EVANDRO PIVA

EFEITO DA ESPESSURA DE UMA CERÂMICA FELDPÁTICA E DA DOSAGEM DE ENERGIA COM DIFERENTES APARELHOS FOTOATIVADORES SOBRE A MICRODUREZA DE CIMENTOS RESINOSOS DE DUPLA ATIVAÇÃO

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

PIRACICABA
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Orientador: Prof. Dr. Lourenço Correr Sobrinho

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A Comissão Julgadora dos trabalhos de Defesa de Tese de DOUTORADO, em sessão pública realizada em 16 de Abril de 2004, considerou o candidato EVANDRO PIVA aprovado.

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2. Prof. Dr. GILBERTO ANTÔNIO BORGES _____

3. Prof. Dr. MANOEL DAMIÃO SOUSA NETO _____

4. Prof. Dr. MARIO FERNANDO DE GOES _____

5. Prof. Dr. SIMONIDES CONSANI _____

DEDICO ESTE TRABALHO

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À minha noiva Adriana,

Ao meu irmão Juliano...

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“Aquilo que desejamos já nos foi concedido e está dentro de nós”.

Masuharu Taniguchi

SUMÁRIO

RESUMO

Os cimentos resinosos de dupla ativação são geralmente utilizados para cimentação de restaurações indiretas. Esses materiais podem tomar presa através da ativação química apenas ou ativação foto/química, também chamada de dupla ativação. Para esses cimentos, um maior grau de conversão e conseqüentemente melhores propriedades mecânicas são obtidas quando o procedimento de fotoativação é utilizado. Entretanto, em determinadas situações clínicas é necessário que a fotoativação seja feita indiretamente, através do material restaurador. No presente trabalho foram realizados dois experimentos baseados na metodologia de dureza Knoop, com aplicação de carga de 50g e tempo de aplicação de carga de 15 segundos. No primeiro experimento, foi avaliada a influência de três diferentes espessuras de cerâmica feldspática (0,7, 1,4 e 2,0 mm) sobre a microdureza em diferentes profundidades de um cimento resinoso de dupla ativação (RelyX ARC, 3M ESPE). O teste de microdureza foi realizado após dois intervalos pós-ativação do cimento; entre 10-20 minutos (microdureza imediata) e após 24 horas de armazenagem a seco e 37°C. Os resultados demonstraram que após o cimento sofrer irradiação indireta através das diferentes espessuras, os valores de microdureza tendiam a diminuir com aumento da espessura da cerâmica. O modelo de regressão linear demonstrou que uma diminuição da microdureza pode ser estimada em função do aumento da profundidade onde a leitura de microdureza era imediatamente realizada no corpo-de-prova (R^2=0.99). O modo de fotoativação direta demonstrou um grau de microdureza estatisticamente maior que os demais modo de ativação para todas as profundidades avaliadas ($p<0.05$). Equivalentes microdurezas foram encontradas entre as diferentes profundidades de leitura para o mesmo grupo, quando os espécimes foram armazenados por 24h. O objetivo do segundo experimento foi avaliar a eficiência de três aparelhos para fotoativação de compósitos (Halógena, LED e arco de plasma de xenônio) na fotoativação indireta

de um cimento resinoso de dupla ativação (Enforce, Dentsply). Novamente a metodologia para o ensaio de microdureza foi aplicada para avaliar o efeito de diferentes dosagens de energia em cimentos resinosos. O tempo de irradiação foi ajustado de acordo com a intensidade de cada aparelho, de modo a caracterizar dosagem energética. As dosagens ensaiadas foram 48 joules.cm^{-2} (Ix2T), 24 joules.cm^{-2} com irradiância máxima (IxT), e 24 joules.cm^{-2} com metade da irradiância e dobro de tempo de irradiação que o anterior (I/2x2T). As fontes LED e halógena mostraram resultados semelhantes quando usadas de acordo com cada dosagem de energia indiretamente aplicadas. Em duas situações (Ix2T e I/2x2T) o LED foi estatisticamente superior ao arco de plasma de xenônio ($p<0,05$). A fotoativação através de estrutura cerâmica, com o aparelho LED e Halógena usados com máxima intensidade e por tempo prolongado, demonstrou-se satisfatória.

Palavras Chave:

1- Cimentos de resina; 2- Cimentos dentários; 3- Dureza; 4- Cerâmica odontológica; 5- Materiais dentários; 6- Luz; 7- Efeitos de radiação

ABSTRACT

Commonly dual-cured resin-based cements are used for adhesive and luting procedures, mainly for placement of indirect restoration. These cements can be cured by two modes, by chemical activation or by photo/chemical activation, which that has been show better degree of conversion and mechanical properties than former. In some clinical circumstances photoactivation should be performed indirectly through dental ceramics. This work intended to test efficiency of light photoactivation units and influence ceramic thickness upon resin cement microhardness. Two investigations were performed using a universal indenter setted for 50 g per 15 s load. On first work, was verified the influence of three ceramic thickness (0.7, 1.4 and 2.0 mm) upon microhardness at different depths of a dual-cured resin cement (RelyX ARC, 3M ESPE). Knoop Hardness test was performed after two times post-activation of resin cement; between 10-20 minutes (immediately) and approximately 24 hours after (storage at 37°C). Results showed that after indirect irradiation trough ceramics microhardness showed a trend to decrease according with ceramic thickness increase. The decrease of resin cement hardness was well predicted by increase of ceramic thickness in immediate microhardness when photoactivation through ceramic was performed (R^2=0.99). Direct photo activation showed significantly higher microhardness for all depths on immediate test than others groups ($p<0.05$). After storage for 24 hours microhardness was generally equivalent between depths for same group. The aim of second work was to evaluate effectiveness of three light photoactivation units (Halogen, LED and xenon plasm arc) on indirect photoactivation of a dual-cured resin-based cement (Enforce, Dentsply). Knoop hardness methodology was used. Energy dosages were created from adjustments of irradiation time according to irradiance of each device. Energy dosage tested were 48 joules.cm^{-2} (Ix2T), 24 joules.cm^{-2} with maximum irradiance (IxT) and 24 joules.cm^{-2} (I/2x2T) with half irradiance and double irradiation time. LED and Halogen units showed similar

microhardenss when energy dosage was applied indirectly. LED was statistically superior ($p<0.05$) than xenon plasm arc device in two activation modes (Ix2T and I/2x2T). Photoactivation through ceramic for elapsed time and with maximum intensity showed to be a reliable procedure for LED and Halogen sources.

Keywords: 1- Resin cements; 2- Dental Cements; 3- Hardness; 4- Dental porcelain; 5- Dental materials; 6- Light; 7- Radiation effects.

INTRODUÇÃO GERAL

Tradicionalmente, o cimento de fostato de zinco é um dos materiais mais difundidos e utilizado na cimentação de elementos protéticos (Rosenstiel *et al.*, 1998). Esse material pode ser utilizado para a cimentação de próteses com infraestrutura metálica, ou inclusive para alguns sistemas cerâmicos livre de metal. Entretanto, algumas limitações desse material como a falta de adesão, solubilidade e limitações estéticas, estimularam o desenvolvimento de outros materiais para cimentação como cimentos de ionômero de vidro e cimentos resinosos (Rosenstiel *et al.*, 1998; Diaz-Arnold *et al.*, 1999).

Foram criados os cimentos de ionômero de vidro e suas variações, como cimentos de ionômero de vidro modificado por resina e resinas modificadas por poliácidos (McLean *et al.*, 1994). Outra alternativa aos cimentos de fosfato de zinco, são os cimentos resinos. Esses materiais têm como vantagens, principalmente a integração adesiva aos substratos, baixa solubidade, facilidade de manipulação e estética compatíveis com as restaurações cerâmicas livre de metal. Em determinadas situações a presença de cimento resinoso aumenta a resistência à fratura por fadiga de coroas cerâmicas livre de metal, quando comparado com cimento de ionômero de vidro e cimento de fosfato de zinco (Groten & Probster, 1997).

De acordo com a normatização da ISO 4049 (*The International Organization for Standardization*), todos os cimentos resinosos para fixação e cimentação de peças protéticas, pertencem ao *Tipo 2* de materiais restauradores à base de polímeros. Portanto, 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; C*lasse 2,* quando a energia é fornecida a partir de fonte foto-ativadora para uso intra-oral (*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 foto iniciador (canforoquinona), que também está presente nos compósitos restauradores fotoativados (Peters & Meiers, 1996).

Apesar da variedade de cimentos disponíveis atualmente, não há um cimento que seja ideal para todas as situações clínicas (Rosenstiel *et al.*, 1998). 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 livre de metal, são comumente cimentadas com cimentos resinosos de dupla ativação. A vantagem da escolha de cimento ativado quimicamente é baseada no processo de polimerização, que acontecerá mesmo em áreas onde a exposição à luz é crítica. Neste contexto, o modo de ativação química irá teoricamente garantir uma reação de presa satisfatória em locais de difícil acesso a luz. Entretanto, a fotoativação desses materiais é um fator essencial na reação de polimerização, melhorando de modo geral as propriedades mecânicas e estéticas dos cimentos resinosos de dupla ativação (Watts & Cash, 1994).

Quando a fotoativação do cimento resinoso é realizada indiretamente, alguns aspectos devem ser levados em consideração. À medida que há um aumento na espessura do material restaurador, a dispersão e a absorção da luz aumenta, reduzindo dessa maneira a quantidade de energia emergente do dispositivo de fotoativação, que atinge a camada de cimento (Watts & Cash, 1994). Outros estudos investigaram o efeito da cor, opacidade (Linden *et al.*, 1991) e espessura da estrutura cerâmica sobre o grau de conversão de cimentos resinosos, medida indiretamente através da microdureza (Blackman *et al.*, 1990; Uctasli *et al.*, 1994). As evidências comprovam que há um efeito atenuador proporcional à espessura da cerâmica (Warren, 1990) e da opacidade do material

podendo acarretar uma menor microdureza para cimentos resinosos (Uctasli *et al.*, 1994).

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 (Rueggeberg *et al.*, 1994; Halvorson *et al.*, 2002).

Em trabalho recente Braga *et al.*, 2002), após avaliar diferentes cimentos resinosos em função do método de polimerização, não encontrou correlação entre microdureza e resistência flexural. De acordo com esses autores, fatores como o tipo e tamanho de partículas também podem interferir nos resultados, bem como os tipos de monômeros que constituem a cadeia polimérica (Asmussen & Peutzfeldt, 1998) e a concentração dos inibidores (Ferracane & Greener, 1986).

Os cimentos de ativação dupla demonstram melhora em suas propriedades mecânicas quando for empregada fotoativação adequada, do que quando são somente polimerizados pelo modo autopolimerizável (Cardash *et al.*, 1993; Uctasli *et al.*, 1994; Darr & Jacobsen, 1995; el-Mowafy *et al.*, 1999; Hofmann *et al.*, 2001; Foxton *et al.*, 2003). Uma menor micordureza pode ser sinôniomo de polimerização incompleta dos compósitos resinos para cimentação causa diminuição nas propriedades mecânicas do cimento, além de aumentar a sorpção de água (Uctasli, 1991). Além disso, a presença de moléculas não polimerizadas podem ser desprendidas do material, causando inflamação tecidual (DeWald & Ferracane, 1987). 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 a partir do filamento de tungstênio. Entretanto, outras tecnologias estão disponíveis, entre elas estão o arco de plasma de Xenônio (Hofmann *et al.*, 2000; Peutzfeldt *et al.*, 2000) e a luz emitida por diodos – LED (Jandt *et al.*, 2000; Stahl *et al.*, 2000; Kurachi *et al.*,

2001). Esses sistemas estão se tornando cada vez mais difundidos e utilizados. Contudo, muitas dúvidas permanecem a respeito da efetividade de fotoativação indireta desses dispositivos e sobre o efeito nas propriedades finais dos cimentos resinosos irradiados de diferentes formas através da estrutura dentária.

Além disso, torna-se importante o conhecimento científico sobre o assunto para direcionar as atividades clínicas, através de critérios para escolha de materiais e técnicas que possam ser empregados, disponibilizando informações relevantes para melhorar a performance e a longevidade clínica das restaurações indiretas.

PROPOSIÇÃO

O presente estudo teve como objetivos;

1- Avaliar o efeito de um método de fotoativação através de diferentes espessuras de cerâmica, sobre a microdureza de um cimento resinoso de dupla ativação após dois intervalos de tempo pós-ativação.

2- Verificar, através da dureza do cimento resinoso, a eficiência de três dispositivos para fotoativação de compósitos usados com equivalentes dosagens de energia, na irradiação indireta de cimento resinoso de dupla ativação.

CAPÍTULO I

(Artigo submetido ao periódico *The Journal of Prosthetic Dentistry*)

Influence of ceramic thickness on mediate and 24h post-irradiation microhardness of a dual-cured resin-based cement

Purpose: The aim of this study was to evaluate the influence of different ceramic thickness upon dual cure resin-based cement microhardness profile at two different post-cure intervals: immediately and 24 hours post-activation. **Materials and Methods:** Resin cement RelyX ARC shade A3 (3M ESPE) was used to form disk shaped specimens. Feldspatic ceramic Duceram Plus (Degudent) was used to produce discs of three different thickness: 0.7, 1.4 and 2.0 mm. These disks were used for the indirect photoactivation mode, through ceramic (Ptc). Additionally, direct photoactivation (DPa) and chemical activation (control) modes were used. A halogen light unit, XL 2500 (3M ESPE) was used with irradiance ≤650 mW/cm^2 during 40 s. Since five activation modes of resin cement were investigated, ten groups (n=10) were produced: five for immediate Knoop Hardness (KH) test and the remaining groups tested after 24-hours dry storage at 37° C. The KH was evaluated at different depths of the cross-sectional area of resin cement specimens. Factorial ANOVA with split plot and

Tukey's test were applied at significant level of $p<0.05$. **Results:** Statistically significant interaction was found between all factors tested individually and combined ($p<0.05$). The decrease of resin cement hardness was well predicted as a function of increasing of the resin cement depth in immediate microhardness, when photoactivation through ceramic was performed ($R^2=0.99$). Direct photo activation showed significantly higher microhardness for all depths on immediate test than others groups ($p<0.05$). The cement hardness after 24 hours was statistically higher than immediate values ($p<0.05$) for Ptc with 1.4 and 2.0 mm ceramic thickness. **Conclusion:** Within the limitation of this study, when immediately tested the cement hardness was progressively reduced by ceramic thickness increasing. However the 24-hours storage eliminated the majority of the differences in microhardness observed soon after activation of cement.

KEYWORDS: Resin cements, composite resin, hardness, dental porcelain, dental materials, radiation effects.

Introduction

Bonding to traditional silica based ceramics is a predictable procedure yielding durable results when certain guidelines are followed[1]. Adhesive technique may be applied in veeners, inlays, onlays, full coverage restorations[2] and

prosthesis. Resin-based composites are the first choice material for the adhesive luting of ceramic restorations[3]. The success of tooth-colored ceramic depends on a great extent on the resin cement, which must ensure an effective bond between porcelain restorations and tooth structure and guarantee the final marginal adaptation[4].

Resin-based cements can be classified according to activation method as self cured, light cured and dual cured cements. When compared with light cured cements self-cured have some disadvantages. Mixing of these materials bears the risk of entrapping air bubbles that may create voids in the adhesive interface. “Photo-activated only” resin-based cements are easy to use and better control of set reaction is found. This feature allows adequate work time for adjustments and margins finishing until photoactivation. Thus, after light-activation of resin cement, the restoration can be finished without further delay. On the other hand, when chemical-activation reaction is present, materials uniformly set even at the bottom of deep cavities, where access for curing light is limited. Moreover, for chemically activated resin-based cements, the conversion degree is independent of photoactivation effectiveness.

The dual-cure resin-based cements were developed to combine the most desirable properties of the chemical-activated and the light-activated materials and thereby providing adequate curing in deeper areas when photo-activation effectiveness is doubtful and provides quick hardness when light irradiated. Dual cure cements, which are typically used for cementation of inlays, onlays and all ceramic crowns, cure chemically upon mixing of a base and catalyst components

self-cure mode) and also when exposed to light from a light-curing unit (dual-cure or light/chemical activation mode)[6].

It has been reported that self-cure alone is insufficient for dual-cure cements to achieve maximum hardening[6-8]. This fact can be a concern when thick ceramic inlays are luted[6]. Thus, indirect photo-activation of resin cement through ceramic is a critical procedure, since mechanical properties improvements and hardening of resin cements depends on the efficacy of this process.

The content of amine, peroxide and inhibitor are adjusted for reaction kinetics, affecting the setting time[9]. Working time should be long enough to allow careful placement of the restoration and excess removal before the cement sets. Moreover, the speed of the setting reaction has to be adjusted to meet conflicting demands. It must be slow enough to allow a sufficient working time and quick enough to permit finishing of the restoration in a timely manner and fashion. Setting time should be as short as possible to guarantee sufficient strength, to allow minor adjustments by the dentist, and also to permit the patient to masticate normally, without any risk of displacement or fracture[10].

Several studies investigated the effect of ceramic color, opacity[11] and thickness on the polymerization of resin cements measured by microhardness[12,13]. There is an light attenuation effect according to increasing of thickness and opacity[13]. It has been showed that thickness of porcelain instead of the opacity or color, was the primary factor that affects light transmission[14,15].

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) that different activation modes will not influence the microhardness of a dual-cured resin cement at different depths; 2) that two post-activation times (immediately and 24-hours post-irradiation) will exhibit similar performances in relation to the microhardness of the tested resin cement.

Materials and Methods

Feldspatic ceramic material, Duceram Plus (Degudent), shade dentin A3, was condensed in a metallic mold to form a cylindrical specimen that was fired in ceramic furnance (Austromat M, Dekema Austromat-Keramiköfen, Freilassing, Germany), according to manufacture's instructions. One cylinder of 8.0 (±0.01) mm diameter was obtained. This specimen was sectioned with diamond disc at low speed, under water refrigeration to obtain discs in three thicknesses (0.7, 1.4 and 2.0 mm), which were submitted to finishing and glaze firing. One specimen for each thickness was selected for use in the present study.

The resin cement RelyX ARC (3M ESPE, St. Paul, MN, USA), shade A3, was mixed according to manufacturers' directions and inserted in nylon mold with a centered hole with 5.0 (±0.01) mm diameter and 1.0 (±0.01) mm deep. The nylon mold 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[13]. 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)[16].

The resin cement was chemically activated (self-cured) or photo/chemical activated (dual-cured). When photo/chemical activation was used, the resin cement was light irradiated by two modes; by direct photo-activation (DPa) or photo-activated through ceramic (Ptc activation mode). In this last model, the ceramic discs were interposed between the tip of light guide of light photo-activation unit (LPU) and the polyester film that covered the resin cement before irradiation. The resin cement was photo-activated by tungsten halogen light unit XL 2500, (3M-ESPE, St. Paul, MN, USA) with irradiance ≤650 mW/cm^2, during 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). The light photoactivation unit was connected to an electrical voltage stabilizer and supported by an apparatus to allow standardization (90 degree) in tip light guide placement for irradiation.

Two post-cured times were investigated: 1- immediate, specimens were evaluated between 10-20 minutes after photo-activation or between 20-30 minutes for chemically activated group (self-cured mode). 2– 24 hours post-cure, specimens were stored in bottles in dry and dark conditions at 37°C for approximately 24 hours.

Ten groups (n=10) were tested. According to activation mode and time post-activation before microhardness readings, groups were labeled as follow;

Mode DPa; when direct photoactivation was performed, without ceramic interposed. Two groups were created with this mode for Knoop Hardness (KH) readings: immediately, between 10 to 20 minutes post-activation by light irradiation (DPa-immed) or approximately after 24 hours post-activation (DPa-24h).

Mode Ptc0.7mm: The ceramic disc with 0.7 mm thickness was interposed between resin cement and light tip of LPU. Then indirect photo activation trough ceramic was performed. Groups according to time post-activation of resin cement. Ptc0.7mm-immed and Ptc0.7mm-24h.

Mode Ptc1.4mm: Similar to group Ptc0.7mm, but with ceramic disc with 1.4 mm. groups; Ptc1.4mm-immed and Ptc1.4mm-24h

Mode Ptc2.0mm: Similar to group Ptc0.7mm, but with ceramic disc with 2.0 mm. Groups; Ptc1.4mm-immed and Ptc1.4mm-24h

Mode Cat: the resin cement was used in chemically activated mode (Cat). Cement mixing was performed in a dark room with a red light filter (for x-ray film revelation) to avoid photoinitiator sensitisation.. This group was used as a control. Two groups were created; Cat-immed and Cat-24h. Ten minutes were waited for cement hardening before removing the specimens from the mold

The preliminary procedures before indentation were completed until 10 minutes after catalyst and base past mixture. Specimens were fixed with in a pre-molded acrylic mold, 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 according to different depths, at 100, 300, 500, and 700 μm from surface irradiated. Three indentations were made for each depth, with 1 mm distance between them and means were calculated for each depth, of same specimen.

Measurements were manually 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 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) and sub parcel represented by factor depth 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

Statistically significant interaction between factors investigated alone (time post-irradiation, activation mode and depth of lecture) or associated by two factor or three factors ($p<0.01$) were found. Table 1 shows comparisons among groups and statistically significant differences according to Tukey's test.

For all comparisons between groups for each depth, always KH of layers for DPa-immed showed statistically superior hardness compared to Ptc2.0mm-immed and Cat-immed ($p<0.05$). At the 24 hours post-cure time generally less differences were observed among groups for same depth, when compared to those KH found for immediate groups. Microhardness of Cat-24h was statistically lower than others groups ($p<0.05$).

When photoactivation was performed though ceramic, the KH means for groups Ptc1.4mm-24h and Ptc1.4mm-24h were statistically superior to correspondent depth in immediate groups ($p<0.05$).

When comparisons were made between post-cure times of each activation mode, it was observed that KH numbers showed a trend to decrease according to increasing of ceramic layer (Figure 1). According to linear regression the Knoop hardness of resin-based cement tested after immediate post-cure time can be well predictable for thickness of ceramic through that photoactivation is performed ($R^2=0.99$ for Ptc0.7mm, Ptc1.4mm and Ptc2.0mm), being this evidence statistically significant ($p<0.01$). In DPa-immed group the predictive coefficient was $R^2=0.95$ ($p<0.01$).

Photoactivations modes evaluated after 24 hours storage generally do not showed statistically significant regression, only Ptc0.7mm-24h showed a weak linear predictive model with R^2=0.59 (p=0.02). Ptc2.0mm-24h and Cat-24h groups showed fit only for quadratic regression (p<0.01).

Discussion

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

When discussing about activation mode through different layers of ceramics it has been showed that the amount of total transmission is the sum of directly transmitted light and diffuse light transmission by scattering[17]. Optical properties of ceramics, like shade determines translucency of dental ceramic that is inversely related with sample thickness[17]. Thus light emitted for photoactivation unit will undergo higher attenuation when thicker ceramic layers are used. In the present study this relationship was clearly identified by immediate hardness decrease according to progressive increase of ceramic discs (groups) for same depth.

With light curing, it can be anticipated that the microhardness of the composite will reduce with increasing depth of resin. It can be explained by an increase of attenuation effect, according to increasing of thickness and opacity[13]. As useable curing wavelengths are attenuated in the ceramic and resin cement, less camphorquinone will be activated[18,19]. Additionally, with light dissipation,

probably, there will be losing of the useful wavelength of activation light provided by the photoactivator device when it emerges of ceramic disc, according to spectral irradiance. However, this hypothesis should need further investigations.

In present study Knoop hardness of resin-based cement tested immediately after activation has showed a progressive decreasing as a function of increasing of cross-sectional depth where hardness tests were performed (R^2 range of 0.95 to 0.99). When specimens were stored at dry conditions at 37°C, the linear predictive model generally was not significant Such finding could be related to the influence of storage conditions on microhardness of composites.

Improved degree of conversion after different storage conditions has been showed under Infrared spectroscopy studies[20]. Some studies have demonstrated that for light-activated restorative composites, post-irradiation hardness increases quickly over the first hour and slows to reach a maximum value within 24h[21,22]. This positive effect was clearly observed for chemically activated groups. In this group Knoop hardness increased approximately three times after storage for 24 hours, when comparing to immediate reading of hardness.

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 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.

The manufacturer of resin cement used states that rheological properties were modified by addition of a dimethacrylate polymer that allows improved handling and easy cleanup characteristics. As setting time generally is controlled by inhibitors components[9], probably the hardness of depth of cure by photo-activation process may be affected.

This model of study is important to know resin cement features at the time of first loading, since this is the setting time recommended by manufacturer. Generally immediate loads happen in oral environment after crowns luting procedure. After this time the restoration fixed or cemented will be able to withstand immediate loads in oral environment. Partial setting, with low degree conversion and hence small microhardness can leave to decrease in cohesive bond strength between deeper layers and adhesive bond strength at resin cement/hybrid layer interfase. Afterward, Knoop hardness test at different depth of resin cements can provide parameters to predict clinical performance of all ceramic restorations luted with resin cement.

Based on data of present investigation photo-activation time was insufficient for resin cement hardening under thicker ceramic layer. The exposure times recommended by the manufactures were not enough to compensate light attenuation undergo for ceramic energy transmission [23]. Time of photoactivation for cure advocate by manufacturer should be treated with caution, because sometimes the regular time is not adequate to produce a reliable hardness on different depths of cement[24]. Increasing in ceramic thickness requires increased exposure times for resin curing. Nevertheless even when dual-cured resin based cement is used, the

shear bond strength after 90 minutes was much lower than the maximum strength found after 7 days[10]. For this reason patients should be informed to avoid major force application to the restoration at firsts hours or even first 24 hours after cementation [10,25,26].

The irradiance of light source, the exposure time and light transmission of irradiated material are significant variables that affect the hardness or conversion profile[27]. 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 [9].

One available method to improve degree of cure of resin-based cement is to increase irradiance time[23,24]. For restorative composite resin has been shown that there is an energy-conversion relationship that can be used to predict the conversion profile of composites under variable light-curing conditions[27,28]. It has been showed that irradiation in several direction for an elapsed time provide better hardening for thicker ceramic layers[29].

In this study, the chemical activated reaction of resin cement was insufficient to compensate the attenuation of light by ceramic and obtaining an adequate hardness. Also, the catalyst has the potential of discoloration when dual-cured resin cements are used in anterior porcelain veeners[11]. The real role of the chemical catalyst in the dual cured resin cements after irradiation exposure is controversial[30]. The present study disclosed that chemically induced polymerization of dual cure resins will proceeds after 20-30 minutes post-activation and microhardness became significantly higher after 24 hours storage. However,

despite the manufacture's claiming, there is no evidence of a substantial chemically-induced polymerization of dual cured resin after light exposure is completed[30].

The microhardness of only chemical activation mode of the tested dual-cured resin cement was low in both situation, immediate and 24 hours storage. This evidence are in agreement with others studies[6,15,30] and can be related to the remaining double bonds, that generally is higher in chemical activated composites than in photo activated resin based cements[31].

Low levels of monomers conversion of resin-based luting cements is accompanied by decreasing in mechanical properties, performance characteristics and increased water sorption[13]. Moreover, the presence of residual monomers in poorly polymerized resin may be leached from material, causing tissue irritation[32], affecting the overall biocompatibility of system[33]. Other possible complications of inadequate polymerization include risk of marginal fracture, secondary caries risk, poor color stability and stain uptake. It has also been related adverse chemical interaction between catalytic components of chemically-cured or dual-cured composites with single-step, self-etching adhesive[34-36]. This adverse chemical interaction was a major cause of reductions of bond strength[35]. Dual-cured composites also showed similar adverse interaction when left on the top of adhesives layer for a period of time delay before light activation. Similar condition will occur when partial cure of resin cement is found under deeper layers, until post-cure and chemical reaction proceeds, reduction of adhesive bond strength probably will be expected.

For these reasons photo-activation of these resin-based materials was necessary to maximize strength and rigidity[37]. The dual cured resin-based cement RelyX ARC depended upon photo-activation to achieve higher hardness values[38]. Also, the “unphotopolymerized” conditions lowered the mean modulus of elasticity at 24 hours and at 3 months for Relyx ARC but not for other resin cement tested[37]. The “unphotopolymerized” conditions lowered the flexural strength of the dual-polymerization resin luting agents compared with the “photopolymerized” conditions at 24 hours and at 3 month [37].

The microhardness profile between 100μm layer and 500μm layer observed in group Cat–24h could be attributed to oxygen inhibition at superficial layers of composites. Other investigations found that microhardness and conversion degree trend to increase at the center of the specimen and decrease near the surface[39].

Generally conversion degree correlates well with microhardness,[40,41,39] that will not require complex instrumentation[31]. Once the degree of monomer conversion (polymerization process) obtained by FTIR spectroscopy, commonly is used to know reaction kinetics of polymer-based composites, the microhardness profile is useful in determining the evolution of the mechanical properties during the polymerization process.

Studies of initial microhardness behavior in resin cements used in dual-cure mode, is critic because once chemically activated this reaction will proceeds. The design of the present study allowed the detection of an initial depth of cure profile of the resin cement using Knoop hardness test. Nevertheless, such in vitro physical testing must be assessed in conjunction with other important properties, including

solubility, film thickness, biocompatibility, and handling before material selection[37]. Due to mechanical tests to produce a range of product-specific results and not all products polymerize adequately in every clinical situation [42], it is important guide the choice of a dual-polymerizable cement based on its intended use

There is a demand for more studies about absorbance of ceramic structures and light transmission properties for different materials. Additional investigations will help to optimize the degree of cure when indirect photoactivation through ceramic material is performed. Further investigations in this topic will be helpful to characterization of emergent light profile and the effect on degree of cure of resin cements layers.

Conclusions

Within the limitations of this study the following conclusions may be drawn:

1. The ceramic, in the thickness tested, has the potential to attenuate the light and reduce the cure of resin cement.
2. The increase of ceramic thickness was related with significantly decrease in Knoop hardness values.
3. A improvement of microhardness at deeper layers was found after 24 hours storage

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Table 1 Means and Standard deviations for Knoop Hardness values according with groups, depth of test and post-cure time.

Activation mode (Groups)	RESIN CEMENT DEPTH							
	100µm		300µm		500µm		700µm	
	immed	24 hs	immed	24 hs	immed	24 hs	immed	24 hs
Direct Photo-activation *(DPa)*	47.04(2.42)**a**	46.33(2.73)**a**	47.20(4.29)**a**	47.00(4.12)**a**	43.00(5.38)**a**	46.11(2.85)**a**	40.61(4.98)**a**	*45.45(4.24)**a**
0.7 mm ceramic thickness (Ptc0.7mm)	42.02(4.42)**b**	42.68(2.80)**ab**	38.51(3.53)**b**	*45.04(2.38)**ab**	35.24(4.02)**b**	*46.08(3.25)**a**	32.47(4.05)**b**	*45.11(2.53)**a**
1.4 mm ceramic thickness (Ptc1.4mm)	37.33(3.83)**c**	*42.56(2.14)**ab**	34.44(2.95)**bc**	*42.8(2.3)**ab**	32.81(2.32)**b**	*43.24(2.6)**a**	30.67(3.01)**b**	*42.69(1.91)**a**
2.0 mm ceramic thickness (Ptc2.0mm)	33.51(4.49)**c**	*40.09(3.75)**b**	30.16(3.83)**c**	*42.28(3.48)**b**	26.24(3.53)**c**	*43.51(2.11)**a**	23.76(4.69)**c**	*41.84(3.83)**a**
Chemical Activation (Cat)	9.69(2.56)**d**	*16.38(6.83)**c**	11.29(1.66)**d**	*34.45(3.18)**c**	11.17(2.25)**d**	*34.72(3.52)**b**	11.15(1.71)**d**	*28.13(4.3)**b**

Different letters in columns represents differences statistically significant between groups ($p<0,05$).
(*)The value are statistically higher from that on row (comparisons for same depth) for comparisons of post-cure microhardness test ($p<0,05$).

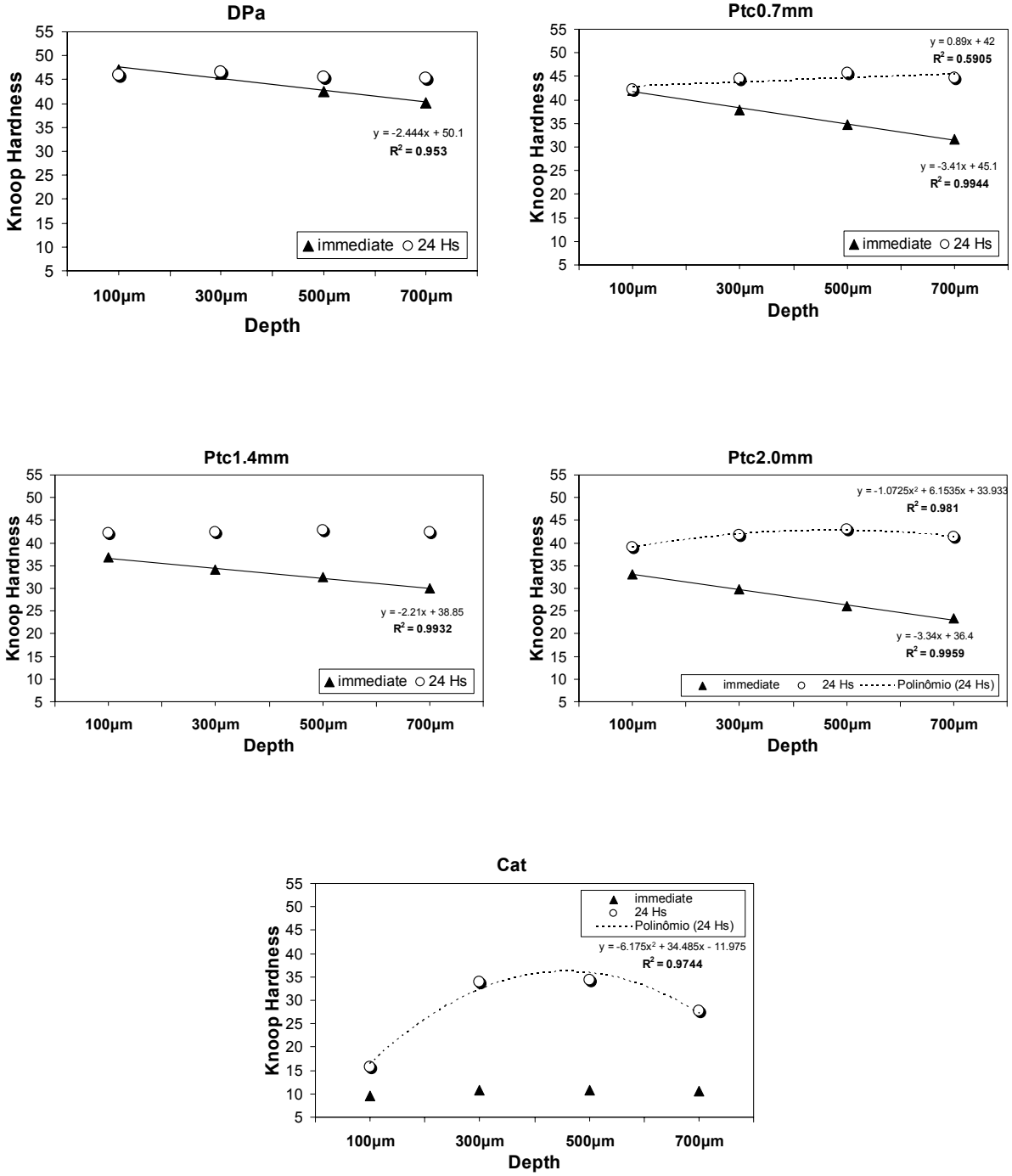

Figure 1 Graphs illustrating profile of microhardness, according to two post-activation time, as a function of depth tested. Statistically significant regressions are plotted ($p<0.05$).

CAPÍTULO 2

(Artigo submetido ao periódico *Operative Dentistry*)

Effect of energetic density with different light sources upon Knoop hardness of dual-cured resin cement

Clinical Relevance

LED and Halogen light photo-activation units has equivalent performance upon microhardness of resin cement irratiated through thick ceramic layer.

Summary

The aim of this study was to evaluate Knoop hardness of a dual-cured resin-based luting cement irradiated, through ceramic substrate. Three ligth photo-activation units (LPUs) were used; tungsten halogen light (HAL= XL 2500, 3M ESPE), light-emited diode (LED = Ultrablue Is, D.M.C.) and Plasm arc curing (PAC = Apollo 95 E, DMD). Resin-based cement (Enforce, Dentsply) was used to form disk shaped specimens. Irradiance (I) of each LPU, irradiation time (T) was

adjusted to form three different dosages; 24J.cm^{-2}(I/2x2T), 24J.cm^{-2}(IxT) and 48J.cm^{-2}(Ix2T). Energy dosages were tested through 2.0 mm ceramic (Duceram Plus, Ducera). Three groups undergo direct irradiation (DPa), according to LPU, over resin cement and a group without light exposure was used as a negative control. Thirteen groups were tested (n=10). Knoop Hardness numbers were taken from 100 μm depth at cross sectional areas of specimens stored for 24 hours at 37°C. ANOVA Two-way and Holm-Sidak method test was used for comparisons of activation mode and dosages. All groups were compared with control using ANOVA one-way and Holm-Sidak test to identify differences among groups with significance level $p<0.05$. Dosages through ceramic with 48J.cm^{-2} with LED and HAL showed statistically higher microhardness than control ($p<0.05$). Four simulations with PAC were similar, only DPa was higher than control ($p<0.05$). In conclusion, LED shows similar performance with HAL in dosage through ceramic simulations but microhardness increase was dependent on amount or energy dosage only for Hal source.

Keywords: resin cements, hardness, radiation effects, curing devices, dental ceramic.

INTRODUCTION

Resin-based luting materials have been widely used for adhesive luting of ceramic restorations (Kramer & others, 2000). According to the activation mode,

resin-based cements are divided into three groups; chemically activated (self-cured), photo-activated and photo/chemically activated resin cements (dual-cured). As a result of effort to assembly advantages of chemically activated and photo-activeted resin cements, dual-cured cements were idealized. Dual-cured cements have in their composition both a photoinitiator (camphoroquinone) and chemical activation components (peroxide/amine). Thus, activation of resin cement is initiated both chemically and by visible light using a wavelength of 400-500 nm (Jung & others, 2001).

There are several factors that can influence the degree of polymerization of a resin cement, for instance, the porcelain shade (Cardash & others, 1993) and thickness, (Blackman & others, 1990; Strang & others, 1987b) the light unit, and the curing time. (Linden & others, 1991). In these conditions, dual-activated resin luting agents usually is chosen due to best combination of mechanical and physical properties when photo-activation efficiency is doubtful.

Photo polymerization of dual-cure resin-based materials was necessary to maximize strength and rigidity (Attar & others, 2003). Also, for dual-cure cements, the maximum bond strength is achieved only when photoactivation is properly made (Hasegawa & others, 1991; Peters & Meiers, 1996).

The understanding of methacrylate-based polymerization of dental restorative materials is becoming increasingly important due to recent introduction of new curing sources (e.g. plasma arc lamps, LEDs and lasers) as well as curing techniques (Halvorson & others, 2002). Argon lasers, high-intensity halogen lights, and xenon plasma arc lamps have all been shown to achieve rapid polymerization

(Tarle & others, 1998; Meniga & others, 1997; Knezevic & others, 2002). Traditionally resin-based cements are irradiated with tungsten halogen curing light. This device is the most used and studied for composite cure (Burgess & others, 2002; Ozyesil & others, 2004; Rueggeberg & others, 1994) and for use in indirect photo-activation (Blackman & others, 1990; Ozyesil & others, 2004).

Comparing to conventional tungsten HAL lamps blue LEDs have some advantages like the efficiency of conversion of electrical energy to useable curing energy, the amount of energy in a useful narrow of wavelength (spectral irradiance) compared with wider narrow of wavelength of HALs, the durability and not requirement for wavelength and heat filters (Tsai & others, 2004). Resin-based composites have been shown satisfactory mechanical properties when irratiated with LEDs light photo-activation units (Jandt & others, 2000; Tsai & others, 2004).

PAC method produces light energy by two electrodes that are placed very close to each other, emitting light when a high voltage is applied rather than by heating a tungsten filament as a halogen lamp (Rueggeberg, 1999). Compared to conventional halogen curing lights, PAC units also operate at a similar wavelength range between 430 and 500 nm. Several products on the market provide a light intensity between 1200 and 1800 mW/cm^2.

Then, differences among output irradiance of these devices may be adjusted for energy dosage by irradiation time adjusting, provided an optimization of irradiation time without affect the degree of cure of composites. This concept is centered on the theory that the cure of photoinitiated resin composites is based on

the total energy delivered to it (Rueggeberg, 1999, Halvorson & others, 2002; Halvorson & others, 2003).

Since differences exist between photo-activation methods including energy density, spectral distribution of the light emitted by the curing unit and the polymerization process itself, became important the understanding of photo-activation process of dual-cure resin cements. There is a relative lack of information about the best method for dual cements photoactivation through ceramics (*i.e.* device and irradiation time), taking in account the new curing light devices like LED and PAC light.

The objective of this study is to test the following null hypothesis. 1. The different devices (HAL, LED and PAC) have similar effect upon microhardness of dual-cure resin cement when equivalent energy dosage is applied. 2- Same reduction of light intensity compensated by time would not significantly reduce Knoop hardness.

MATERIALS AND METHODS

Feldspatic ceramic material, Duceram Plus (Ducera, Dental GmbH&Co.KG, Germany), VITA shade dentin A3 (Vita Zahnfabrik H, Rauter GmbH & Co. KG, Bad Säckingen, Germany) was condensed in a metalic mold to form a cylindrical specimen that was fired in ceramic furnance (Austromat M, Dekema Austromat-Keramiköfen, Freilassing, Germany) processed according to manufacture's

instructions. One specimen, discs shaped ceramic with 2.0 (±0.01) mm thickness and 8.0 (±0.01) mm diameter were 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 manufacturer's instructions in a room with temperature (23°C ± 1), and relative humidity (higher than 30%) controlled, according to ISO 4049, 2000 (The International Organization for Standardization). Volume proportion between base and catalyst was standardized by an acrylic mould with equivalent containers. The resin cement was mixed and inserted in nylon mould with 5.0 (± 0.1) mm diameter in centre hole and 1.0 (± 0.1) mm deep. The nylon mould was previously 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 (Uctasli & others, 1994). The hole in the mould was covered by polyester film (± 25 μm thickness).

The resin cement was activated by two modes, only chemically activated (self-cured mode) or photo/chemical activated (dual-cured mode). One group (n=10) was mixed in a dark room with a red light filter (for x-ray film revelation) to avoid photoinitiator sensitization by useful wavelength. This group was used as a control. When photo/chemical activation was used, the resin cement was light irradiated by two modes; by direct photo-activation (DPa) or irradiated through ceramic (PtC). In this last model, the ceramic disc was interposed between the tip

of light cure unit and the polyester film, above de resin cement and irradiation was performed according to three energy dosages situations.

Three light activation units were used. 1 - (HAL), Tungsten halogen curing light (XL 2500, 3M ESPE) with mean irradiance of 589 mW/cm^2; 2 – (LED) Ligh-emitting diode (Ultrablue Is, D.M.C. Equipamentos LTDA, São Carlos, SP, Brasil) with mean irradiance of 614 mW/cm^2;. and 3 - (PAC) xenon plasma arc device (Appolo 95SE, DMD – Medical Diagnostic Systems, Westlake Village, CA, USA) with mean irradiance of 1656 mW/cm^2;. The devices were connected to voltage stabilizer and supported by an apparatus to allow tip light guide placement at direction of 90° degree.

A hand-held radiometer (Hilux, Dental Curing Ligth Meter, Benbionglu Dental Inc.) was used to check irradiance of three devices. Means were taken after 10 activation sequences for each device. Times were adjusted, according to irradiance of each light photo-activation unit (LPU) to accomplish the energy dosages desired, according to formula.

$$D = \frac{P}{A} \times T = \frac{mW}{cm^2} \times s = \frac{mJ}{cm^2}$$

“P” is the potency of LPU, (mW), “A” area of output guide of LPU (cm^2). “T” irradiation time used (s). For P/A was used de irradiance data given for hand held radiometer (mW/cm^2). “D”= Energy dosage was obtained in mJ/cm^2. For convenience J.cm-^{2} was used, (1J=1000mJ).

Three energy dosages were used;

1 - 24J.cm^{-2}(IxT), 24 Joules with maximum value for irradiance (I) in mW/cm^2 achieved for each photo activation unit for time (T=irradiation time); Establishment of this dosage was based in a condition of 600 mW/cm^2 of irradiance with a halogen LPU, during 40 seconds

2 – 24J.cm^{-2}(I/2x2T) half irradiance potential and double time for each device. For decrease irradiance to approximately half, the tips of light guide of LPUs were keept at distance of 7.7 mm, 4.8 mm and 4.75 mm for HAL, LED and PAC respectively.

3 – 48J.cm^{-2}(Ix2T) full irradiance potential for double time. These dosages were applied through ceramic. One group for each photo-activation device was made with direct activation, without ceramic interposed, for test microhardness in optimum photo-activation conditions. The time used in this group was according to manufacturer (HAL = 40s, LED = 40s and PAC = 3s). One group was made with light absence environment for obtain the chemical-cure microhardness profile. Combination of LPUs and test conditions resulted in thirteen groups (n=10) were tested in this study (Table 2).

Specimens were stored in culture stove in dry and dark conditions at 37°C for approximately 24 hours. After storage specimens were fixed with in a pre-molded acrylic mould, sticky wax was previously used to improve fixation and fill eventual space of lack of adaptation. Resin cement disks were sectioned longitudinally and the surface was finished and polished in waterproof 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 fifty grams-force for fifteen seconds. Measurements were manually made within 40X magnification across the section of resin cement specimens. Knoop Hardness number was provided automatically by tester's software, based in indentation measurement performed for operator. The testing was conducted by only 1 person to maximize standardization.

Knoop Hardness numbers at 100 μm depth from irradiated surface were obtained Three indentations were made, with 1 mm distance between them and arithmetic means were calculated for each specimen.

Data were analyzed in statistical software (SigmaStat 3.0 for windows, SPSS Inc.). Overall changes in knoop hardness, for hardness at 100 μm caused by LPUs and activation method were evaluated by Two-way Analysis of Variance (ANOVA) and by Holm-Sidak method (All pairwise multiple comparison procedure) as a post-hoc test. One-way ANOVA and Holm-Sidak test was performed for comparison of all groups versus chemical activation (Ca) or "unphotopolymerized" group (control). All test were performed at significance level of $p<0.05$.

RESULTS

Microhardness means at 100μm deep, showed a statistically significant interaction between LPUs and activation method ($p=0,02$). When direct photo activation was performed HAL showed statistically higher microhardness mean

than LED and PAC ($p<0.05$). No differences were found between LED and PAC. (Table 2).

For 48J.cm^{-2}(Ix2T) and 24J.cm^{-2}(I/2x2T) energy dosage simulations, LED showed statistically higher ($p<0.05$) Knoop Hardness mean than PAC and HAL showed intermediate values. Knoop hardness obtained with LED were similar with HAL for all situations of energy dosage used (Ptc mode). Devices were similar when 24J.cm^{-2}(IxT) dosage was used.

For each device, the effect of energy dosage through ceramic (Ptc) with 48J.cm^{-2}(Ix2T) was similar with DPa, exception for LED. When LED device was used with 48J.cm^{-2}(Ix2T) dosage, the effect upon Knoop hardness of dual-cure resin cement used, after 24 hours storage, was statistically higher than DPa and 24J.cm^{-2}(IxT) using this device ($p<0.05$), buy similar with 24J.cm^{-2}(I/2x2T) . HAL device setting for 48J.cm^{-2} showed statistically superior microhardness than others mode of indirect irradiation ($p<0.05$). It was not found differences among activations mode related with PAC.

When groups were compared with chemically-activated mode (Control) Dpa with Hal and PAC showed statistically superior microhardness ($p<0.05$). PAC showed similar hardness than control for this energetic dosage. In regard to others dosages only LED adjusted for 24J.cm^{-2}(I/2x2T) showed statistically superior microhardness ($p<0.05$) than control (Figure1).

DISCUSSION

The first null hypothesis was rejected because hardness was significantly affected by LPU used, according to specific activation method. The results showed an interaction statistically significant between LPU and activation method. It means that efficiency of irradiation mode, mainly the energy dosage applied, is dependent of LPU used and consequently depends on features of light energy produced. Although irradiation time was adjusted in function of irradiance of each energy dosage simulation, probably other factors rather than the amount of energy delivered affect the microhardness means of resin cement tested. The different features of output wavelengths, spectral irradiance, light power of device, as well as differences in the efficiency of the optical delivery system, including the light guiding tips can be responsible for effect on microhardness level (Knezevic & others, 2001; Dannheimer & others, 1996; Strang & others, 1987a). Sufficient intensity at the correct wavelength and adequate exposure time are critical variables for satisfactory polymerization.

When Halogen and PAC where tested in direct photoactivation mode they showed similar effects between direct photo activation and 48J.cm^{-2} upon microhardness. But when devices were compared Halogen performed better than PAC directly used. In addition HAL 48J.cm^{-2} dosage was statistically superior to control and others dosages being similar with DPa. Thus, when irradiation is performed with HAL source, the highest Knoop hardness with photo-activation through ceramic was found when the higher energy dosage was applied. Based in

these evidence second null hypothesis can be rejected. It means that microhardness was dependent of energy dosage used when HAL device was employed. Thus energy dosage concept can be used when similar conditions are present. Generally energy dosage concept is well correlated with cure profile of resin composites (Halvorson & others, 2002).

In present study, a 2.0 mm of feldspatic disc shaped ceramic was used. This condition is a challenge for proper initiators photo sensitization of resin-based cements, due to light by transmittance attenuation and light dissipation in ceramic structure. Then, large irradiation times should be evaluated for energy dosages technique. Commonly, larges exposures time are advocated, since increasing curing time (to a certain limit) gives better physical properties in photoactivated composites (Tanoue & others, 1998), specially when photo-activation through thicker ceramic layers should be performed (Breeding & others, 1991; Warren, 1990). Additionally with prorogated photo-cure time, change direction of photo-cure device along irradiation time seems to be beneficial since this technique showed better microhardness values and satisfactory bond strength for resin-based cement, beneath thicker layers (*i.e.* 3mm thickness) of ceramic (Foxton & others, 2003).

The commercially available LED curing light used in this study provided an adequate microhardness with power dosages of $48J.cm^{-2}$(Ix2T) and $24J.cm^{-2}$(I/2x2T), that was superior and similar respectively with positive control (irradiation directly without ceramic interposed) and higher than negative control (only chemical activation). Similarity between microhardness means, although energetic

dosage of 48J.cm^{-2}(Ix2T) is twice higher than 24J.cm^{-2}(I/2x2T), can be due elapsed exposure time, more almost three times more than irradiation time for PAC in same conditions. This can be particularly important in some clinical conditions, like irradiation with certain distance of curing tip of proximal areas of inlays.

It was found that incrasing exposure time may overcome the effect of less specific wavelength (i.e. 410-490 nm) for composite resin (Nomoto, 1997). If some change of useful wavelength happened through ceramic then it could be compensate by higher exposures times used in for halogen and LED devices.

Based in present finding LED 48J.cm^{-2}(Ix2T) showed significantly higher microhardness values than direct photo-activation. The former was applied with twice more time than last, but trough 2.0 mm ceramic. As LED was similar to HAL for simulated situation dosage through ceramic and DPa comparisons were different, perhaps there are dissimilarities in photo-activation process of resin-based cements between these two LPUs. Recently Tsai & others, 2004 found that LED lights gave greater curing depths with A3 shade, while HALs lights gave greater curing depths with C4 shade. Since ceramic used was shade A3 perhaps this fact may contributed. Certainly, one of the most important factors to consider is final irradiation time that was double of direct photo activation.

Efficacy of photo-activation depends on transmition of curing light through ceramic and camphoroquinone photosensitization. Camphorquinone has peak absorption of wavelengths around 470 nm (Pradhan & others, 2002; Nomoto, 1997). The emission of wavelength narrow in this specific excitation peak is related with scraping depth of composite (Pradhan & others, 2002). According to other

studies LEDs have a spectral radiance near camphorquinone exitantion peak. But as showed for Pradhan & others, 2002) the degree of cure with less specific narrow band for LPU can be compensated for higher exposure times. Probably for these reason, irradiation for approximately 80 s with LED overcome hardness but more studies are need for a better comprehension of photoinitiation process of resin cements when indirectly irradiated. Additionally, manufactures should provide a graph indicating the minimum acceptable exposure for each product for specified curing lamps (Musanje & Darvell, 2003) since photoinitiators used have irradiance features requirements (Hofmann & others, 2000). When directly photoactivated Halogen light output can fulfill better the requirements of photoinitiatior of resin cement used. Manufacturers should make adjustment in composite photoinitiators for new light sources devices for photo activation. Other initiators would require sources that are spectrally matched with their individual absorption band.

PAC showed similar means for all energy dosages used. Due to none situation did not differs from control, it is difficult to predict if degree of cure was lower or if chemical cure itself overcome hardness level provide for photo-activation process. Only direct photo-activation group with PAC was statistically superior to control.

Although power dosage for PAC was adjusted to be similar to HAL and LED, other factors may be responsible for lower hardness means. The delay between light emission (3s), features of light provided, like spectrum, energy absorbance and attenuation feature of ceramic may affect negatively cure kinetics. Also,

smaller performance of PAC device through ceramic substrate can be attributed to spectral changes during light transmittance through ceramic.

Only direct photo-activation with 3s irradiation show hardness mean at 100μm depth higher than control. Despite the five situations that differs of chemical activation mode, dosages with extended times for HAL and LED showed advantages in relation with others (except for LED 24J.cm^{-2} (I/2x2T). In those situations when means were similar with control, the results should be carefully interpreted based on microhardness methodology conditions. It was reported that post-cure storage conditions has influence on composite microhardness (Quance & others, 2001). Additionally, post-irradiation hardness increase is rapid over the first hours, and slows to reach a maximum value within 24 hours. (Pilo & Cardash, 1992).

According to microhardness values for resin cement used can be stated that a satisfactory degree of cure was obtained for chemical-activated mode (self-cured). This is emphasized by the fact that several groups do not differ with chemical mode (control). Several resin-based cements brands show a significant decrease in hardness (el-Mowafy & others, 1999; Hasegawa & others, 1991; Peutzfeldt, 1995) and flexural strength modulus (Blackman & others, 1990; Warren, 1990) when material is only chemically-activated. Reduced elastic modulus of resin cements can be responsible for failure rates of inlays (Anusavice, 1997). The good degree of cure of enforce, also found in other studies (Braga & others, 2002a), let this composite less dependent of photo-activation efficiency

than some other resin cement commercially available. Additionally, the post-cure temperature storage has an important effect on Knoop hardness values of composites (Quance & others, 2001).

For these reasons, dual-cure cements performed better when well light irradiated than when they are cured only by self-cure mode (Cardash & others, 1993; Hofmann & others, 2001; Foxton & others, 2003). Also, incomplete polimerizaton of resin-based luting cements cause decreased in mechanical properties, increase water sorption (Uctasli & others, 1994). Moreover, the presence of unreacted molecules in poorly polymerized resin may be leached from material, causing tissue irritation (DeWald & Ferracane, 1987). In the other hand, polymerization stress within the luting cement could not be completely compensated for by larger luting spaces (Hahn & others, 2001 and dual cure mode can show more polymerization contraction stress of resin cements than self-cure mode, which may represent higher risk of marginal leakage (Braga & others, 2002b).

CONCLUSIONS

Within limitations of this study it is possible concludes that.

1- Halogen and Led light unit had similar performance, according to means of microhardness of resin cement when irradiance through ceramic is used.

2- Chemical activation only mode was similar to photo/chemical activation mode of several simulations studied, representing a good chemical reaction profile of resin cement investigated.

3- Only HAL source showed relationship between increase of microhardness and energy dosage increase.

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Table 1: Design of study

LPU (Light photo-activation unit)	**Without ceramic**		**Photo-activation (dual-cured mode) through ceramic structure with 2,0 mm thick (PtC)**		
	Chemical activation only (Qa)	**Direct Photo-activation (DPa)**	**24 J.cm^{-2}(I/2x2T)**	**24 J.cm^{-2}(IxT)**	**48 J.cm^{-2}(Ix2T)**
HAL (Tungsten halogen curing light)	——	*I*– 589 mW/cm^2 *T* – 40 s *S* – 1 *D* – 0 mm	*I*– 300 mW/cm^2 *T* – 82 s *S* – 1 *D* – 7,7 mm + ceramic	*I* – 589 mW/cm^2 *T* – 40 s *S* – 1 *D* – contact with ceramic	*I* – 589 mW/cm^2 *T* – 82 s *S* – 1 *D* – contact with ceramic
LED (Light-emitting diode)	——	*I* – 613,8 mW/cm^2 *T* – 40 s S– 1 *D* – 0 mm	*I*– 300 mW/cm^2 *T* – 78 s *S* – 1 *D* – 4,8 mm + ceramic	*I*– 613,8 mW/cm^2 *T* – 40 s *S*– 1 *D* – contact with ceramic	*I* – 613,8 mW/cm^2 *T* – 78 s *S* – 2 *D* – contact with ceramic
PAC (Xenon Plasm Arc light)	——	*I* – 1653 mW/cm^2 *T* – 3 s *S* – 1 *D* –0 mm	*I* – 825 mW/cm^2 *T* – 29 s *S** – 9 + 1 (2s) *D* – 4,75 + ceramic	*I*– 1653 mW/cm^2 *T* – 15 s *S** – 5 *D* – contact with ceramic	*I* – 1653 mW/cm^2 *T* – 29 s *S** – 9 + 1 (2s) *D* – contact with ceramic

"I": Irradiance or energy density of LPU, used for specific energy dosage. "I/2"; around half full potencial irradiance of LPU "T": Irradiation time. "S": Sequences of turn-on need to achieve irradiation time. (S*) For PAC, there was a 3 s delay among "turn-on". "D": Distances between tip of light guide and resin cement surface.

Table 2: Means of microhardness for 100 µm depth according with groups and respective light irradiation mode LPUs used.

Light photo-activation unit (LPU)	Light irradiation mode							
	DPa		Energy dosages through 2.0 mm ceramic (PtC)					
			48J.cm^{-2} (Ix2T)		24J.cm^{-2} (IxT)		24J .cm^{-2}(I/2x2T)	
Halogen	*51,54 (2,55)	**a, A**	*50,88 (3,72)	**a, A**	45,52 (1,08)	**a, B**	46,99 (2,26)	**ab, B**
LED	47,23 (4,38)	**b, B**	*50,48 (2,80)	**a, A**	47,17 (4,16)	**a, B**	*48,74 (3,51)	**a, AB**
PAC	*47,69 (2,09)	**b, A**	46,22 (2,91)	**b, A**	46,61 (2,31)	**a, A**	45,51 (2,19)	**b, A**

Different capital letters in rows represents statistically significant differences ($p<0.05$) between light irradiation modes for same LPU. Different minuscule letters in columns represent differences statistically significant ($p<0.05$) between LPU for each light activation modes. Values in parenthesis show standard deviation (SD) for each mean. (*) means are statistically higher than control (mean 44.7 and SD 3.79)

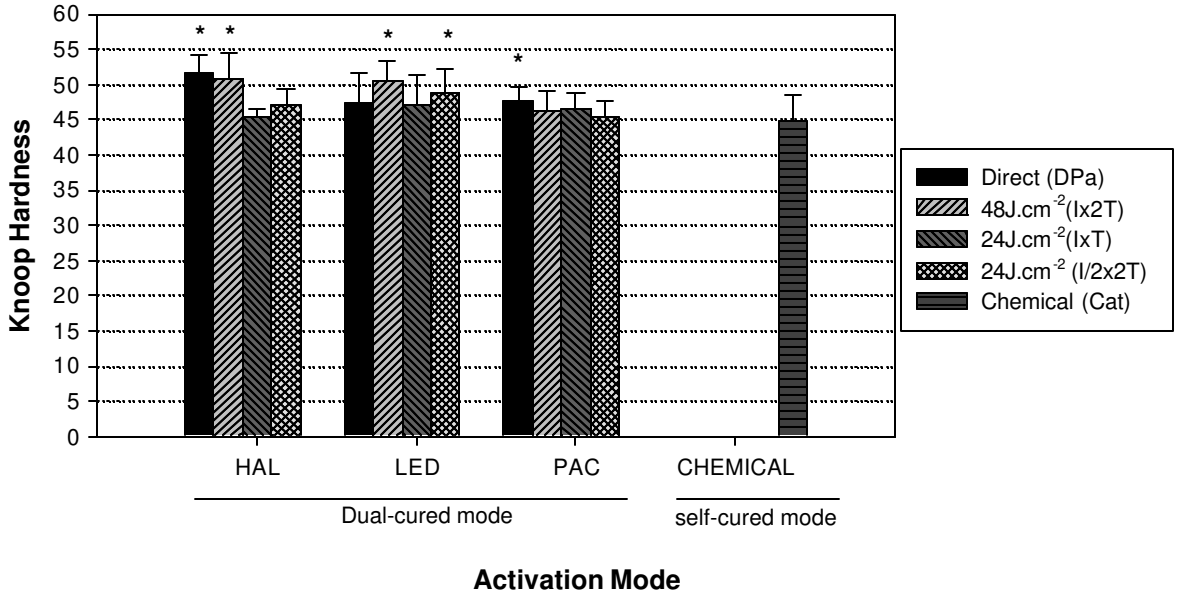

Figure 1: Hardness at 100 µm depth for each photo activation unit (n=10) according with simulations conditions. Asterisk represents differences statistically significant of group compared with chemical activation mode (control) ($p<0.05$).

CONCLUSÕES GERAIS

Com as limitações dos estudos é possível concluir que;

1- A estrutura cerâmica reduz a eficiência de fotoativação de maneira proporcional à espessura do material.
2- A armazenagem e o tempo pós-ativação do cimento resinoso de dupla ativação mostraram efeito sobre o aumento da microdureza principalmente nas camadas mais profundas do cimento, quando comparado a microdureza imediata do material.
3- Uma redução progressiva de microdureza pode ser estimada em função do aumento da profundidade de leitura sob camadas mais espessas de cerâmica, quando a microdureza é testada imediatamente após a fotoativação.
4- Os aparelhos para fotoativação com lâmpada halógena e com LED obtiveram desempenho similar quanto a microdureza de cimento resinoso.
5- As dosagens devem ser adequadas para cada aparelho, para se obter uma eficiente microdureza de cimento resinoso fotoativado indiretamente.

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* De acordo com a norma da UNICAMP/FOP, baseada no modelo Vancouver. Abreviatura dos periódicos em conformidade com o Medline.

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Artigo: "*Influence of ceramic thickness on mediate and 24h post-irradiation microhardness of a dual-cured resin-based cement".*

Influência da espessura da cerâmica sobre microdureza mediata e 24h pós-irradiação em um cimento resinoso de dupla ativação

1A. Materiais e métodos

1A.1. Confecção dos espécimes de cerâmica

O material selecionado para o estudo foi a cerâmica odontológica Duceram Plus, (Ducera, Dental GmbH&Co.KG, Alemanha), cor dentina A3 da escala Vita (Vita Zahnfabrik H, Rauter GmbH & Co. KG, Bad Säckingen, Alemanha), classificada como cerâmica feldspática (Tabela 1A.1). O material foi manipulado e, em seguida, condensado em um molde metálico, para que após a sinterização, um cilindro de cerâmica com 8,0 (±0,01) mm de diâmetro fosse formado. O material então foi levado para sinterização em um forno para cerâmica (Austromat M, Dekema Austromat-Keramiköfen, Freilassing, Alemanha). Todas as etapas foram executadas com base nas instruções dos fabricantes dos materiais e equipamentos que estavam sendo utilizados.

O cilindro, obtido do processo de sinterização, foi seccionado com disco diamantado sob refrigeração em água, montado em cortadeira, regulada para corte em baixa rotação. Estruturas de cerâmica com formato de disco eram formadas e submetidas ao acabamento com lixa d'água de granulação decrescente (180, 320, 400, 600 e 1200) numa politriz metalográfica. Após o acabamento, os discos foram levados ao forno para o processo de glazeamento.

Foram obtidos 3 discos de cerâmica com 8,0 mm de diâmetro e três espessuras diferentes; 0,7 (±0,01) mm, 1,4 (±0,01) mm e 2,0 (±0,01) mm (Figura 1A.1). Todas as medidas foram aferidas com paquímetro digital com precisão de 0,01 mm (Starrett, Elmhurst, IL, EUA).

1A.2. Obtenção dos corpos-de-prova de cimento resinoso

Uma matriz de nylon foi previamente coberto com *spray* de tinta, cor preta (Colorgin Spray, Sherwin-Wiliams do Brasil Ind Com Ltda, São Bernardo do Campo, SP). O objetivo desse procedimento foi deixar a matriz opaca, de modo que a transmissão de luz ficasse restrita apenas ao disco cerâmico e ao cimento resinoso (Uctasli *et al.*, 1994). Essa matriz possuía dimensões padronizadas, que possibilitavam a obtenção de espécimes de cimento resinoso com 5,0 (±0,01) mm de diâmetro e 1,0 (±0,01) mm de profundidade. (Figura 1A.1)

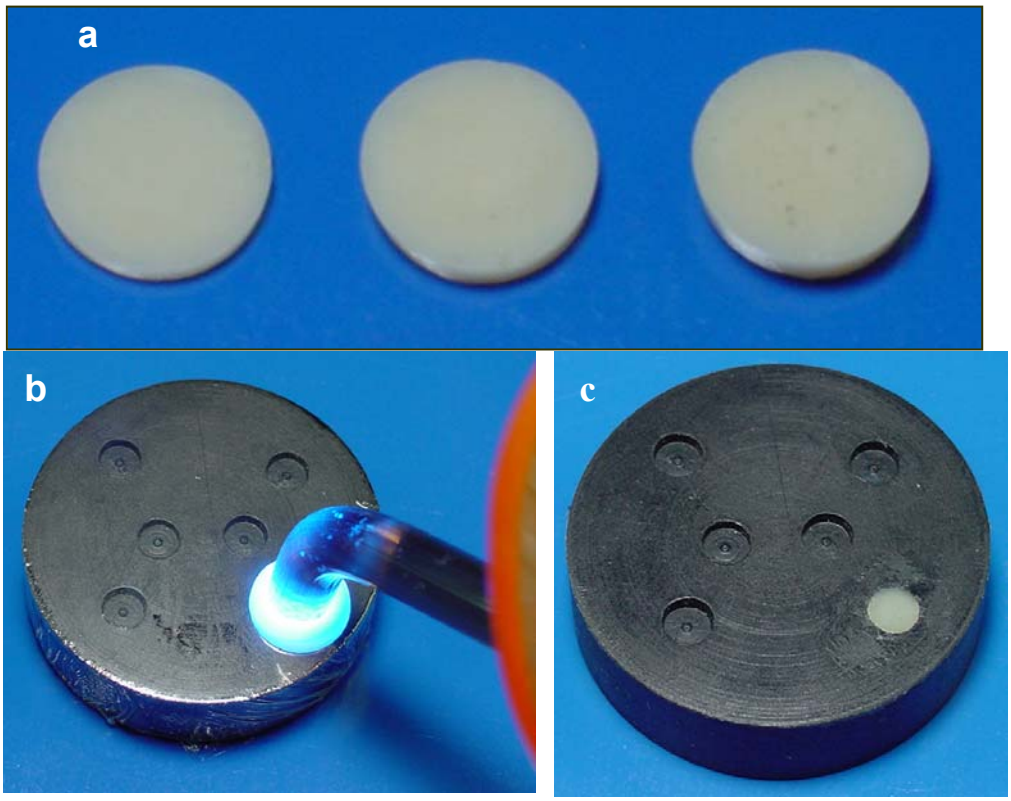

Figura 1A.1. "*a*" Discos de cerâmica com espessuras diferentes. "*b*" cimento resinoso sendo fotoativado através de estrutura cerâmica. "*c*" espécime em forma de disco, obtido a partir de polimerização do cimento resinoso.

O cimento resinoso RelyX ARC (3M ESPE, St. Paul, MN, USA), cor A3, foi utilizado no presente estudo (Tabela 1A.1). As pastas base e catalizadora do cimento resinoso foram automaticamente dosadas, pela bisnaga dispensadora do sistema, em proporções com volumes equivalentes. A mistura manual das pastas foi efetuada por 10s, com auxílio de espátula n° 24 em bloco de papel descartável, de acordo com as instruções do fabricante. Após manipulado, o cimento foi inserido na matriz até o completo preenchimento do espaço reservado para o cimento. Para evitar o contato do cimento com a cerâmica, um filme de poliéster

(±25 μm de espessura) foi colocado sobre o cimento resinoso e a matriz. Todos os procedimentos, desde a manipulação do cimento até a obtenção dos espécimes, foram realizados em ambiente de temperatura (23°C ±1°C) e umidade controladas (acima de 30% de umidade relativa) conforme especificação da ISO 4049 (*The International Organization for Standardization*).

Uma marcação foi realizada na matriz para permitir o alinhamento entre a ponta da sonda condutora de luz do aparelho e o orifício da matriz, garantindo a irradiação de toda a extensão do cimento resinoso. Durante o planejamento da pesquisa, tomou-se o cuidado de certificar-se que o diâmetro do orifício da matriz que era preenchido por cimento resinoso, fosse menor que o diâmetro de saída de luz, na ponta da sonda do aparelho foto ativador. Essa medida visou garantir uma completa exposição do cimento resinoso ao longo de sua extensão, na abertura da matriz. Foi utilizado o aparelho XL 2500 (3M ESPE, St. Paul, MN, USA) para fotoativação de compósitos, com emissão de luz a partir de lâmpada halógena com filamento de tungstênio. A irradiância (intensidade) era de ≤650 mW/cm^2, e foi constantemente monitorada por radiômetro portátil Model 100 Curing Radiometer (Demetron Research Corporation, Danburry, CT, USA). O aparelho para fotoativação era ligado a corrente elétrica por meio de um estabilizador de voltagem, com o intuito de minimizar possíveis oscilações na corrente elétrica, que pudessem afetar significativamente a irradiância final do dispositivo. O aparelho foi também suspenso em um suporte que permitia deixar a sonda do aparelho perpendicular com a superfície da cerâmica e conseqüentemente com o cimento resinoso.

Basicamente, as variáveis investigadas neste estudo foram; 1- Modo de ativação; Apenas ativação Química (modo *self-cure*) ou modo de ativação foto/quimico *(*dupla ativação *ou dual-cure)* nas situações: fotoativação direta ou através de cerâmica (0,7 mm, 1,4 mm e 2,0 mm); 2- tempo, decorrido desde o início da manipulação até a leitura de microdureza (mediato, ou 24 horas após a radiação) 3 – Profundidade de leitura (100µm, 300µm, 500µm e 700µm) a partir da superfície do cimento voltada para o meio externo. Dez grupos experimentais (n=10) foram testados a partir das variáveis em estudo. O delineamento do estudo está representado na Tabela 1A.2.

Grupo Fd - Após a aplicação do cimento na matriz, um filme de poliéster foi colocado entre o cimento e a ponta do aparelho para fotoativação. A sonda do aparelho deveria ficar alinhada com o centro do orifício da matriz, preenchida com cimento resinoso e com angulação de 90° em relação ao orifício da matriz. O aparelho foi acionado durante 40 segundos (com irradiância de ≤650 mW/cm^2). Após a irradiação os espécimes foram retirados da matriz e armazenados individualmente em frasco opaco, em estufa à temperatura de 37°C (±1°C) durante 24 horas. Desse modo, 10 espécimes foram armazenados por 24h previamente ao teste de microdureza, formando o subgrupo *Fd–24h* (Fotoativação direta com 24 horas de armazenagem pós-irradiação). Outro subgrupo (n=10) foi submetido aos procedimentos de preparo dos espécimes para leitura mediata de microdureza, formando o subgrupo *Fd–med* (Fotoativação direta com leitura mediata). A leitura mediata era realizada entre o intervalo de 10 a 20 minutos desde o início da manipulação.

Grupo Fac0,7- (Fotoativação através de cerâmica com 0,7 mm). Neste grupo os mesmos procedimentos feitos no grupo anterior foram repetidos, entretanto, um disco de cerâmica com espessura de 0,7 (±0,01) mm foi interposto entre a ponta do aparelho e o filme de poliéster sobre o cimento resinoso. Assim, neste grupo a fotoativação era realizada através da estrutura cerâmica. Novamente 2 subgrupos foram criados, de acordo com o tempo pós-irradiação decorrido até o início da leitura de microdureza; *Fac0,7-med* e *Fac0,7-24h* (mediata e 24 h respectivamente).

Grupo Fac1,4 – Semelhante ao grupo Fac0,7, porém um disco de cerâmica de 1,4 (±0,01) mm foi interposto entre a ponta da sonda do aparelho e o filme de poliéster sobre o cimento resinoso. Subgrupos formados; Fac1,4-med e Fac1,4-24h.

Grupo Fac2,0 – Nesse grupo foi utilizado disco de 2,0 (±0,01) mm. Subgrupos formados; Fac2,0-med e Fac2,0-24h.

Grupo AQ – *(Somente* Ativação química). Quando o a fotoativação não era realizada, ou seja, o cimento era utilizado no modo somente ativação química, a iluminação ambiente convencional era desligada. Uma lâmpada com filtro vermellho para revelação radiográfica era acionada, visando-se evitar a exposição do compósito a luz com comprimento de onda necessário para a ativação do cimento resinoso. Desse modo, minimizava-se a fotoativação do compósito.

1A.3. Leitura da Microdureza

Na etapa que compreendia a preparação dos corpos-de-prova para leitura de microdureza, os mesmos 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. Testes preliminares não indicaram a interferência desse procedimento nos valores de microdureza coletados.

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 era utilizada por 4 vezes para fins de padronização de tamanho de disco.

O teste de dureza Knoop foi realizado em um aparelho para ensaios universal de microdureza, microdurômetro HMV-2 (Shimadzu, Tokyo, Japan) com procedimento automático de aplicação de 50g de carga durante 15 segundos. As leituras de microdureza foram realizadas mediante aumento de 40x. Após a calibração manual o aparelho convertia automaticamente a distância entre as extremidades da diagonal maior do losango (formato resultante após a edentação no compósito que era mensurado manualmente pelo operador), em valores de dureza Knoop, com base na fórmula a seguir.

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

Fórmula para cálculo da microdureza, onde “DK”, representa a dureza Knoop, “*P”* é a carga aplicada em gf (gramas-força) e “*d”* é o comprimento da diagonal maior do losango em µm.

Para cada corpo-de-prova 4 médias de dureza Knoop foram obtidas, de acordo com a profundidade de leitura. As médias correspondiam a microdureza nas profundidades de 100 µm, 300 µm, 500 µm e 700 µm da superfície externa do disco. As médias aritméticas para cada profundidade resultavam de três leituras em cada profundidade, 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 1A.2). 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 10 a 20 minutos) fosse extrapolado.

As edentações foram realizadas de acordo com uma seqüência pré-estabelecida (Figura 1A.2). 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 edentações era realizada a partir da superfície inferior.

Portanto, 4 médias de dureza para cada corpo-de-prova foram criadas, como resultado de doze leituras.

O Teste estatístico de 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) 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. O nível de significância utilizado em todos os testes foi de 5% (α=0,05).

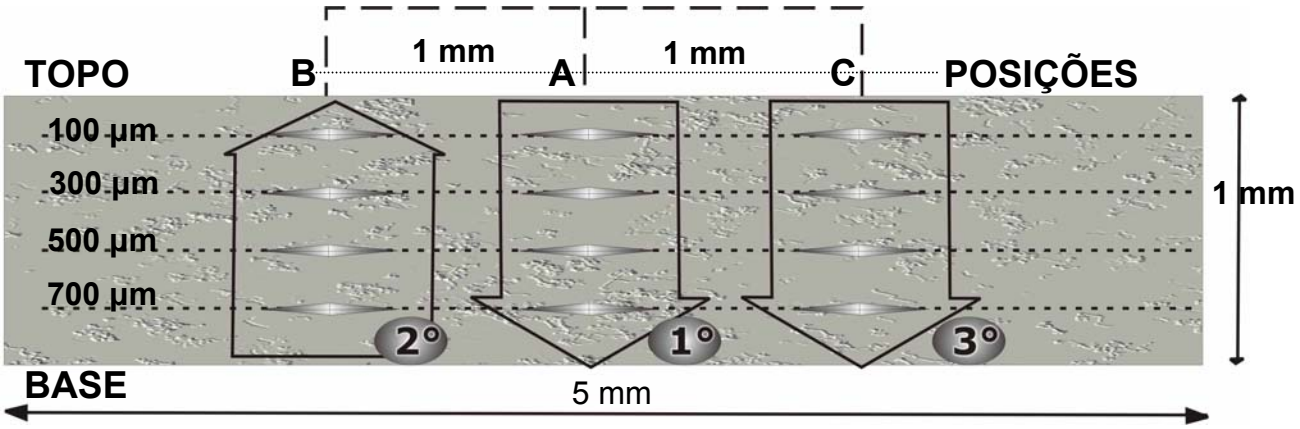

Figura 1A.2. Representação esquemática da seqüência utilizada na leitura de microdureza. Na terceira seqüência, metade dos corpos-de-prova, para cada grupo, eram analisados no sentido contrário ao exposto na figura.

Tabela 1A.1. Descrição dos materiais utilizados

Material	Fabricante	Composição	Lote
Cerâmica			
Duceram Plus (cor dentina A3)	Duceram Plus, (Ducera, Dental GmbH&Co.KG, Alemanha)	K_2O_3, Al_2O_3, $6SiO_2$, SnO, ZrO, Na_2O, CaO e pigmentos	2459
Cimento resinoso			
RelyX ARC (cor A3)	3M ESPE Dental Products, St. Paul, MN, USA.	**Monômeros** - Bis-GMA, TEGDMA **Tamanho de partícula –** 1,5 µm **% de partículas em peso** – 67,5	CYEA

Bis-GMA: bisfenol A diglicidil éter dimetacrilato; TEGDMA: Trietileno glicol dimetacrilato;

Tabela 1A.2. Delineamento do estudo e formação dos grupos, de acordo com as variáveis em estudo

		GRUPOS (de acordo com o método de ativação e respectivas representações)									
		Fotoativação direta sobre o cimento *(Fd)*		Através de 0,7 mm de cerâmica *(Fac0,7)*		Através de 1,4 mm de cerâmica *(Fac1,4)*		Através de 2,0 mm de cerâmica *(Fac2,0)*		Apenas ativação química *(AQ)*	
Intervalo pós-ativação previamente a leitura(*)		10-20 min (Fd–med)	24 h (Fd–24h)	10-20 min (Fac0,7–med)	24 h (Fac0,7–24h)	10-20 min (Fac1,4–med)	24 h (Fac1,4–24h)	10-20 min (Fac2,0–med)	24 h (Fac2,0–24h)	20-30 min (AQ–med)	24 h (AQ–24h)
Profundidade de leitura (**)	100 µm	x	x	x	x	x	x	x	x	x	x
	300 µm	x	x	x	x	x	x	x	x	x	x
	500 µm	x	x	x	x	x	x	x	x	x	x
	700 µm	x	x	x	x	x	x	x	x	x	x

(*) Tempo decorrido desde o início da manipulação. 10 e 20 minutos representam o intervalo de tempo onde a leitura de microdureza era realizada. Para o cimento resinoso de ativação química houve um tempo adicional de 10 minutos para remoção dos espécimes da matriz, para evitar a deformação dos mesmos, durante a remoção da matriz. (**) Profundidade medida a partir da superfície externa do cimento, voltada para a cerâmica.Os grupos Fd, Fac0,7, Fac1,4 e Fac2,0 correspondem ao modo de ativação foto/quimicamente ativado (*Dual-cure*), enquanto o grupo AQ corresponde ao modo ao de ativação química (*Self-cure*)

2A. Materiais e métodos

Artigo: *"Effect of energy density through ceramic with different light units on microhardness of a dual-cure resin cement"*

Efeito da dosagem de energia através de estrutura cerâmica com diferentes fontes de luz sobre a microdureza de um cimento resinoso de dupla ativação.

2A.1. Confecção de espécime de cerâmica

O material selecionado para o estudo foi a cerâmica odontológica Duceram Plus, (Ducera, Dental GmbH&Co.KG, Alemanha), cor dentina A3 da escala Vita (Vita Zahnfabrik H, Rauter GmbH & Co. KG, Bad Säckingen, Alemanha), classificada como cerâmica feldspática (Tabela 1A.1). O material foi manipulado e, em seguida, condensado em um molde metálico. Obteve-se um cilindro de cerâmica com 8,0 (±0,01) mm de diâmetro. O material foi levado para sinterização em um forno para cerâmica (Austromat M, Dekema Austromat-Keramiköfen, Freilassing, Alemanha). Todas as etapas foram executadas com base nas instruções dos fabricantes dos materiais e equipamentos que estavam sendo utilizados.

O cilindro, obtido do processo de sinterização, foi seccionado com disco diamantado sob refrigeração em água, montado em cortadeira, regulada para corte em baixa rotação. Após acabamento com lixa d'água de granulação decrescente (180, 320, 400, 600 e 1200) instaladas em politriz metalográfica, obteve-se um disco de cerâmica com 2,0 (±0,01) mm de espessura e 8,0 (±0,01) mm de diâmetro, que foi utilizado no experimento. Após o acabamento, o disco foi levado novamente ao forno para o processo de glazeamento. Todas as medidas eram realizadas com paquímetro digital com precisão de 0,01 mm (Starrett, Elmhurst, IL, EUA).

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2A.2. Obtenção dos espécimes com cimento resinoso.

Para o presente estudo o cimento resinoso Enforce com flúor (Dentsply Indústria e Comércio Ltda, Petrópolis, RJ, Brasil) foi selecionado para uso (Tabela 2A.1.) Uma matriz em resina acrílica confeccionada a partir da moldagem de esferas metálicas foi utilizada para garantir a padronização de dosagem entre as pastas do cimento resinoso. As pastas base e catalizadora do cimento resinoso eram dosadas, por volume, em proporções equivalentes. A mistura era realizada durante 20 segundos, com o auxílio de espátula n° 24 e bloco de papel descartável, conforme as instruções do fabricante. Após ser manipulado, o cimento era inserido na matriz de nylon, até o completo preenchimento do espaço reservado para o cimento. Um filme de poliéster (±25 μm de espessura) era

colocado sobre o cimento resinoso e a matriz para evitar o contato do cimento com a cerâmica. Todos os procedimentos desde a manipulação do cimento até a obtenção dos espécimes eram realizados em ambiente de temperatura (23°C ±1°C) e umidade controlada (acima de 30% de umidade relativa) conforme especificação da ISO 4049.

Uma marcação era realizada na matriz para permitir o alinhamento entre a ponta da sonda condutora de luz do aparelho e o orifício da matriz, garantindo a irradiação de toda a extensão do cimento resinoso. Durante o planejamento da pesquisa, tomou-se o cuidado de certificar-se que o diâmetro do orifício da matriz que era preenchido por cimento resinoso, fosse menor que o diâmetro de saída de luz, na ponta da sonda dos aparelhos fotoativadores. Essa medida visou garantir uma completa exposição do cimento resinoso ao longo de sua extensão, na abertura da matriz.

2A.3. Aparelhos fotopolimerizadores

Três diferentes aparelhos para fotoativação foram utilizados. A escolha dos aparelhos foi baseada no modo de produção de luz, pelo aparelho. Como aparelho fotoativador por emissão de luz halógena, produzido a partir de lâmpada com filamento de tungstênio, foi utilizado o aparelho fotoativador XL 2500 (3M ESPE, St Paul, MN, USA). O aparelho com luz emitida por diodo (LED – *Light emitting diode*) - Ultrablue Is, (D.M.C. Equipamentos Ltda), foi escolhido para representar a classe dos LEDs. Como dispositivo para foto ativação de compósitos, com luz

emitida a partir de arco de plasma de xenônio, foi utilizado o aparelho Apollo 95E (DMD – Medical Diagnostic Systems, Westlake Village, CA, USA). A Tabela 2A.2 informa as principais características dos aparelhos utilizados.

Todos os aparelhos foram ligados à corrente elétrica, estabilizada, por intermédio de estabilizador de voltagem, com o intuito de controlar eventuais oscilações na corrente elétrica, que por sua vez poderiam alterar a irradiância final do dispositivo.

Um suporte adaptado foi utilizado para garantir uma angulação de 90° com a ponta ativa da sonda do aparelho e o local a ser irradiado. Essa medida foi adotada com intuito de padronizar o posicionamentop da sonda do aparelho. Por intermédio de radiômetro portátil, (Hilux, Dental Curing Ligth Meter, Benbionglu Dental Inc.), com capacidade de leitura de 0 a 2000 mW/cm^2, dez aferições foram realizadas em intervalos semelhantes aos utilizados durante a execução da metodologia (irradiação de 40s com intervalos de 2-5 minutos entre os acionamentos). Obteve-se então uma média de irradiância desenvolvida para cada aparelho, esta média foi utilizada para o cálculo do tempo de irradiação para cada dosagem de energia.

Adicionalmente os discos de cerâmica foram adaptados ao sensor de potência do aparelho radiômetro. O disco foi irradiado com cada um dos três aparelhos. Foram obtidos valores de irradiância correspondente à potência de luz emergente da cerâmica e captada pelo sensor do radiômetro. Essas informações estão listadas na tabela 2A.2.

Para cada um dos três aparelhos fotoativadores, luz halógena (HAL), emitida por filamento de tungstênio, luz emitida por diodo (LED) e luz emitida por arco de plasma de xenônio (APX) foram empregadas 3 modos diferentes de dosagens de energia. Cada combinação entre aparelho de dosagem formava um grupo (n=10). Nessas circunstâncias o cimento resinoso foi utilizado no modo de dupla ativação, com ativação foto/química, através de estrutura cerâmica.

Fórmula para cálculo de dosagem, utilizada para cada aparelho.

$$D = \frac{P}{A} \times T = \frac{mW}{cm^2} \times s = \frac{mJ}{cm^2}$$

Onde “P” é a potência do aparelho dada em mW, “A” é a área da ponta da sonda do aparelho. “T” é o tempo de irradiação (emissão de luz) em segundos. Como potência do aparelho dividido pela área corresponde ao valor de irradiância, foi usado o valor obtido pelo radiômetro. “D”= Dosagem de energia, que é dada em mJ/cm^2 (correspondente a utilização de 1mW por $1cm^2$ durante o tempo de 1 segundo), por razões de simplificação utilizou-se neste estudo J/cm^2 (1J=1000mJ). “mW/cm^2 “ – MilliWatt por centímetro quadrado, irradiância do dispositivo de foto ativação medida por radiômetro.

Tempo e potência foram dosados para cada aparelho, com a finalidade de obter-se três condições de dosagem listadas a seguir: 1- dosagem de $\underline{24\ J.cm^{-2}}$

(IxT), ou seja, o valor de irradiância do aparelho utilizado por tempo regular (recomendado pelo fabricante do cimento resinoso, o valor tomado como referência foi 40 segundos de irradiação com dispositivo halógeno com cerca de 600 mW/cm^{-2} de irradiância), resultando assim em uma dosagem de energia de 24 joules por centímetro quadrado. 2- dosagem de 24 J.cm^{-2} (I/2x2T), que era aplicada com a metade do valor da irradiância máxima para cada aparelho, mas com o dobro do tempo de irradiação que a situação anterior. Essa situação simulava o uso de um aparelho com baixa irradiância por tempo prolongado 3- 48 J.cm^{-2} (Ix2T) nessa situação a capacidade de irradiância máxima do aparelho era mantida constante e o tempo era duplicado, duplicando-se assim a dosagem energética. Essa última situação simulava o procedimento de irradiação prolongada com a capacidade máxima de cada aparelho. O delineamento experimental utilizado neste estudo está representado na Tabela 2A.3.

Em um grupo, a fotoativação não foi realizada, ou seja, o cimento era utilizado no modo somente ativação química. Para confecção desse grupo as luzes convencionais eram desligadas e uma lâmpada com filtro vermellho para revelação radiográfica foi acionada de modo a não proporcionar exposição do compósito a luz com comprimento de onda necessário para a ativação do cimento resinoso. Desse modo minimizava-se a chance de fotoativação do compósito.

Na etapa que compreendia a preparação dos corpos-de-prova para leitura de microdureza, os mesmos foram fixados em uma matriz pré-moldada em resina acrílica, incluída em PVC. Os discos foram adaptados nos espaços previamente moldados. Eventuais desajustes foram preenchidos pela adição de cera pegajosa

plastificada previamente a colocação dos discos. Testes preliminares não indicaram a interferência desse procedimento nos valores de microdureza coletados.

Após a fixação do(s) disco(s) 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, sendo descartada em seguida, para fins de padronização do procedimento.

2A.5. Leitura da Microdureza

O teste de dureza Knoop era realizado em aparelho para ensaios universal de microdureza, microdurômetro HMV-2 (Shimadzu, Tokyo, Japan) com procedimento automático de aplicação de 50g de carga durante 15 segundos (Figura 2A.1). As leituras de microdureza foram realizadas mediante aumento de 40x. Após a calibração manual o aparelho convertia automaticamente a distância entre as extremidades da diagonal maior do losango (formato resultante após a edentação no compósito que era mensurado pelo operador), em valores de dureza Knoop.

Para cada corpo-de-prova, uma médias de dureza Knoop era obtida, correspondente à profundidade de 100 μm da superfície voltada para o meio externo da matriz. Cada média correspondia à 3 valores de microdureza, 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.

Os valores de microdureza foram tabulados em planilha do programa Excel do pacote Office XP para Windows e transferidos para o programa SigmaStat 3.0 for Windows (SPSS Inc.) para a análise estatística dos dados.

Análise de variância segundo dois critérios (forma de ativação e aparelho) foi utilizada para identificar possíveis interações entre os fatores investigados. O método de comparações múltiplas de Holm-Sidak (teste *Post-Hoc*) foi aplicado para realizar as comparações entre as médias. O nível de significância utilizado em todos os testes foi de 5% (α=0,05).

Todos os grupos foram comparados com o grupo quimicamente ativado (controle) através da Análise de variância segundo um critério e método de comparações múltiplas de Holm-Sidak. Todos os grupos formados pela associação entre aparelho e dosagem foram comparados com o modo de ativação químico (controle).

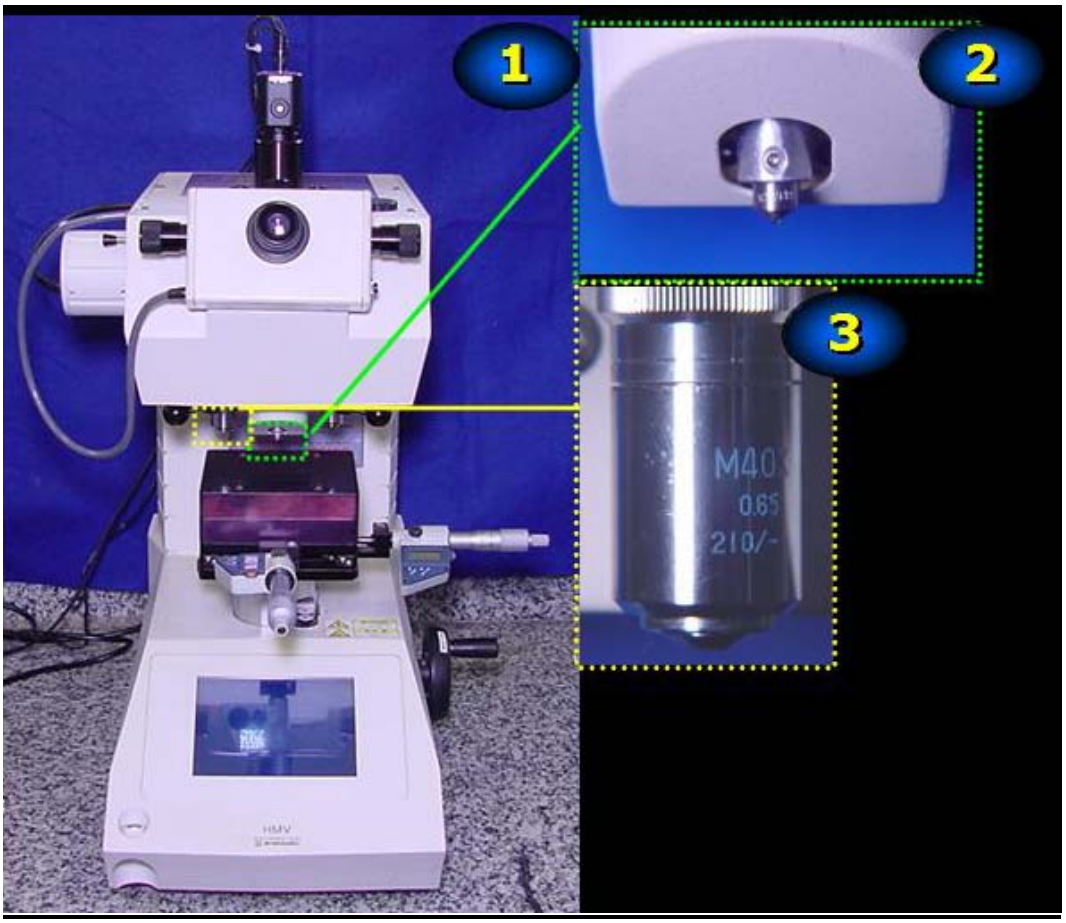

Figura 2A.1. (1) Aparelho universal para ensaios de dureza, (2) ponta edentadora utilizada para aplicação de carga para dureza Knoop (3) lente de aumento de 40X utilizada na leitura de microdureza.

Tabela 2A.1. – Caracterização do cimento resinoso utilizado neste estudo

Cimento resinoso	Composição (66% de carga por peso)	Lote	Manipulação
Pasta base matizada Enforce com Flúor (Dentsply Indústria e comércio Ltda. Petrópolis-RJ, Brasil)	TEGDMA, BDMA, Vidro de Boro Silicato de Alumínio e Bário Silanizado Sílica pirolítica silanizada Canforoquinona EDAB, BHT, DHEPT Pigmentos minerais	L64877	Proporcionamento por volume em partes semelhantes Mistura com espátula por 15-20 segundos
Pasta catalizadora Enforce com Flúor (Dentsply Indústria e comércio Ltda. Petrópolis-RJ, Brasil)	Dióxido de titânio Sílica pirolítica silanizada Pigmento mineral Resina Bis-GMA BHT, EDAB, TEGDMA Peróxido de Benzoíla	L64384	

Bis-GMA: bisfenol A diglicidil éter dimetacrilato; TEGDMA: Trietileno glicol dimetacrilato; BHT: 2,6-Di-tert-butil-4-metilfenol; EDAB: 4-etil diaminobenzoato; EBPADM; Bis-fenol-A-dimetacrilato etoxilado

Tabela 2A.3. Delineamento experimental

Aparelhos para fotoativação	Sem cerâmica		Com 2,0 mm de cerâmica feldspática interposta entre a sonda de fibra óptica e o cimento resinoso (*Fac)*		
	Sem fotoativação	Irradiação direta sobre o cimento (*FaD*)	24 J.cm^{-2}(I/2x2T)	24 J.cm^{-2}(IxT)	48 J.cm^{-2}(Ix2T)
Halógena	——	*I*– 589 mW/cm^2 *T* – 40 s *S* – 1 *D* – 0 mm	*I*– 300 mW/cm^2 *T* – 40 s *S* – 1 *D* – 7,7 mm + cerâmica	*I* – 589 mW/cm^2 *T* – 40 s *S* – 1 *D* – contato com cerâmica	*I* – 589 mW/cm^2 *T* – 82 s *S* – 1 *D* – contato com cerâmica
LED	——	*I* – 613,8 mW/cm^2 *T* – 40 s S– 1 *D* – 0 mm	*I*– 300 mW/cm^2 *T* – 40 s *S* – 1 *D* – 4,8 mm + cerâmica	*I*– 613,8 mW/cm^2 *T* – 40 s S – 1 *D* – contato com cerâmica	*I* – 613,8 mW/cm^2 *T* – 78 s *S* – 2 *D* – contato com cerâmica
APX	——	*I* – 1653 mW/cm^2 *T* – 3 s *S* – 1 *D* –0 mm	*I* – 825 mW/cm^2 *T* – 29 s *S** – 9 + 1 (2s) *D* – 4,75 + cerâmica	*I*– 1653 mW/cm^2 *T* – 15 s *S** – 5 *D* – contato com cerâmica	*I* – 1653 mW/cm^2 *T* – 29 s *S** – 9 + 1 (2s) *D* – contato com cerâmica

"I": Irradiância ou intensidde média do aparelho, utilizada para os cálculos de dosagem de energia. "I/2"; Quando o aparelho é utilizado com metade de seu potencial de radiância. "T": Tempo de irradiação utilizado para cada dispositivo. "S": Seqüências em que o aparelho era acionado para emitir luz durante o tempo desejado.(*) Para o grupo APX aguardava-se 3 segundos entre cada acionamento, por questões técnicas relativas à fonte de luz. "D": Distância entre a ponta dos respectivos dispositivos e a superfície do cimento resinoso.

Tabela 2A.2. – Caracterização dos dispositivos para fotoativação de compósitos resinosos.

Aparalho	Fonte de Luz	Tipo de Sonda	Tempo de máx. de acionamento	Irradiância média (*) mW/cm^2 e Desvio Padrão (D.P.)	Irradiância de saída de cerâmica, mW/cm^2 (Perda %)
XL 2500 (3M ESPE Dental Products, St. Paul, MN, USA)	Halógena (1 Lâmpada com filamento incandescente de tungstênio)	Fibra óptica Ø (Diâmetro) Entrada – 7,38 mm Ø Saída – 6,94 mm	Indeterminado	589,2 (12,14)	15 (97,5%)
Ultrablue Is (D.M.C. Equipamentos LTDA, São Carlos, SP, Brasil).	LED (*Light-emitting diode*) (1 LED - 5 Watts)	Sonda convergente Ø Entrada – 14,9 mm Ø Saída – 8,02 mm	45 s	613,8 (9,39)	21 (96,6%)
Apollo 95SE (DMD – Medical Diagnostic Systems, Westlake Village, CA, USA)	Lâmpada emissora de luz a partir de Arco de plasma de xenônio Potência em Watts – não informada pelo fabricante	Cabo com líquido condutor óptico de 1.800 mm Ø Entrada – 7,0 mm Ø Saída – 8,0 mm	3 s	1656,1 (32,03)	83 (95%)

(*) Para o cálculo da potência média foram utilizadas os valores após 5 segundos para fonte de luz halógena e LED, para o arco de plasma de xenônio foi utilizado o valor final captado pelo radiômeto após 3 segundos.

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FACULDADE DE ODONTOLOGIA DE PIRACICABA

Piracicaba, 29 de março de 2004.

Dr. Michael A. Cochran
Editor - Operative Dentistry
Indiana University School of Dentistry
Indianapolis - Indiana - USA

Dr. Cochran,

I am sending the manuscript "**Effect of energetic density with different light sources upon knoop hardness of a dual-cured resin-based cement**". I would like to submit this manuscript to the publication in the Operative Dentistry..

Sincerely,

Lourenço Correr Sobrinho
corresponding