



UNIVERSIDADE ESTADUAL DE CAMPINAS
FACULDADE DE ODONTOLOGIA DE PIRACICABA

LUCAS DUTRA RISSATO

**EFEITO DO ATRASO NA FOTOATIVAÇÃO DE COMPÓSITOS
BULK-FILL DUAIS NA ADAPTAÇÃO INTERNA E
PROFUNDIDADE DE POLIMERIZAÇÃO**

**DELAYED LIGHT CURING OF DUAL-CURE BULK-FILL
COMPOSITES ON INTERNAL ADAPTATION AND DEPTH OF CURE**

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**DELAYED LIGHT CURING OF DUAL-CURE BULK-FILL
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Trabalho de Conclusão de Curso apresentado à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para obtenção do título de Cirurgião Dentista.

Undergraduate final work presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Dental Surgeon.

Orientador: Prof. Dr. Américo Bortolazzo Correr

Coorientador: May Anny Alves Fraga

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RESUMO

Objetivo: Avaliar o efeito do atraso na fotopolimerização de compósitos bulk-fill duais na adaptação interna e microdureza (KHN) em profundidade. **Materiais e Métodos:** Trinta e cinco dentes foram preparados em forma de caixa e restaurados de acordo com os seguintes protocolos (n = 5): Filtek Bulk-Fill fotopolimerizado imediatamente após a inserção (FBF); Bulk EZ fotopolimerizado imediatamente após a inserção (BEZ-I); Bulk EZ fotopolimerizado 90 segundos após a inserção (BEZ-DP); Bulk EZ autopolimerizado (BEZ-SC); HyperFIL fotopolimerizado imediatamente após a inserção (HF-I); HyperFIL fotopolimerizado 90 segundos após a inserção (HF-DP); HyperFIL autopolimerizado (HF-SC). Após 24 horas, as amostras foram seccionadas axialmente e a adaptação interna foi avaliada usando réplicas em microscópio eletrônico de varredura (MEV). A KHN foi avaliada em seis profundidades (0,3; 1; 2; 3; 4; 5 mm). A análise estatística foi realizada utilizando $\alpha = 0,05$. **Resultados:** KHN diminuiu significativamente com a profundidade, exceto no modo de autopolimerização, quando KHN foi semelhante em todas as profundidades. O atraso na fotopolimerização aumentou significativamente a KHN em profundidades maiores. A adaptação interna foi dependente do material. A fotopolimerização não influenciou a adaptação interna de HyperFIL, enquanto o atraso na fotopolimerização reduziu significativamente as fendas internas (%) de Bulk EZ. **Conclusão:** O atraso na fotopolimerização melhorou a profundidade de polimerização das resinas compostas bulk-fill duais. A fotopolimerização não influenciou a adaptação interna do HyperFIL, mas o atraso na fotopolimerização melhorou a adaptação interna de Bulk EZ.

Palavras-chave: Resinas Compostas. Polimerização. Adaptação Marginal Dentária.

ABSTRACT

Objective: To evaluate the effect of delayed light curing of dual-cure bulk-fill composites on internal adaptation and microhardness (KHN) in depth. **Materials and Methods:** Bulk-fill composites were placed in thirty-five box-shaped preparations and cured according to the following protocols (n = 5): Filtek Bulk-Fill light cured immediately after insertion (FBF); Bulk EZ light cured immediately after insertion (BEZ-I); Bulk EZ light cured 90 seconds after insertion (BEZ-DP); Bulk EZ self-cured (BEZ-SC); HyperFIL light cured immediately after insertion (HF-I); HyperFIL light cured 90 seconds after insertion (HF-DP); HyperFIL self-cured (HF-SC). After 24 hours, the samples were axially sectioned and internal adaptation was evaluated using replicas under a scanning electron microscope. KHN was evaluated in six depths (0.3; 1; 2; 3; 4; 5 mm). The statistical analysis was performed using $\alpha = 0.05$. **Results:** KHN significantly decreased with depth, except for in self-curing mode, when KHN was similar in all depths. Delayed light curing significantly increased KHN at higher depths. Internal adaptation was material-dependent. Light curing didn't influence internal adaptation of HyperFIL, while delayed light curing significantly reduced internal gaps (%) of Bulk EZ. **Conclusion:** The delayed light curing improved the depth of cure of dual-cure resin composites. Light curing didn't influenced internal adaptation of HyperFILL, but delayed light curing improved internal adaptation of Bulk EZ.

Key words: Resin composites. Polymerization. Dental Marginal Adaptation.

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1 INTRODUÇÃO

Os materiais utilizados na prática clínica odontológica foram evoluindo e sendo desenvolvidos ao longo do tempo e com o grande avanço dos estudos e tecnologia. Dentre eles se apresentando com grande destaque estão os materiais restauradores, que não diferentemente passaram por grandes transformações. Deve-se ressaltar a redução da utilização do amálgama de prata como material restaurador definitivo, oferecendo espaço para a soberania das resinas compostas, visto que suas propriedades mecânicas e estéticas possibilitam uma maior viabilidade de procedimentos mais conservadores com um menor desgaste da estrutura dental remanescente (Fernandes et al., 2014).

Assim, os preparos cavitários tradicionais, com dimensões maiores do que o necessário, que desgastavam uma quantidade significativa de estrutura dental sadia para serem confeccionados, tiveram sua utilização bem reduzida para a utilização de preparos restauradores mais conservadores. E isso se deve essencialmente da grande evolução dos sistemas adesivos, que possibilitou essa grande mudança na prática clínica da Odontologia restauradora (Alex, 2015).

Além disso, os compósitos restauradores resinosos tiveram cada vez mais suas partículas reduzidas para possibilitar um melhor acabamento e polimento. Outros estudos, buscaram diminuir a contração de polimerização destes materiais, a qual se apresentava como uma das maiores desvantagens da utilização dos compósitos resinosos (Fernandes et al., 2014).

Neste contexto, devido aos avanços obtidos na redução da tensão de contração de polimerização e buscando obter maior facilidade na utilização dos compósitos resinosos, além de superar o limite de 2 mm por incremento existente nos compósitos tradicionais, foram desenvolvidos compósitos bulk-fill, os quais foram lançados no mercado nacional em 2010, estando disponíveis comercialmente nas viscosidades fluída e regular (Silva et al., 2010).

Os compósitos bulk-fill se diferem dos compósitos tradicionais pois podem ser inseridos em incrementos de até 4 mm de profundidade. Isso é possível devido a maior translucidez destes compósitos em relação aos tradicionais (Ende et al., 2016). Para isto, foram realizadas modificações na matriz orgânica e nas partículas de carga, objetivando também diminuir as tensões geradas pela contração de polimerização. Na matriz orgânica foram adicionados monômeros mais flexíveis e de maior peso molecular e alterado o sistema iniciador (a partir de novos fotoiniciadores ou até maior concentração dos tradicionais). As modificações nas cargas ocorreram pela adição de partículas maiores e/ou com índice de

refração similar a matriz orgânica. Com essas modificações, foi demonstrado que os compósitos resinosos bulk-fill podem reduzir a deformação das cúspides e o estresse de polimerização, bem como aumentar a resistência à fratura (Fugolin e Pfeifer, 2017).

Assim, a utilização deste tipo de compósito faz com que o procedimento seja realizado de maneira mais rápida e fácil, diminuindo o número de passos clínicos quando comparada a técnica incremental. Entretanto, a efetividade da polimerização em profundidade e a desadaptação interna devem ser cuidadosamente analisadas, visto que tais problemas ainda ocorrem com os compósitos bulk-fill (Benetti et al., 2015). Também foi relatado que a técnica incremental com compósitos tradicionais produz melhor adaptação interna quando comparada a técnica de restauração em incremento único utilizando compósitos do tipo bulk-fill (Alqudaihi et al., 2019). Além disso, os compósitos bulk-fill possuem, em geral, menor microdureza em profundidade quando comparado aos compósitos convencionais (Kelić et al., 2016).

Tendo em vista as características citadas, buscando suprir as desvantagens e carências dos compósitos bulk-fill convencionais, surgiram os compósitos bulk-fill duais, que são comercialmente apresentados como duas pastas (Fraga et al., 2021). A ativação química desses compósitos poderia suprir os problemas gerados pela atenuação da luz em profundidade e melhorar a polimerização nesta região. Além disso, a ativação química ocorre de forma mais lenta, atrasando o ponto gel do material. Um estudo que avaliou um compósito desta classe de materiais demonstrou que, apesar de sofrer contração volumétrica, o material tem melhor adaptação interna e maior uniformidade de polimerização do topo da restauração em relação a base (Hayashi et al., 2019). Além disso, uma menor viscosidade de materiais bulk-fill, duais ou fotoativados demonstrou reduzir a formação de fendas marginais e internas em preparos do tipo classe II (Sampaio et al., 2020). Adicionalmente, compósitos bulk-fill duais demonstram também um maior grau de conversão em comparação com os compósitos bulk-fill fotopolimerizáveis, quando em mesmas condições de polimerização. Além de variações menores de microdureza em profundidade quando preparos de 6 mm foram avaliados (Wang e Wang, 2020).

Atrasar o ponto gel de materiais duais tem sido uma alternativa utilizada em cimentos resinosos para reduzir as tensões geradas podendo ser benéfica para a cimentação de restaurações indiretas em dentes vitais, sem afetar as propriedades mecânicas (Soares et al., 2016). Em compósitos bulk-fill duais, essa medida demonstrou reduzir a flexão de cúspide e não afetar as propriedades mecânicas do material (Hughes et al., 2019). Entretanto, o procedimento de atraso na fotoativação não é um consenso entre os protocolos de aplicação

estabelecidos pelos fabricantes desses compósitos, o que justifica o estudo de diferentes protocolos de fotoativação para esses materiais.

O objetivo neste estudo foi avaliar se o atraso na fotoativação diminui a formação de fendas e melhora a polimerização em profundidade, a partir da utilização de compósitos bulk-fill duais.

2 ARTIGO: DELAYED LIGHT CURING OF DUAL-CURE BULK-FILL COMPOSITES ON INTERNAL ADAPTATION AND DEPTH OF CURE

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Objective: To evaluate the effect of delayed light curing of dual-cure bulk-fill composites on internal adaptation and microhardness (KHN) in depth. **Materials and Methods:** Bulk-fill composites were placed in thirty-five box-shaped preparations and cured according to the following protocols (n = 5): Filtek Bulk-Fill light cured immediately after insertion (FBF); Bulk EZ light cured immediately after insertion (BEZ-I); Bulk EZ light cured 90 seconds after insertion (BEZ-DP); Bulk EZ self-cured (BEZ-SC); HyperFIL light cured immediately after insertion (HF-I); HyperFIL light cured 90 seconds after insertion (HF-DP); HyperFIL self-cured (HF-SC). After 24 hours, the samples were axially sectioned and internal adaptation was evaluated using replicas under a scanning electron microscope. KHN was evaluated in six depths (0.3; 1; 2; 3; 4; 5 mm). The statistical analysis was performed using $\alpha = 0.05$. **Results:** KHN significantly decreased with depth, except for in self-curing mode, when KHN was similar in all depths. Delayed light curing significantly increased KHN at higher depths. Internal adaptation was material-dependent. Light curing didn't influence internal adaptation of HyperFIL, while delayed light curing significantly reduced internal gaps (%) of Bulk EZ. **Conclusion:** The delayed light curing improved the depth of cure of dual-cure resin composites. Light curing didn't influence internal adaptation of HyperFILL, but delayed light curing improved internal adaptation of Bulk EZ.

Clinical Significance: Light curing is fundamental for improving mechanical properties of dual-cure resin composites; moreover, depending on dual-cure resin composite, the delay in light curing can reduce the internal gaps.

Keywords: Bulk-fill resin composite, internal adaptation, microhardness, light curing, dual cure bulk-fill.

1. Introduction:

Bulk-fill resin composites gain popularity due to the possibility to be placed in a single increment of 4-5 mm thickness. Changes were made in the organic matrix and filler particles to reduce the polymerization shrinkage stress ¹ and increase translucency compared to the traditional resin composites. ² More flexible and higher molecular weight monomers were added, as well as the initiator system was changed. ³⁻⁵ The size of the filler increased and its refractive index was closest to that of the organic matrix. These modifications reduced the cusp deformation and the polymerization stress, as well as increase fracture resistance. ⁶

Bulk fill composite makes the restoration technique faster and easier, decreasing the number of clinical steps when compared to the incremental technique. ⁷ Nevertheless, gaps are formed in the bottom of restoration ⁸ and light is attenuated in bulk fill composites, impairing the degree of conversion at higher depths. ⁹ It has also been reported that the incremental technique produces better internal adaptation when compared to the single increment restoration technique using bulk-fill composites. ¹⁰ Furthermore, bulk-fill composites have lower microhardness at higher depths than conventional composites used with incremental technique. ¹¹

Dual-cure bulk-fill composites have emerged to overcome issues related to light attenuation and improve the depth of polymerization. ¹² Self-cure doesn't depend on light. Moreover, it reduces polymerization kinetics and polymerization occurs slowly, delaying the gel point. A previous study found the dual-cured bulk-filled resin composite showed no decrease of degree of conversion through the depth and the highest cavity adaptation despite its tendency for higher volumetric shrinkage. ⁸ Additionally, dual bulk-fill composites also demonstrated a higher degree of conversion than photopolymerizable bulk-fill composites under the same polymerization conditions. Such materials showed smaller variations in microhardness at a depth when 6 mm preparations were evaluated. ¹³

It was found that delayed light activation reduce the polymerization stress of the resin cements without jeopardizing degree of conversion. ¹⁴ In dual-cure bulk-fill composites, delayed light curing reduce shrinkage stresses without negatively affecting the degree of cure or mechanical properties. ¹⁵ Bulk EZ manufacturer recommend that light curing be made at least 90 seconds after mixing, when 90 seconds after past mixing, while HyperFIL manufacturer recommend that resin composite be light cured immediately after past missing. It was found that dual-cure resin composite light cured 90 seconds after mixing had microhardness similar from the top to 5 mm depth, while dual-cure resin composite light cured immediately after mixing showed a reduction in microhardness at higher depths. The light

curing delay procedure is not a consensus for dual-curing resin composites. Finally, by delaying light curing, improved internal adaptation and depth of polymerization can be expected.

Therefore, the aim of this study was to evaluate the effect of the delay in light curing on internal adaptation and depth of cure of dual-cure bulk-fill composites. The hypotheses tested were that: (1) there would be influence of restorative protocols on internal adaptation of resin composites and, (2) there would be influence of restorative protocols on microhardness of resin composites.

2. Materials and methods

Five restorative protocols were evaluated in this study (Figure 1). The resin composite materials are described in table 1.

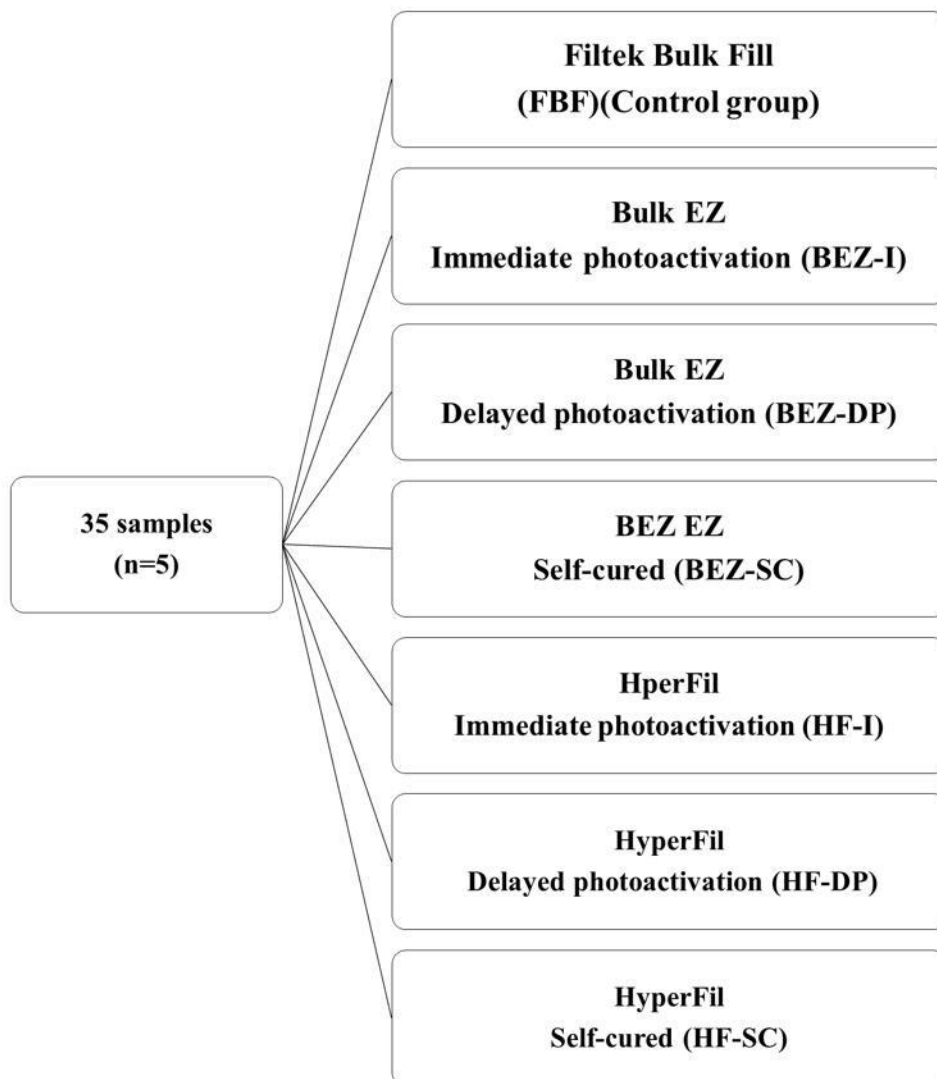


Figure 1 - Organizational chart of the experimental design

Table 1. Composite resins, manufacturer and composition.

Material	Manufacturer	Matrix composition	Filler type/loading (wt%)	Shade
Bulk EZ	Danville Materials, Carlsbad, CA, USA	Monomers; EBPDMA, TEGDMA, BisGMA, UDMA	Barium glass, Ytterbium, Trifluoride 50–70 wt%	A2
HyperFIL TM DC	Parkell, New York, NY, USA	Monomers; BisEMA, UDMA, and other dimethacrylate monomers.	Barium glass/silica 70-75 wt%	Universal
Filtek Bulk-fill flow	3M ESPE, SL Paul, MN, USA	Bis-GMA, Bis- EMA(6), UDMA, TEGDMA, Procrylat resins	Zirconia/silica, ytterbium, trifluoride 64.5%	A2

Abbreviations: BisGMA, bisphenylglycidyl dimethacrylate; BisEMA, ethoxylated bisphenol-A dimethacrylate; EBPDMA, 1,6-bis-[2-methacryloyloxyethoxycarbonylamino]-2,4,4-trimethylhexane; PEGDMA, Polyethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

Box-shaped preparation

This study was approved by the local Research Ethics. Thirty-five healthy human molars had their roots sectioned 3 mm below the cementum-enamel junction using a diamond disc mounted in a metallographic cutting machine (Isomet, Buehler Ltd., Lake Buff, IL, USA).

Box-shaped preparations were made on the teeth following the previous protocol proposed¹⁶. The teeth were positioned with the occlusal surface facing downward to allow the preparation to be made from the pulp chamber to the enamel direction. A preparation standardizing machine (Elquip, São Carlos, SP, Brazil) was used to prepare box-shaped preparations (4 mm wide x 6 mm long and 5 mm deep). Cylindrical diamond burs no. 3099 (KG Sorensen, São Paulo, SP, Brazil) were used, which were replaced after each 5 preparations. The preparations were finished with F and FF diamond tips of the same numbering.

Restorative procedure

Thirty-five samples were divided into seven groups (n = 5). The number of samples per group was based on previous studies.^{12,17} The Clearfil SE Bond 2 (Kuraray Noritake Dental, Tokyo, Japan) adhesive system was applied according to the manufacturer's instructions in all preparations. The primer was actively applied for 20 s and gently air-dried for 20 s. The bond was applied, gently air-dried, and photoactivated for 10 s with a multiple-peak LED device (Valo Cordless, 1,000 mW/cm² radiant exitance). The restoration was performed in a darkened environment with controlled humidity (50% ± 5) and temperature (25 °C ± 1). The restorative protocol for each group is described below:

- FBF: Filtek Bulk fill was placed in a single increment and immediately light cured for 20 seconds with an irradiance of 1,000 mW/cm² (Valo Cordless).

- BEZ-I: Bulk EZ was placed into the cavities in a single increment and immediately light cured for 20 seconds with an irradiance of 1,000 mW/cm² (Valo Cordless).

- HF-I: HyperFIL was placed into the cavities in a single increment and immediately light cured for 20 seconds with an irradiance of 1,000 mW/cm² (Valo Cordless).

- BEZ-DP: Bulk EZ was placed into the cavities in a single increment and after 90 seconds, light cured for 20 seconds with an irradiance of 1,000 mW/cm² (Valo Cordless).

HF-DP: HyperFIL was placed into the cavities in a single increment and after 90 seconds, light cured for 20 seconds with an irradiance of 1,000 mW/cm² (Valo Cordless).

- BEZ-SC: Bulk EZ was mixed, placed into the cavities in a single increment, and the specimens were stored at 37 °C ± 1 in a dark container.

HF-SC: HyperFIL was mixed, placed into the cavities in a single increment, and the specimens were stored at 37 °C ± 1 in a dark container.

Internal adaptation

The samples were axially cut into four slices in the occlusal-cervical direction using a diamond disc (Isomet Diamond Wafering Blades, No. 11-4244, Buehler Ltd., Lake Buff, IL, USA). The slices were polished with SiC abrasive papers (600, -1200 and -2000, Norton Abrasivos, Vinhedo, SP, Brazil) and diamond grits in suspensions of 6, 3, 1, and 0.05 µm (Buehler Ltd. Lake Bluff, IL, USA). After polishing, the samples were washed abundantly, dried and polyvinylsiloxane impressions (Adsil ultra soft+putty, and regular paste, Coltene, Bonsucesso, Rio de Janeiro, Brazil) were taken to obtain epoxy resin replicas (Buehler, Lake Bluff, IL, USA) of the slices. The replicas were fixed in stubs, gold-sputtered and analyzed in scanning electron microscopy (SEM). The images of the entire length of the bonding interface were analyzed at 75X magnification.

The SEM images of replicas were reconstructed with the aid of ImageJ software (NIH, Bethesda, MD, USA), obtaining the total perimeter of preparation and the length of gaps. The internal gaps (%) were expressed as a percentage of the length of gaps by the total length of the preparation.

Knoop microhardness (KHN)

The central slice of each sample was selected to perform the KHN test. Knoop microhardness analyses was carried out in a microdurometer HMV 2000 (Shimadzu, Tokyo, Japan) with a 50 gF (0.490 N) static load and 10 seconds indentation time. Three indentations

were made at 0.3 mm, 1 mm, 2 mm, 3 mm, 4 mm, and 5 mm and a mean calculated for each depth.

The microhardness percentage (KHN%) at 1 mm, 2 mm, 3 mm, 4 mm, and 5 mm was calculated according to the following equation: $MH\% = (KHN \text{ depth} / KHN \text{ top}) * 100$

Statistical analysis

Data were tested for normality (Shapiro-Wilk test) and homoscedasticity (Levene's test). Internal adaptation data were evaluated by one-way ANOVA and pairwise comparisons by Tukey's test. Microhardness data were evaluated by split-plot ANOVA and pairwise comparisons by Bonferroni test. For all analyses the level of significance was set to $\alpha=0.05$.

3. Results

Internal adaptation was significantly influenced by restorative protocols ($p = 0.001$). The internal adaptation results obtained from replicas are shown in Table 2. HF composite showed the highest percentages of gaps. Light curing methods didn't influence the internal adaptation of HF. BEZ had significantly less gap formation in self-cure and delayed light curing mode. FBF had internal gaps (%) similar to BEZ-DP and BEZ-SC, but significantly less gaps (%) than other groups.

The microhardness data are shown in Table 3. There was a significant interaction between factors (depth and restorative protocols, $p < 0.001$). When light cured, KHN significantly decreased with depth. KHN was similar in all depths when dual-cure resin composites were used in the self-curing mode. Bulk EZ and HyperFIL were not significantly influenced by light curing protocol (immediate or after 90 seconds) up to 3 mm depth. At higher depths (4 mm and 5 mm), delayed light curing significantly increases the KHN for Bulk EZ and HyperFIL. Light curing significantly increased KHN of Bulk EZ in all depths. Nevertheless, light curing did not increase KHN of HyperFIL at 5 mm depth.

Table 2. Mean and standard deviation of internal gaps (%).

Groups	Mean	Standard deviation	Tukey
FBF	45.9	7.0	B
BEZ-I	59.8	4.6	A
BEZ-DP	43.4	4.4	B
BEZ-SC	42.6	8.7	B
HF-I	69.8	3.6	A
HF-DP	59.4	6.9	A
HF-SC	65.4	4.5	A

* Different capital letters represent significant differences between groups.

The FBF group (control) had the lowest KHN values in all depths. In addition, KHN of FBF significantly decreased with depth.

Table 3. Mean and standard deviation of KHN values at different depths.

Group	Depth					
	0mm	1mm	2mm	3mm	4mm	5mm
FBF	31.3 (0.7) Ac	29.0 (1.8) Bd	27.6 (2.2) Cd	26.2 (2.7) Dd	24.5 (2.6) Ee	23.1 (2.4) Fd
BEZ-I	44.8 (0.9) Aa	42.2 (0.5) Ba	39.7 (1.2) Ca	37.4 (1.1) Dab	35.2 (0.8) Ec	32.4 (1.7)Fbc
BEZ-DP	44.5 (1.0) Aa	42.8(1.2) Ba	41.1 (0.6) Ca	39.1 (2.0) Dab	39.2 (1.0) Da	37.9 (0.9) Ea
BEZ-SC	31.1 (1.8) Ac	31.1 (1.7) Ac	31.2 (1.5) Ac	31.2 (1.6) Ac	31.3 (1.6) Ad	31.4 (1.6) Ac
HF-I	44.3 (1.7) Aa	41.8 (1.7) Ba	39.8 (1.8)Ca	38.1 (1.5) Dab	35.8 (0.7) Ebc	33.9 (1.2) Fb

HF-DP	44.9 (2.1) Aa	41.6 (1.7) Ba	40.5 (1.6) Ca	39.9 (1.4) Ca	39.5 (0.9) Da	38.6 (1.2) Ea
HF-SC	37.0 (0.6) Ab	37.4 (0.2) Ab	37.5 (0.2) Ab	37.5 (0.2) Ab	37.3 (0.3) Ab	37.3 (0.3) Aa

*Bonferroni test: Different capital letters represent significant differences between depths. Different lowercase letters represent significant differences between restorative protocols ($p < 0.05$).

KHN% data are shown in Figure 2. A black dashed line was also drawn to represent the limit boundary of 80% of the top/bottom KHN relation achieved by each resin composite. Self-cured groups (BEZ-SC and HF-SC) had similar KHN% at all depths. All the light-cured groups had decreased KHN% with depth; however, in the delayed light-curing groups (BEZ-DP and HF-DP) groups the top/bottom KHN relations were higher than 80% in all depths.

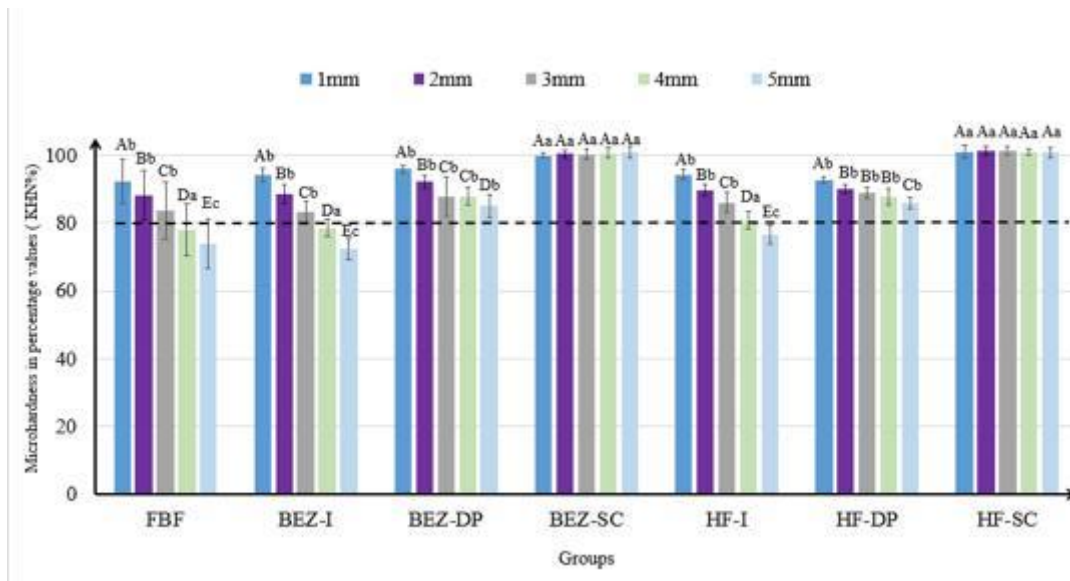


Figure 2. Microhardness in percentage values (MH%) for 1 mm, 2 mm, 3 mm, 4 mm and 5 mm in relation to the top for each sample group, according to the following equation: $KHN\% = (KHN \text{ depth} / \text{top of KHN}) * 100$. *Bonferroni test: Capital letters show differences between depths. Lowercase letters present differences between restorative protocols. The dotted line represents the threshold percentage top/bottom ratio considered suitable for depth polymerization.

4. Discussion

The internal adaptation of data were significantly affected by the restorative protocols. Thus, the first hypothesis of this study was accepted. Groups restored with HF showed higher gaps than others. Several factors are responsible for marginal misfit, such as viscosity of resin composite, cavity design, and restorative procedures. In addition, polymerization shrinkage stress may induce gap formation, which depends on degree of conversion and elastic modulus of resin composites.¹⁸ Previous studies found a higher degree of conversion in HF groups than in other resin composites.^{12,13,19} Although degree of conversion values improves the mechanical properties, it also may increase the polymerization shrinkage and, consequently, gap formation. In addition, HF composite presents a higher viscosity than the other materials evaluated in this study. The high viscosity impairs the flow of the resin composite, which can reduce the internal adaptation.²⁰

When immediately light cured, HF showed a similar gap formation than BEZ-I. It is recommended that BEZ be light cured only 90 seconds after its insertion to reduce the polymerization shrinkage stress. A previous study found that the delay period before light activation significantly reduced the maximum rate of polymerization of a resin cement similar to the reaction rate of the cements tested in self-cure mode.¹¹ Self-cure produces a more slowly autoacceleration by the action of free radicals formed by the oxy-reduction reaction, postponing the vitrification of the material.²¹ Therefore, more monomers are converted before vitrification reducing the polymerization shrinkage stress.²¹ When BEZ was self-cured (BEZ-SC) or light cured after 90 seconds (BEZ-DP), internal adaptation increased substantially, which demonstrates the positive effect of self-cure in reduce internal gaps. It was shown that delay in light curing of dual composites improved internal adaptation, microhardness in-depth and cusp flexion due to the reduction of polymerization shrinkage stress.²² Therefore, light curing after 90 seconds is important to improve the mechanical properties of dual-cure resin composites. These results corroborate with a previous study, in which BEZ presented less internal gaps than conventional bulk-fill composites.^{8,12} Conversely, in our study, HF did not show a reduction in the formation of gaps even when the light curing delay was performed. Thus, the behavior of dual-cure materials cannot be considered in a generalized manner, as it is material-dependent.

FBF, BEZ-SC or BEZ-DP had no significant differences in gap formation. As previously discussed, internal adaptation depends on the flowability of resin composite and polymerization shrinkage stress. FBF is a flowable bulk fill resin composite, which may improve

the adaptation due to the higher flow of the material in the cavity. Moreover, FBF has less filler content, which is related to a lower elastic modulus and, consequently, lower polymerization stress.²¹

KHN values were significantly influenced by restorative protocols. Thus, the second hypothesis of the study was accepted. Microhardness is a mechanical surface property that is related to the resistance of a material to scratching or permanent deformation when under pressure.²³ In Dentistry, microhardness provides subsidies on the clinical performance of the materials.²³ When the resin composite microhardness is evaluated at different depths, it may indicate the depth of polymerization of the material.²⁴

The control group (FBF) showed lower KHN values than BEZ and HF light-cured (BEZI, BEZ-DP, HF-I, and HF-DP). Microhardness is determined by several factors such as degree of conversion, organic matrix composition and filler particles content.²⁵ The higher the content (by volume) of cargo, the higher the KHN. FBF has a lower percentage of filler particles than HF and BEZ, which may be responsible for the lower hardness of this composite resin. Other studies have shown that dual bulk-fill composites present higher microhardness and degree of conversion than light-cured bulk-fill flowable materials.^{13,19} In addition, previous study found lower FBF microhardness compared to other bulk-fill flowable composite resins.

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FBF is a light curing resin composite. It is know that light is attenuated even in bulk fill composites, impairing the degree of conversion at higher depths. On the other hand, dual cure bulk-fill composites have two distinct ways of activation: oxy-reduction chemical polymerization, when the initiator molecule contacts an accelerator, and a polymerization activated by light when free radicals are formed from a dissociated molecule by a light cure unit.²⁶ Two mechanisms of photoactivation contribute to improve the microhardness in the groups BEZ-I, BEZ-DP, HF-I and HF-DP that received two kinds of activation. Thus, an advantage of dual-cure bulk-fill composites is the higher microhardness and degree of conversion than light-cured bulk-fill flowable materials.^{13,19}

On the other hand, in groups BEZ-SC and HF-SC (only self-cured), KHN values were lower compared to light-cured ones. Although the self-cure was more homogeneous, the lack of light curing can negatively affect the mechanical properties of these composites and, probably, their degree of conversion,²⁷ highlighting the relevance of light curing for the mechanical properties of these materials.²⁸ These data corroborate with previous studies that demonstrated that self-curing was unable to reach enough degree of conversion in dual bulk-fill composites.^{19,28}

For KHN% values, only BEZ-DP, BEZ-SC, HF-DP and HF-SC groups had microhardness values higher than 80% at 4 mm and 5 mm depth (Figure 2). According to the literature, an adequate depth of polymerization is achieved when top/bottom percentage ratio is greater than 80%.^{29,30} It is known that self-cure produces homogenous degree of conversion and microhardness because polymerization initiation doesn't depend on external factors. Although self-cured produced homogeneous polymerization in all depths (KHN% higher than 80%), the absolute microhardness were lower than in the light-cured groups. Thus, delay in light curing can be mentioned as an advantage because it does not affect the mechanical properties and provides adequate polymerization in depth.

In addition, immediate light curing (BEZ-I and HF-I) don't produce adequate polymerization up to 5 mm. It can be hypothesized that immediate polymerization reached the maximum polymerization rate of the composite (maximum R_p) more quickly, which may have increased the viscosity of the material suddenly. Increased viscosity leads to composite vitrification and decreased monomer mobility.³¹ This fact may have limited conversion by self-curing. For BEZ-DP and HF-DP, the 90-second delay in light curing allowed a greater action of self-curing, which becomes essential in deeper layers of the restoration, allowing these groups to have a less pronounced decrease in microhardness and maintenance of the mechanical properties at higher depths.

Thus, the light curing protocol is a determining factor in depth of polymerization of dual bulk-fill composites. When light-curing was performed after 90 seconds, adequate microhardness (KHN% 80% top in relation to the bottom) was achieved without reduction of the absolute microhardness, which did not occur in the self-cured group. Internal adaptation was material-dependent, but light-curing protocol can also reduce the gap formation. BEZ-DP restorative protocol showed more promising results regarding deep microhardness and internal adaptation. A limitation of this study was the lack of evaluation of the degree of conversion and the polymerization shrinkage stress. Further studies comparing the degree of conversion and polymerization shrinkage stress of these restorative protocols could be done to better elucidate why lower internal gaps and improved microhardness were found when light curing was done 90 seconds after mixing.

5. Conclusions

The delayed light curing can reduce the internal gaps in dual-cure resin composites and improves the polymerization in-depth.

Light curing is mandatory to maximize the mechanical properties of dual-cure resin composites.

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3 CONCLUSÃO

A partir deste estudo, pode-se concluir que a fotoativação apresenta-se como um aspecto fundamental para manutenção das propriedades mecânicas; entretanto, o atraso na fotoativação pode melhorar a polimerização em profundidade e reduzir as fendas internas de acordo com o material.

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^{1*} De acordo com as normas da UNICAMP/FOP, baseadas na padronização do International Committee of Medical Journal Editors - Vancouver Group. Abreviatura dos periódicos em conformidade com o PubMed.

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ANEXOS

Anexo 1 – Verificação de originalidade e prevenção de plágio

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PARECER CONSUBSTANCIADO DO CEP

DADOS DO PROJETO DE PESQUISA

Título da Pesquisa: Efeito do atraso do ponto gel na adaptação interna e profundidade polimerização de compósitos restauradores bulk fill duals

Pesquisador: May Anny Alves Fraga

Área Temática:

Versão: 2

CAAE: 30885220.3.0000.5418

Instituição Proponente: Faculdade de Odontologia de Piracicaba - Unicamp

Patrocinador Principal: Financiamento Próprio

DADOS DO PARECER

Número do Parecer: 4.044.066

Apresentação do Projeto:

Transcrição editada do conteúdo do registro do protocolo e dos arquivos anexados à Plataforma Brasil

A EQUIPE DE PESQUISA citada na capa do projeto de pesquisa inclui MAY ANNY ALVES FRAGA (Cirurgião Dentista, Mestranda no PPG em Materiais Dentários da FOP-UNICAMP, Pesquisadora Responsável), LUCAS DUTRA RISSATO (Graduando no curso de Odontologia da FOP-UNICAMP, Pesquisador participante) e AMÉRICO BORTOLAZZO CORRER (Cirurgião Dentista, Docente da área de Materiais Dentários da FOP-UNICAMP, Pesquisador participante), o que é confirmado na declaração dos pesquisadores e na PB.

Delimitação da pesquisa: Trata-se de estudo laboratorial comparativo que envolverá 35 dentes molares humanos obtidos doação de um dentista que os extraiu por razões clínicas e independentes da pesquisa. O objetivo neste estudo será avaliar se o atraso na fotoativação de compósitos bulk fill duals diminui a desadaptação interna e melhora a polimerização em profundidade. Para isso, serão confeccionadas 35 cavidades do tipo box-shaped, que serão restauradas conforme os grupos (n=5): Dois compósitos bulk fill duals (Bulk EZ e HyperFil) serão testados, variando os protocolos de ativação (1. Fotoativação Imediata, 2. Atraso de 90 segundos na fotoativação, 3. Ativação química), os quais serão comparados a um compósito bulk fill

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Outros	comentarios.pdf	00:24:50	Fraga	Acelto
Declaração de Pesquisadores	declarapesquisadores.pdf	21/04/2020 00:23:13	May Anny Alves Fraga	Acelto
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Situação do Parecer:

Aprovado

Necessita Aprovação da CONEP:

Não

PIRACICABA, 23 de Maio de 2020

Assinado por:
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Anexo 3 – Parecer do Assessor PIBIC



Relatório Final

Efeito do atraso no ponto gel na adaptação interna e profundidade polimerização de compósitos restauradores bulk fill duais

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 ver relatório (../arquivos/rel_final/AlunoCod_24946_1-RelFinal_2020.pdf)

— **Parecer do orientador emitido em 13/09/2021 08:53:37**

Desempenho do aluno no projeto: O desempenho do aluno no projeto foi excelente, se dedicou ao projeto de pesquisa e cumpriu o cronograma de pesquisa, mesmo com as dificuldades impostas pela pandemia. O bolsista participou de discussões e de outras atividades no laboratório de Materiais Dentários com alunos do Programa de Pós-Graduação em Materiais Dentários da FOP-UNICAMP.

Desempenho acadêmico do aluno: O aluno apresenta desempenho acadêmico excelente, estando classificado como 2º melhor em relação à sua turma de ingresso. Com relação ao rendimento acadêmico, o bolsista cumpriu satisfatoriamente as obrigações curriculares, apresentando ótimo desempenho acadêmico.

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O relatório final de atividades apresentado pelo aluno está excelente. Muito bem estruturado e discussão dos resultados apropriada. Recomendo a publicação dos achados em uma revista científica. O aluno cumpriu todas as atividades acadêmicas também de modo excelente, possuindo o segundo melhor CR de sua turma. Assim, recomendo a aprovação do relatório final de atividades.

● **Aprovado**

Anexo 4 –Comprovante de submissão



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Para: Você

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