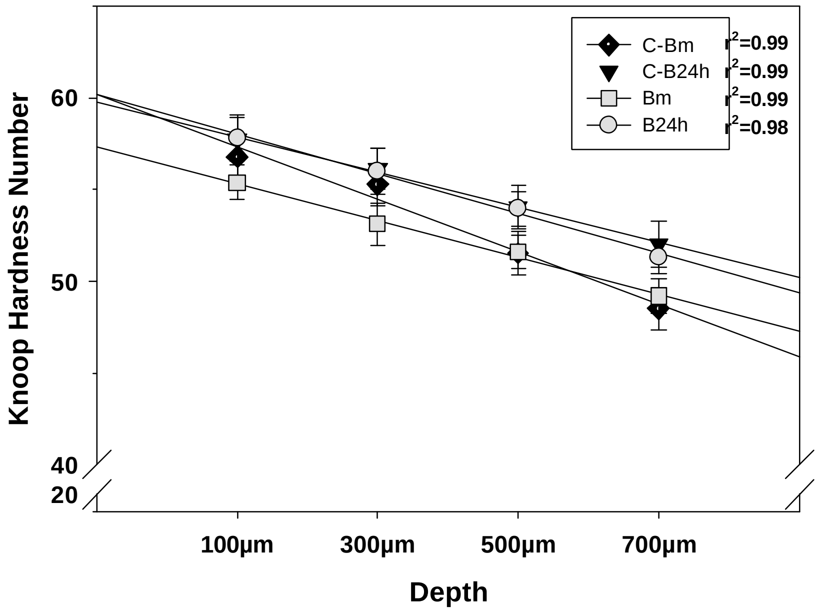

Shade A2

Figure 3 - Linear regression of KHN as a function of depth of cross-sectional area of specimens for shade A2.



Shade B1

Figure 4 - Linear regression of KHN as a function of depth of cross-sectional area of specimens for shade B1.

TABLE 1- Comparisons between groups

Cross-sectional depth	OP				A2				B1			
	C-Om	C-O24h	Om	O24h	C-Am	C-A24h	Am	A24h	C-Bm	C-B24h	Bm	B24h
100µm	^{AB}35.65 (±8,91)	^{A}50.11* (±3.14)	^{A}33.9 (±3.34)	^{A}37.34 (±2.27)	^{A}56.78 (±4.08)	^{A}57.75 (±5.14)	^{A}55.39 (±4.9)	^{A}57.82 (±3.91)	^{A}55.15 (±4.14)	^{A}56.15 (±4.54)	^{A}50,76 (±3,68)	^{A}51.6 (±4.8)
300µm	^{AB}33.04 (±8,56)	^{AB}47.64 (±3.73)	^{AB}30.91 (±3.71)	^{BC}35.47 (±2.8)	^{AB}55.28 (±3.68)	^{AB}56.15 (±4.62)	^{AB}53.13 (±5.06)	^{A}55.99 (±3.63)	^{AB}52.82 (±4.48)	^{AB}53.97 (±4.42)	^{AB}48,26 (±4.42)	^{A}49.74 (±4.81)
500µm	^{BC}28,68 (±7,13)	^{BC}45.63 (±3.36)	^{BC}29.24 (±4.23)	^{C}33.62 (±3.15)	^{BC}51.53 (±4.35)	^{AB}54.07 (±4.98)	^{AB}51.62 (±4.8)	^{AB}53.97 (±3.61)	^{BC}50.57 (±4.57)	^{BC}51.52 (±4,62)	^{BC}45.38 (±3.54)	^{A}48.52 (±3.7)
700µm	^{C}24,6 (±8,27)	^{C}43.34 (±4.06)	^{C}26.3 (±3.57)	^{D}30.35 (±3.53)	^{C}48.5 (±5.1)	^{B}52.03 (±3.92)	^{B}49.2 (±4.87)	^{B}51.3 (±4.79)	^{C}48.2 (±4.75)	^{C}49,19 (±4,95)	^{C}42.94 (±3.71)	^{A}48.05 (±2.48)
Power of test (One- Way ANOVA[1])	0.91	0.995	0.998	1.00	0.999	0.712	0.712	0.963	0.939	0.926	0.999	0.315
Linear Regression(2)												
r^2 value	0.999	0.999	0.989	0.978	0.976	0.997	0.993	0.992	1.00	1.00	0.999	0.981
p value (3)	<0.001	<0.001	=0.006	=0.011	=0.012	=0.002	=0.004	=0.004	<0.001	<0.001	<0.001	=0.01

Different letters in columns represents statistically significant differences among means for in-depth readings($p<0.05$). (*)Comparisons among controls groups and among through ceramic irradiation, asterisk indicates higher mean, similar means were connected by horizontal bar. (1) When power of test is set below 0.8, absences of differences should be cautiously interpreted. (2) Linear regression for each mean of group (dependent variable) as a function of depth of KHN reading (independent variable). (3) The "p" value was obtained using Analysis of variation for linear regression.

CONSIDERAÇÕES GERAIS

A dureza dos cimentos resinosos é uma propriedade mecânica importante para avaliar o comportamento da resistência e do desgaste deste material. Os diferentes tipos de aparelhos fotoativadores, espessura da coroa de cerâmica livre de metal e cor dos cimentos resinosos são fatores que influenciam diretamente na dureza dos cimentos.

Os cimentos resinosos de dupla ativação necessitam de polimerização complementar através da luz, podendo ser utilizado o aparelho de lâmpada halógena e/ou LED (diodo emissor de luz). Neste estudo, a interação entre os aparelhos fotoativadores mostrou que o aparelho fotoativador por lâmpada halógena apresentou valores de dureza Knoop estatisticamente superior em relação ao aparelho LED para as cores do cimento A2, A3 e opaco para a condição mediato e XL, A2 e A3 após 24 horas. Por outro lado, quando os dois métodos de fotoativação foram comparados para a mesma cor do cimento, os aparelhos fotoativadores com lâmpada halógena e LED (24 horas) promoveram valores de dureza Knoop estatisticamente superiores aos valores fotoativados na condição mediato. Com relação a cor dos cimentos ficou evidente que a cor opaca dos cimentos resinosos Variolink II e Enforce apresentaram os menores valores de dureza Knoop em relação as outras cores, para as duas fontes de luz e dois períodos de ativação (mediato e 24 horas). Isso ocorre provavelmente pela maior absorção dos pigmentos que compõem essa cor diminuindo a passagem de luz pelo cimento, tendo como conseqüência menor polimerização e dureza.

Outro fator importante a ser considerado é a profundidade de polimerização do cimento onde a literatura mostra que a região de superfície apresenta melhor polimerização em relação a região de fundo, como conseqüência maior dureza. Neste estudo observamos que, a dureza Knoop mediata mostrou valores de dureza Knoop na região de topo (100 µm) estatisticamente superior em relação a dureza na região de fundo (700 µm). A regressão linear positiva da dureza Knoop foi obtida em função da profundidade da amostra do cimento resinoso (Enforce) pela fotoativação direta e indireta pela interposição da cerâmica.

Todos esses fatores associados à espessura da cerâmica de uma prótese podem interferir na polimerização do cimento resinoso. Os materiais cerâmicos apresentam diferentes estruturas com diferentes tipos e quantidades de cristais. Alguns sistemas cerâmicos apresentam em sua composição somente cristais, com ausência total da fase vítrea. Assim, dependendo do tipo e espessura da cerâmica, a quantidade de luz emitida pelos aparelhos fotoativadores tendem a diminuir, consequentemente a fotoativação do cimento resinoso. Além disso, se for empregado um sistema cerâmico com ausência ou reduzida quantidade da fase vítrea, provavelmente o cimento resinoso dual não será fotoativado, necessitando o uso de um cimento quimicamente ativado.

CONCLUSÕES GERAIS

Com base nos achados destes estudos é possível concluir que:

1 – O cimento resinoso Variolink II opaco apresentou o menor valor de dureza Knoop em relação as outras cores, para as duas fontes de luz e dois períodos de ativação (mediato e 24 horas).

2 – O aparelho fotoativador por lâmpada halógena apresentou valores de dureza Knoop estatisticamente superior em relação ao aparelho LED para as cores do cimento A2, A3 e opaco para a condição mediato e XL, A2 e A3 após 24 horas.

3 – Quando os dois métodos de fotoativação foram comparados para a mesma cor do cimento, os aparelhos fotoativadores com lâmpada halógena e LED (24 horas) promoveram valores de dureza Knoop estatisticamente superiores aos valores fotoativados na condição mediato.

4 – A regressão linear positiva da dureza Knoop foi obtida em função da profundidade da amostra do cimento resinoso (Enforce) pela fotoativação direta e indireta pela interposição da cerâmica.

5 – O cimento resinoso Enforce na cor opaca mostrou valores de dureza Knoop estatisticamente inferiores as outras cores;

6 – A dureza Knoop mediata mostrou valores de dureza Knoop na região de topo (100 µm) estatisticamente superior em relação a dureza na região de fundo (700 µm).

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ANEXO

MATERIAIS E MÉTODO

1 - Materiais

A descrição dos materiais, nome comercial e fabricante estão listados na Tabela 1, composição dos cimentos e cerâmica e especificações técnicas dos aparelhos fotoativadores (Tabelas 2 e 3).

Tabela 1- Descrição dos materiais e equipamentos que foram utilizados no estudo.

MATERIAL/ EQUIPAMENTO	CORES	FABRICANTE
Cerâmica feldspatica Duceram Plus	Dentina A3	(DeguDent, Germany)
Cimento resinoso Variolink II	- XLbranco transparente (A1) - A2 - Amarelo 210 (A3) - Branco opaco	Ivoclar Vivadent AG, Shann Liechtenstein
Cimento resinoso Enforce com flúor	- Opaco - A2 - B1	Dentsply Indústria e Comércio Ltda, Petrópolis, Brasil
Aparelho fotoativador XL 2500		(3M ESPE, St. Paul, MN, USA)
Aparelho fotoativador LED – Ultrablue Is		(D.M.C. Equipamentos Ltda)

Tabela 2 - Composição da cerâmica, cimentos resinosos.

MATERIAL	COMPOSIÇÃO*
Cerâmica Duceram Plus (Feldspática reforçada com leucita)	K_2O_3, Al_2O_3, $6SiO_2$, SnO, ZrO, Na_2O, CaO e pigmentos
Cimento resinoso Variolink II Cores: Transparente XL, A3, A2 e Opaco	BISGMA, TEGDMA, UEDMA, vidro de bário, trifluoreto de itérbio e flúorsilicato de bário e alumínio. Tamanho de partícula (0,7 µm) % de partículas em peso (75%)
Cimento resinoso Enforce Cores: A2, B1 e Opaco	BISGMA, TEGDMA e flúorsilicato de bário e alumínio Tamanho de partícula (1,5 µm) % de partículas em peso (67,5)

* Informações dos fabricantes.

Tabela 3 – Descrição e especificações técnicas dos aparelhos fotoativadores.

APARELHO	FONTE DE LUZ	TIPO DE ONDA	TEMPO MÁXIMO DE ACIONAMENTO	IRRADIÂNCIA MÉDIA (mW/cm^2)
XL 2500* (3M)	Halógena (1 Lâmpada com filamento incandescente de tungstênio)	Fibra ótica Diâmetro Entrada: Saída:	Indeterminado	615
Ultrablue Is (D.M.C.)*	LED Light-emitting diode (1 LED – 5 Watts)	Sonda Convergente Entrada: Saída:	Indeterminado	610

* Informações dos fabricantes.

2 – Método

2.1 - Confecção dos discos em cerâmica feldspática para ensaio de dureza Knoop.

Foram confeccionados cilindros em cerâmica feldspática Duceram Plus (Degussa Dental), utilizando uma matriz metálica rosqueável com 8 mm de diâmetro por 10 mm de altura.

O pó de cerâmica foi aglutinado com o líquido modelador fornecido pelo fabricante e condensado por vibração na matriz metálica, sendo o excesso de líquido aglutinante removido com papel absorvente. Uma pressão de 10 kgf foi aplicada sobre a cerâmica para promover compactação da mistura. O cilindro de cerâmica obtido sob compressão foi removido da matriz e levado ao forno Dekema (Austromatic- Dekema, Germany) para o procedimento de sinterização da cerâmica, seguindo as instruções do fabricante: (1) temperatura inicial de

575^o C; (2) tempo de secagem de 3 minutos; (3) tempo de elevação da temperatura de 55^o C/minuto; (4) tempo de subida de 2 minutos; e, (5) temperatura final de 910^o C, com vácuo por 1 minuto.

Após o esfriamento, o cilindro de cerâmica foi seccionado com disco flexível diamantado dupla face (KG Sorensen, Brasil) em baixa rotação sob refrigeração, para obter um disco em cerâmica com espessura próxima de 2 mm. Em seguida foram submetidos ao acabamento, com lixas d'água de granulação decrescente (180, 320, 400, 600 e 1200) numa politriz metalográfica (Metalserv, England) até atingir a espessura de 2 mm. Os discos com 8 mm de diâmetro na espessura de 2 mm foram finalizados com autoglazeamento na temperatura final de 890^o C, sem vácuo (Figura 1a). Todas as medidas foram aferidas com paquímetro digital com precisão de 0,01 mm (Starrett, Elmhurt, IL, EUA).

Em seguida, os discos cerâmicos foram submetidos à limpeza em ultrassom (Thornton – T 07) por 6 minutos, antes do procedimento de aplicação do agente cimentante. Foram confeccionados 3 discos cerâmicos na espessura de 2 mm.

2 – Obtenção dos corpos-de-prova dos cimentos resinosos

As pastas base e catalizadora do cimento resinoso Variolink II (Ivoclar Vivadent AG, Shann Liechtenstein) foram automaticamente dosadas pela seringa dispensável do sistema, em proporções com volumes equivalentes. Para o cimento resinoso Enforce (Dentsply Indústria e Comércio Ltda, Petrópolis, Brasil) as pastas base e catalizadora foram dosadas por volume em proporções equivalentes. A mistura manual das pastas foi realizada por 10 segundos (Variolink II) e 20 segundos (Enforce), com auxílio de espátula n° 24 em bloco de papel descartável, de acordo com as instruções do fabricante. A seguir, os cimentos resinosos foram inseridos com uma espátula numa cavidade com 5 mm de diâmetro por 1 mm de espessura, no centro de uma matriz de nylon, com 40 mm de diâmetro por 10 mm de altura. Um rebaixo no centro do cilindro de 0,5 mm de profundidade por 8 mm de diâmetro permitiu a adaptação dos discos de cerâmica sobre o cimento resinoso. (Figura 1b). Para evitar o contato do cimento com a cerâmica, uma tira de poliéster foi colocada sobre o cimento resinoso e a cerâmica. A matriz de nylon foi previamente revestida com tinta preta (Colorgin Spray, Sherwin-Willians do Brasil), a fim de evitar que a luz interferisse na polimerização adicional do cimento (Figura 1b). Assim foi possível supor que a luz foi transmitida somente para a cerâmica e cimento resinoso. Todos os procedimentos foram realizados em temperatura ambiente (23°C ±1°C) e umidade controlada (acima de 30% de umidade relativa) conforme especificação da ISO 4049 (The International Organization for Standardization).

Após a colocação do cimento na cavidade da matriz, o disco de cerâmica com 8 mm de diâmetro por 2 mm de espessura foi posicionado no rebaixo da matriz com 8 mm de diâmetro por 0,5 mm de profundidade (Figura 1b). A seguir, as pontas dos fotopolimerizadores XL 2500 (3M ESPE, St. Paulo, MN, USA) e LED – Ultrablue Is (D.M.C. Equipamentos Ltda) foram posicionadas em contato a cerâmica, numa angulação de 90^o mantida por um suporte que permitiu deixar a ponta do aparelho perpendicular à superfície da cerâmica e, conseqüentemente, com o cimento resinoso e fotoativar por 40 segundos (Figura 1b). A intensidade de luz foi de 610 mW/cm^2 para o LED – Ultrablue Is e 615 mW/cm^2 para o XL 2500, sendo constantemente monitorada por radiômetro portátil Model 100 Curing Radiometer (Demetron Research Corporation, Danburry, CT, USA). O aparelho para fotoativação foi ligado a corrente elétrica por meio de um estabilizador de voltagem, com o intuito de minimizar possíveis oscilações na corrente elétrica, que pudessem afetar significantemente a intensidade de luz final do dispositivo.

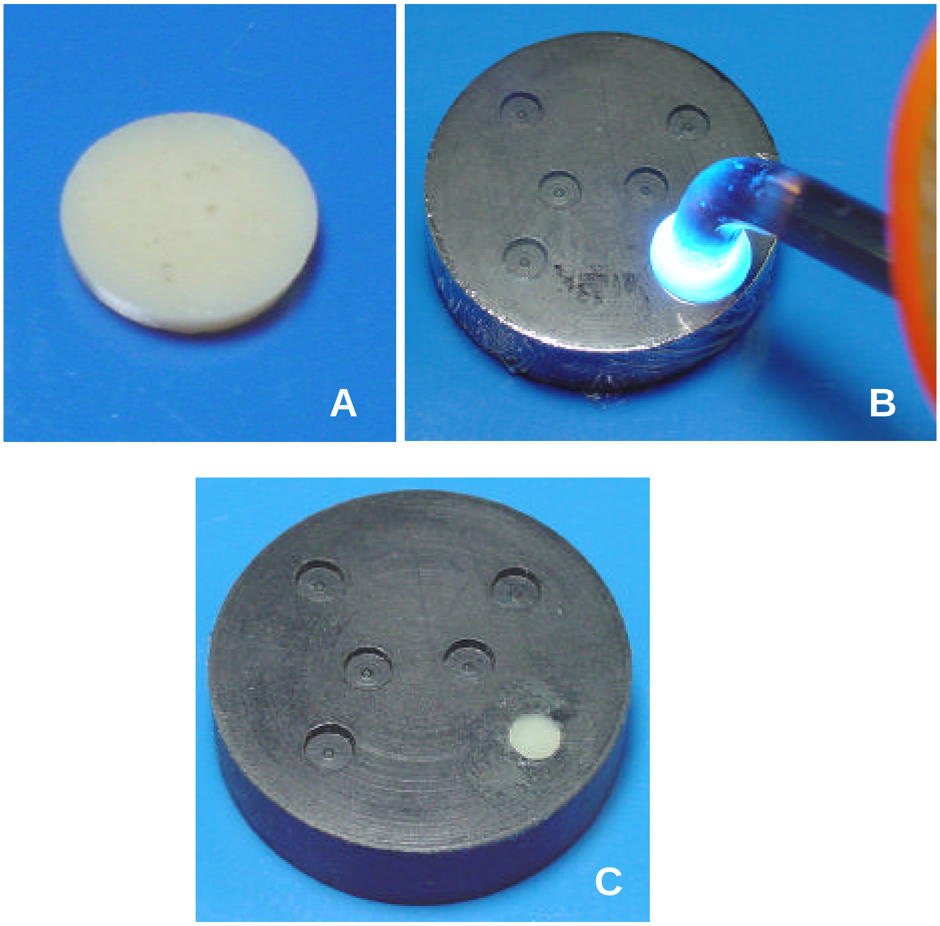

Figura 1 – (a) Disco de cerâmica com 8 mm de diâmetro por 2 mm de espessura; (b) – cimento resinoso sendo fotoativado através da espessura da cerâmica; e, (c) – amostra em forma de disco, obtido a partir da polimerização do cimento resinoso.

Os corpos-de-prova (Figura 1c) para o cimento resinoso Variolink II foram divididos em 8 grupos de 5 amostras cada, conforme o tipo de aparelho fotoativador e cor utilizados neste estudo com interposição da cerâmica feldspática (Quadro I). O grupo controle foi obtido somente com o cimento resinoso com 5 mm de diâmetro por 1 mm de espessura, sem interposição da cerâmica para cada aparelho fotoativador, tipo de cimento e cor, perfazendo um total de 8 grupos de 5 amostras cada, totalizando 80 corpos-de-prova para o cimento resinoso Variolink II, com e sem interposição da cerâmica. Para o cimento resinoso Enforce, seguiu-se a mesma divisão do cimento resinoso Variolink II, somente para as cores B1, A2 e Branco Opaco, perfazendo um total de 6 grupos de 5 amostras cada, totalizando 60 corpos-de-prova, com e sem interposição da cerâmica (Quadro II).

Quadro I – Descrição da divisão dos grupos com os respectivos aparelhos fotoativadores, cimento resinoso Variolink II e cor, que foram utilizados neste estudo com e sem interposição da cerâmica feldspatica.

GRUPO	(n)	Aparelho Fotoativador	Cimento Resinoso	Cor
1	05	XL 2500	Variolink II	XL branco transparente
2	05	XL 2500	Variolink II	Amarelo 210 (A3)
3	05	XL 2500	Variolink II	A2
4	05	XL 2500	Variolink II	BrancoOpaco
5	05	LED - Ultrablue Is	Variolink II	XL branco transparente
6	05	LED - Ultrablue Is	Variolink II	Amarelo 210 (A3)
7	05	LED - Ultrablue Is	Variolink II	A2
8	05	LED - Ultrablue Is	Variolink II	BrancoOpaco

Quadro II – Descrição da divisão dos grupos com os respectivos aparelhos fotoativadores, cimento resinoso Enforce e cor, que foram utilizados neste estudo com e sem interposição da cerâmica feldspática.

GRUPO	(n)	Aparelho Fotoativador	Cimento Resinoso	Cor
1	05	XL 2500	Enforce	B1
2	05	XL 2500	Enforce	A2
3	05	XL 2500	Enforce	Branco Opaco
4	05	LED - Ultrablue Is	Enforce	B1
5	05	LED - Ultrablue Is	Enforce	A2
6	05	LED - Ultrablue Is	Enforce	Branco Opaco

Após a confecção, os corpos-de-prova foram devidamente identificados e armazenados em ambiente escuro numa estufa a 37^{o} C e 100 % de umidade relativa, por 24 horas.

3 – Determinação da dureza Knoop.

Decorrido os períodos de armazenagem, os corpos-de-prova para leitura de dureza Knoop foram fixados em uma matriz pré-moldada em resina acrílica incluída em tubo de PVC. Os discos foram adaptados nos espaços previamente moldados. Eventuais desajustes foram preenchidos pela adição de cera pegajosa plastificada previamente à colocação dos discos. Após a fixação dos discos, o conjunto foi levado até uma politriz metalográfica para acabamento e polimento. Lixas d’água foram utilizadas a partir de granulação decrescente (180, 320, 400, 600 e 1200) até a obtenção de polimento satisfatório para a leitura de microdureza. Cada matriz com discos fixados foi utilizada por 4 vezes para fins de padronização de tamanho de disco.

O ensaio de dureza Knoop foi realizado no aparelho HMV – 2 (Shimadzu, Tokyo, Japan) (Figura 2), calibrado para uma carga de 50 gramas, atuando por 15 segundos. Foram realizadas cinco penetrações em cada corpo-de-prova a uma distância de 100 µm da região de superfície externa do corpo-de-prova, totalizando 200 penetrações para o cimento resinoso Variolink II, com interposição da cerâmica. Duzentas penetrações foram realizadas para o cimento resinoso Variolink II sem interposição da cerâmica. Com auxílio de um microscópio mensurador acoplado ao aparelho, as dimensões das penetrações foram medidas e a dureza Knoop determinada através da seguinte fórmula:

$$\mathbf{DK= \frac{14229P}{d^2}}$$

Fórmula para cálculo da Dureza Knoop:

DK - Dureza Knoop.

P - Carga aplicada em gf (gramas-força)

d^2 - Comprimento da diagonal maior do losango em µm.

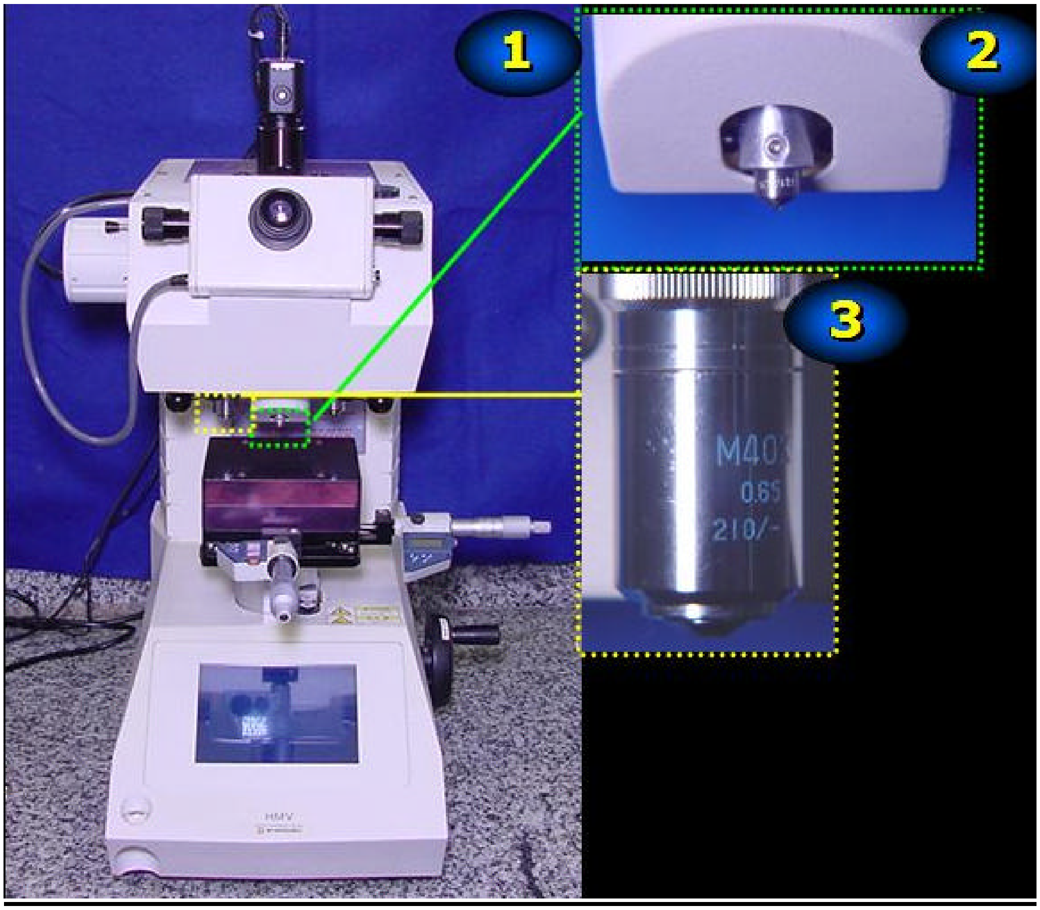

Figura 2 – (1) Aparelho HMV-2 para ensaio de dureza; (2) – ponta edentadora utilizada para aplicação de carga para dureza Knoop; e, (3) – lente de aumento de 40X utilizada na leitura de dureza.

Para cada corpo-de-prova do cimento resinoso Enforce, 4 médias de dureza Knoop foram obtidas, de acordo com a profundidade de leitura. As médias correspondiam a microdureza na profundidade de 100 µm da superfície externa do disco. As médias aritméticas de acordo com a profundidade resultavam de três leituras, com 1,0 mm de distância entre elas, sendo que a edentação central deveria coincidir com o eixo de menor comprimento que passava pelo centro do corpo-de-prova (Figura 3). A iluminação do aparelho foi reduzida e todas as edentações foram feitas previamente à leitura. Essa medida foi tomada para evitar que o tempo estipulado para leitura mediata (de 15 minutos) fosse extrapolado.

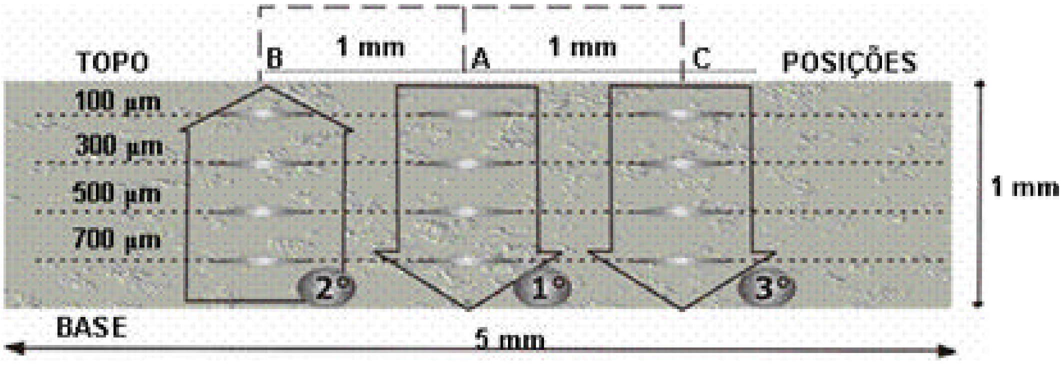

Figura 3 - Representação esquemática da seqüência utilizada na leitura de microdureza. Na terceira seqüência, metade dos corpos-de-prova, para cada grupo, eram analisados no sentido contrário ao exposto na figura.

As penetrações foram realizadas de acordo com uma seqüência pré-estabelecida (Figura 3). Iniciava-se as edentações a partir da superfície da posição A (central) o início da leitura na posição B era realizado a partir do inferior. Na posição C metade dos corpos-de-prova de cada grupo sofriam edentações a partir da superfície superior enquanto na outra metade a seqüência de penetrações era realizada a partir da superfície inferior. Portanto, quatro médias de dureza para cada corpo-de-prova foram criadas, como resultado de doze leituras, quando foi empregado o cimento Enforce. Um total de 360 penetrações foram feitas com interposição da cerâmica e 360 sem interposição da cerâmica.

Os dados foram tabulados e analisados em software estatístico Sigmastat versão 3.01 (SPSS Inc. Headquarters, Chicago, Illinois, EUA) submetidos ao teste de normalidade (Kolmogorov-Smirnov) e ao teste de igualdade de variância (Levene median test). Análise de variância em esquema parcela subdividida, com fatorial na parcela (tempo pós-ativação X modo de ativação) e subparcela representada pela profundidade (4 níveis) para o cimento resinoso Enforce e (1nível) para o cimento resinoso Variolink II foi utilizada para identificar possíveis interações entre os fatores investigados. O método de Tukey para comparações múltiplas foi aplicado para realizar as comparações entre as médias, e verificar quais grupos diferiam entre si. O nível de significância utilizado em todos os testes foi de 5% (α=0,05).