# Effects of LED Lights on Bulk Fill Resin Polymerization

Efectos de las Luces LED en la Polimerización de Resina de Relleno Masivo

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**ABSTRACT:** The present study analyzed the microhardness and degree of conversion of three Bulk Fill resins (M1 - Filtek Bulk Fill; M2 - Tetric N-Ceram Bulk Fill and M3 - Opus Bulk Fill) polymerized by single peak and polywave Light-emitting Diode Curing Lights. A total 90 test specimens (n=10) were obtained using a Teflon matrix for the purpose of testing microhardness; and for degree of conversion: 135 specimens (n=5) by using a 2 x 6 cm matrix. The specimens were light polymerized using 3 light sources (L1 - Optilight Max, L2 - Bluephase, L3 - VALO). They were kept in artificial saliva om an oven at 37±1°C during the experiment. The degree of conversion was measured by FTIR 24 h after obtaining each test specimen. The microhardness readouts were performed with a microdurometer at the time intervals of 48 hours (T0), 7 days (T1), 14 days (T2) and 21 days (T3). M1L3 was found to show the highest microhardness values in T2, and M1 showed the lowest degree of conversion in the deep third with L1. It was concluded that Filtek Bulk Fill resin showed the best results in comparison with the other resins.

KEY WORDS: composite resins, dental material, hardness, degree of conversion, LED.

#### INTRODUCTION

Resins of the Bulk-Fill type were launched on the market to enhance the longevity of restorations, dispense with the time-consuming incremental technique, reduce the time it takes to perform the procedure due to the possibility of polymerizing increments of 4 to 5 mm thickness (de Araújo-Neto *et al.*, 2021; Al-Senan & Al-Nahedh, 2022). This was achieved by the addition of photoinitiators and greater translucence of this type of resin. However, adequate polymerization is necessary to meet the manufacturer's specifications and thus improve the long-term success of these restorations (Besegato *et al.*, 2019; Lempel *et al.*, 2023).

Incompletely polymerized restorations may fail prematurely, due to the increased incidence of secondary caries, failure to adhere to the tooth, defects in the margin or fracture of the restoration, and by the acceleration of the degradation process induced by the lack of polymerization (Daugherty *et al.*, 2018; Boaro *et al.*, 2019). Lack of polymerization promotes a gradual reduction in microhardness at different depths, and authors have suggested that this was due to a reduction in the degree of conversion of resin composites with the increase in the distance from the irradiated surface (Yu *et al.*, 2017; Erhardt *et al.*, 2020; Diab *et al.*, 2021).

Fourier Transform Infrared Spectroscopy (FTIR) continues to be the technique most frequently used for evaluating the degree of conversion, although there are different measuring techniques, including Electron Paramagnetic Resonance (EPR), nuclear magnetic resonance (NMR), differential scanning calorimetry (DSC) and Differential thermal analysis (DTA) (Habib & Waly, 2018; Labrie *et al.*, 2022).

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Changes are also frequently made in the light sources, such as LED (light-emitting diode) that have characteristics such as wavelength, polymerizing temperature, and power density, which allow more effective polymerization of the resins (Lempel *et al.*, 2019; Maucoski *et al.*, 2023). The LED sources may be divided into the single-peak blue LED emitting units, producing a very narrow band of wavelengths centralized between 450 and 470 nm; and the polywave (blue /violet) light-polymerization units. These use a combination of LED chips with different wavelengths of emission to produce a spectral output that covers the band of 440 and 470 nm, and the shorter wavelengths of below 420 nm (Shimokawa *et al.*, 2017; Lempel *et al.*, 2021; Al-Zain *et al.*, 2022).

In view of the technical evolution of light sources and the development of materials with new chemical formulations, there is a need to evaluate the correlation between the light source and the material, in order to verify the effect of these light sources on material properties and how they can influence the longevity of restorations.

An important analysis is to evaluate the degree of conversion of these resins considering the depth of the restorative increment. Another important factor is to evaluate the effect of different wavelengths of light on these different depths.

Therefore, this study aimed to evaluate the Vickers microhardness in the middle and deep thirds of the samples in the time intervals of 48 hours, 7 days, 14 days, and 21 days. An additional aim was to compare the conversion of the monomers through the different thicknesses of three Bulk Fill resins, using polymerization by single peak and polywave LED light units. The null hypotheses tested were as follows: 1) the hardness in the middle and inferior thirds of the samples would be the same after the use of the different light sources; 2) There would be no differences among the light sources tested.

### MATERIAL AND METHOD

**Experimental Design.** For evaluating the microhardness in depth, the factors were:

· Restorative Material (3 levels): M1 - Resin composite of the type of Bulk Fill- FiltekTM Bulk Fill (3M); M2 -Resin composite of the type of Bulk Fill- Tetric N-Ceram Bulk Fill (Ivoclar Vivadent); M3 - Resin composite of the type of Bulk Fill- Opus Bulk Fill (FGM)

·Light Source (3 levels): F1 - Optilight Max (Gnatus); F2 - Bluephase (Ivoclar Vivadent); F3 - VALO (Ultradent)

•Time (4 levels): T0 - 48 hours; T1 - 7 days; T2 - 14 days; T3 - 21 days)

And for the degree of conversion, the factors of variation were:

•Restorative Material (3 Levels): M1 - Resin composite of the type of Bulk Fill- FiltekTM Bulk Fill (3M); M2 -Resin composite of the type of Bulk Fill- Tetric N-Ceram Bulk Fill (Ivoclar Vivadent); M3 - Resin composite of the type of Bulk Fill- Opus Bulk Fill (FGM)

· Light Source (3 levels): F1 - Optilight Max (Gnatus); F2 - Bluephase (Ivoclar Vivavent); F3 - VALO (Ultradent)

·Factor Depth (3 levels): DS - 2 mm; DM - 4 mm; DD - 6 mm)

Fabrication of the specimens. For fabricating the specimens for testing deep microhardness, a 2-piece Teflon matrix was used: the first contained a cavity measuring 6 mm deep, 2 mm thick and 4 mm wide with a rounded base and the second contained a smooth. flat surface. The materials were inserted into the device that contained the cavity, and the device with a smooth surface together with a polyester strip were placed on top of this for better uniformity of sample surfaces. The materials were light polymerized with the different light sources, removed from the matrices, and individually stored in relative humidity conditions with artificial saliva, in an oven at  $37 \pm 1^{\circ}$ C throughout the entire period of the experiment. Polishing was performed with Sof Lex (3M) discs of lesser grain to produce a smoother and more uniform finish (Arnez et al., 2021).

For fabricating the specimens for testing the degree of conversion, a Teflon matrix measuring 6 mm in diameter and 2 mm thick. Three matrices, which were placed one on top of the other, were used. To obtain the correct position of the matrices a silicone device was made. A polyester strip was placed between the matrices, in order to obtain the three depths (S - Superficial, M - Medium and D - Deep), and thus it was possible to obtain the different thicknesses. The materials were light polymerized with the different light sources.

**Deep Microhardness Readouts.** The deep microhardness readouts were performed in the experimental time intervals of 48 hours, 7 days, 14 days and 21 days. For this analysis, the microdurometer (HMV-2000 Shimadzu Corporation, Japan) was used, with a pyramid-shaped diamond coated penetrator of the Vickers type, with a load of 100 g, applied for 10 seconds. In each test specimen, 2 impressions were made in each depth (Arnez *et al.*, 2021).

**Degree of Conversion Readouts.** The degree of conversion was measured by using Fourier Transform Infrared Spectroscopy (FTIR) (FTIR Spectrophotometer IRPrestige-21, Shimadzu, Kyoto, Japan). The resolution of 4 cm-1 was used. The degree of conversion was analyzed by means of two readouts of the spectrum of the unpolymerized sample, and afterwards of the polymerization in the different thicknesses proposed (S, M and D). The calculation of the degree of conversion was made by means of the following formula: % = 100[1 - /] (Labrie *et al.*, 2022).

where:

a = intensity of the band 1634 cm-1 generated by the bonds =C-H of the polymer;

b = intensity of the band 1634 cm-1 generated by the bonds =C-H of the monomer.

**Statistical analysis.** The data obtained were submitted to the Shapiro-Wilk normality test and demonstrated that the sample distribution was normal (p>0.05), so the ANOVA test was applied.

## RESULTS

For the variable degree of conversion, the following factors were found to be statistically significant: Source (p=0.006) and the factor Depth (p=0.011) in isolation and the interaction Material x Source x Time. The factor Depth was significant

(p=0.011), which meant that the depth, represented by the different thicknesses, had a significant effect on the degree of conversion. DS was found to show higher mean values than DP (p=0.012), DM showed intermediate values, without statistical difference in comparison with DS (p=0.787) and DP (p=0.140). For the factor source, F2 was found to show lower mean vales than F1 (p=0,043) and F3 (p=0.007) which may be verified in Table I.

For the interaction Material x Source x Time, when comparing the materials for use of the same source and same third: for L1 in DS and for DM, the M1, M2 and M3 were statistically equal; for L1 in DD: M1 showed a higher mean value than M2 (p=0.017) and M3 (p=0.037), while M2 and M3 were statistically equal (p=1.000). For L2 and L3 in the thirds TS, TM and TP, the M1, M2 and M3 were statistically equal.

When comparing the sources for the same material and same third: For M1, in the thirds DS and DM, source F2 showed lower mean values than F3 and F1 showed intermediate values. For DD, source F1 showed lower mean values than F3 (p=0.010) and F2 showed intermediate values, without statistical difference in comparison with L1 (p=0.300) and L3 (p=0.471). For M2 and M3 in all the thirds, there was no significant difference among the sources (Table II).

In the analysis of microhardness in medium and deep depths, statistical significance was found for the factors Time and Material, when analyzed in isolation, and for the interactions Material x Time, Source x Time and Material x Source.

The factor time had a statistically significant effect on the variable microhardness, and for both the middle third and deep third, the time of 14 days was found to show statistically higher mean values than the other time intervals. For the factor Material, material M1 showed statistically higher mean values than M2 and M3 (Table III).

Table I. Means of degree of conversion of the factor depth (DS, DM and DP) and of the factor Source (L1, L2 and L3).

2 mm (DS)	4 mm (D <sub>M</sub> )	6mm (D <sub>P</sub> )	L <sub>1</sub>	L <sub>2</sub>	L <sub>3</sub>
57.29 ± 19.61	54.15 ± 18.11	49.44 ± 19.40	56.95 ± 17.70	43.42 ± 19.57	60.5 ± 15.22

Table II. Means of degree of conversion for the Interaction Material x Source x Depth.

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	To	$T_1$	$T_2$	$T_3$	M <sub>1</sub>	$M_2$	M <sub>3</sub>
4 mm (D <sub>M</sub> )	43.34 ± 5.46	43.24 ± 5.55	45.88 ± 6.00	43.64 ± 5.73	46.44 ± 5.84	43.03 ± 4.90	42.59 ± 5.78
6 mm (D <sub>P</sub> )	43.37 ± 4.54	43.17 ± 5.39	45.88 ± 5.23	43.66 ± 5.64	46.45 ± 5.12	42.96 ± 5.41	42.65 ± 5.08

	2 mm (D <sub>s</sub> )			4 mm (D <sub>M</sub> )			6 mm (D <sub>P</sub> )		
	F <sub>1</sub>	F <sub>2</sub>	F <sub>3</sub>	F <sub>1</sub>	F <sub>2</sub>	F <sub>3</sub>	F <sub>1</sub>	F <sub>2</sub>	F <sub>3</sub>
м.	64.88 ±	38.14 ±	69.39 ±	52.3 ±	37.58 ±	65.77 ±	26.52 ±	45.10 ±	60.99 ±
111	14.49	11.33	17.12	8.85	16.37	8.78	8.95	14.73	16.73
M <sub>2</sub>	60.42 ±	44.50 ±	69.23 ±	69.85 ±	44.47 ±	59.49 ±	58.95 ±	45.69 ±	61.24 ±
	20.99	17.97	10.52	25.58	22.86	13.37	7.89	24.27	12.67
M <sub>3</sub>	67.84 ±	48.17 ±	53.07 ±	56.26 ±	50.29 ±	51.34 ±	55.52 ±	36.92 ±	53.99 ±
	17.40	23.94	21.77	14.96	20.78	12.81	14.32	27.88	18.70

Table III. Means of microhardness in medium and deep depths of factor Time (T0, T1, T2 and T3) and of factor Material (M1, M2 and M3).

In the direction of the lines of the interaction of the factors Material x Source, it was verified that for M1, Source F3 showed lower values than L2. For the materials M2 and M3, respectively, there was no difference among the sources. In the direction of the columns, for both Source L1 and L2 no differences were verified among the materials; whereas for Source L3, M1 showed higher microhardness values than M2 and M3 (p=0.001); and M2 and M3 showed equal mean microhardness values (p=1.000) (Table IV).

Table IV. Means of microhardness in medium and deep depths of Interaction Material x Source.

		D <sub>M</sub>			DP	
	<b>M</b> 1	M <sub>2</sub>	M <sub>3</sub>	<b>M</b> 1	M <sub>2</sub>	M <sub>3</sub>
L <sub>1</sub>	46.63 ± 5.59	43.94 ± 4.76	42.99 ± 4.89	46.63 ± 5.94	44.05 ± 5.20	43.54 ± 5.41
$L_2$	44.42 ± 6.06	42.29 ± 5.14	44.05 ± 3.45	44.42 ± 6.06	42.53 ± 5.50	43.69 ± 3.68
L <sub>3</sub>	48.31 ± 4.28	43.09 ± 3.93	40.73 ± 6.33	48.30 ± 3.36	42.31 ± 5.53	40.73 ± 6.33

### DISCUSSION

Microhardness evaluations in resin composites are generally used to indirectly test and confirm the effect of degree of conversion of polymer networks (Fronza et al., 2015; Pirmoradian et al., 2020). The gradual reduction in microhardness at different depths suggested that there was reduction in the degree of conversion of resin composites with the increase in the distance from the irradiated surface (Habib & Waly, 2018; Hayashi et al., 2020; Hordones Ribeiro et al., 2023). This concept is of the utmost importance for understanding the longevity of restorations with inadequate polymerization. However, it may be misguided to use the microhardness measurements alone as an indirect method for evaluating the degree of conversion, because they could lead to overestimating the depth of cure (Flury et al., 2014; Fronza et al., 2015; Tarle et al., 2015).

Nevertheless, in the present study, the chemical compositions associated with the variations in light sources were observed to have a great influence on the degree of conversion and microhardness of the studied resins.

Microhardness analysis was performed of the resins of the Bulk Fill type in different thicknesses (2

mm, 4 mm, and 6 mm) to evaluate the influence of increment thickness on microhardness. The results of 4 mm and 6 mm thickness were evaluated, which corresponded to DM and DD, respectively. Therefore, the first null hypothesis was rejected, because it was possible to observe that for both DM and DD, higher mean values were shown for the microhardness in the time of 14 days, than in the other time intervals. This suggested that when thicker increments/layers of the Bulk Fill type resins were polymerized, they could show complete polymerization after 14 days, due to the lower incidence of light at these depths, which could affect the microhardness. The Bulk Fill resin showed higher microhardness values than the other resins, when the depths of DM and DD were compared. These results are contrary to those of the study of Fronza et al. (2015) in which the mean microhardness values showed similar values in depths.

For the microhardness of DM and DP the interaction Source x Time was statistically significant. In the time interval of 48 hours (T0), Source L1 (Optilight Max) was found to show higher microhardness values than Source L2 (Bluephase), which may be related to the polywave style of Source Bluephase (L2) in which its spectral irradiance may not have been uniformly

distributed by the light tip (Sampaio *et al.*, 2017), which may have affected the microhardness produced when compared with the style of the single peak (monowave) source.

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The interaction Material x Source was statistically significant. The mean microhardness values of the Source VALO (F3) showed higher microhardness for the material Filtek Bulk Fill (M1) than for the other resins. The Filtek Bulk Fill (M2) resin showed higher microhardness values than the other resins both at the depths of 4 mm (DM) and 6 mm (DD). This could be related to the lower level of translucence of this resin, due to the addition of small fillers of zirconia particles which could influence the better transmission of light for this material, and thereby increase its microhardness (Guo et al., 2012; Shimokawa et al., 2017). Moreover, the VALO is a LED light source (violet/blue) with a broad spectrum, which uses a combination of two or more different colored LED chips to provide light in the band from 440 nm to 470 nm and wavelengths below 420 nm (Lempel et al., 2021; Maucoski et al., 2023). These broad-spectrum LED units have been reported to polymerize samples of some resins to a greater extent than the single band blue light emitting diode (LED) light curing unit (LCU), which may perhaps also justify the better microhardness of this material polymerized with this type of LED light (Price et al., 2010).

When there is reduced penetration of light and increased distance from the irradiated surface, a reduction in the degree of conversion would be expected, therefore, a reduction in the hardness values, which could also influence the lower microhardness values produced by the other light sources (Al-Zain *et al.*, 2022).

The second null hypothesis tested was rejected, because when the degree of conversion data was analyzed, the factors depth and source showed significance. As for the factor depth, a higher degree of conversion was verified at 2 mm (DS); and at 6 mm (DD) a lower degree of conversion was shown. In the present study none of the thirds showed a degree of conversion of 60 %, a finding that disagreed with Gonçalves *et al.* (2018), a study in which three (Venus Bulk Fill Flow, Filtek Bulk Fill and Filtek Bulk Fill Flow) of the six Bulk Fill resins evaluated were capable of maintaining statistically similar degree of conversion values at 4 mm; and four (Venus Bulk Fill Flow, Filtek Bulk Fill, Filtek Bulk Fill Flow and everX Posterior) were capable of maintaining up to 80 % of their degree of conversion in the inferior part, when compared with the superior part.

Yu *et al.* (2017) evaluated the degree of conversion of different Bulk Fill resins (Beautifil Bulk, Beautifil Bulk Flowable, Tetric N-Ceram Bulk Fill, Smart Dentin Replacement) at the depths of 2 mm, 4 mm and 6 mm, and the degree of conversion values were similar to those of the present study, in which the Bulk Fill resins evaluated showed degree of conversion values of below 60 %. According to the author, this was explained by the power of the light source and composition of the material, which could justify the results found in the present study.

Filtek Bulk contains Urethane Dimethacrylate (UDMA), and the copolymerization of Bisphenol Adiglycidyl dimethacrylate (bis-GMA) with UDMA or Triethylene glycol dimethacrylate (TEGDMA) which increases the conversion and creates a highly reticulated, dense, rigid polymer network, thus increases its microhardness (Gonçalves *et al.*, 2018).

In the present study, the factor light source was statistically significant, in which Source F2 showed lower mean values than those of F1 and F3. According to the literature, radiant exposure (irradiance x duration of exposure) in light polymerization may influence the degree of conversion (Lempel *et al.*, 2019) and the efficacy of light polymerization sources may be evaluated by the degree of conversion by various methods, such as Fourier Transform Infrared spectroscopy (FTIR) and microhardness tests (Algamaiah *et al.*, 2021; Al-Zain *et al.*, 2022).

Bluephase G2 and VALO are examples of polywave LED (blue / violet) that have various emission bands. However, the spectral output of the light polymerization unit must correspond to the absorption spectra of the specific photoinitiator used for each resin (Price et al., 2010), which may justify the Bluephase Light source being shown to produce a lower degree of conversion in the present study. Furthermore, the distribution of irradiation measured in the emission wavelengths of 405 and 460 nm may not have been uniform at the emission extremity of all the polygonal LED / blue / violet units (G-Light, Bluephase G2 and VALO). This may also have had an influence on the lower degree of conversion values produced by the Bluephase light source (F2). This lack of spatial and spectral homogeneity in the emission of light is a source of concern because some resins use photoinitiators that demand shorter wavelengths of light, which have an influence on the degree of conversion (Price *et al.*, 2010; Al-Zain *et al.*, 2022).

This suggested the need to include the smaller short wavelengths in the LED curing lights used for curing Bulk Fill resins, and agrees with Sampaio *et al.* (2017), in stating that the quantity of violet light was insufficient for curing Bulk Fill resins at deeper depths. In general, the light polymerization was the worst in the deeper regions mainly exposed to violet light, and this was translated into lower degree of conversion values in this region. Other studies have also discovered that the increase in thickness had a more negative effect on the transmission of violet light (350-425 nm) than on that of blue light (425-550 nm) (Harlow *et al.*, 2016; Shimokawa *et al.*, 2017), and consequently, could also affect the microhardness of Bulk Fill type resins.

Bulk Fill resins have different photoinitiators, such as those of the present study. Price *et al.* (2010) conducted a study indicating which wavelengths were the most efficient for curing photoinitiators (Lucirin TPO and Camphorquinone). They observed that at the wavelength of 460 nm, Camphorquinone was more efficiently cured; and at the wavelength of 405 nm. Alternative photoinitiators, for example Lucirin TPO, were more efficiently activated. This may justify the fact that Bluephase showed lower degree of conversion values when compared with the other light sources, which may not have efficiently cured the photoinitiators in the resins of the present study.

The interaction Material x Source x Time was statistically significant. When comparing the materials for the same Source and for the same third, for L1 in depth (DD), the material M1 showed lower degree of conversion values than M2 and M3, which were statistically equal. For the other sources at the different depths, all the materials were statistically equal. In the comparison of three same sources of the same material and the same depth, it was verified that for the material M1 in the depths DS, DM and DD the Source F3 showed higher degree of conversion values than those of L1 and L2; and for comparison of the depths for the same material and same Source, Material M1 for Source L1 the DD showed lower degree of conversion values than D and DM, which were statistically equal.

In the present study, the material Filtek Bulk Fill showed a lower degree of conversion; and the literature

is controversial relative to its degree of conversion (Gonçalves et al., 2018; Lempel et al., 2019). In a study conducted by Gonçalves et al. (2018), Filtek Bulk Fill resin showed degree of conversion values of over 60 % in all the depths evaluated (1 mm, 2 mm, 3 mm, and 4 mm). According to Pirmoradian et al. (2020) they affirmed that the initial viscosity of the monomer and flexibility of is chemical structure had an influence on the degree of conversion. The monomer UDMA is viscous due to the intramolecular interaction of the hydrogen bond between its imino (NH) and carbonyl (CO) groups and has a lower viscosity than that of Bis-GMA, due to the weak hydrogen bond of its imino (NH) group in comparison with its hydroxyl groups (OH) (Alshali et al., 2013; Durner et al., 2022). Moreover, the presence of imino groups (NH) in the urethane structure of the monomer UDMA is responsible for the chain transfer reactions that provide an alternative pathway for the continuation of polymerization. This results in an increase in mobility of the radical sites on the network, and consequently, in the increase in polymerization and conversion of monomers (Alshali et al., 2013; Lempel et al., 2019).

Filtek Bulk Fill has a smaller filler content and greater parameter of translucence, and despite these advantageous parameters, Filtek Bulk Fill resin (M2) had the lowest degree of conversion when compared with the other Bulk Fill resins. This agreed with Lempel et al. (2019) who, in their study, found that the same resin showed the lowest degree of conversion. According to previous studies, the degree of conversion values for Filtek Bulk Fill were lower in comparison with those of other Bulk Fill resins. Alshali et al. (2013) considering the chemical composition of Filtek Bulk Fill resin, which contains Procrylate resin, the BisGMA was combined with UDMA and BisEMA, instead of with TEGDMA. Although UDMA has a much lower viscosity than that of BisGMA, when it is mixed with high molecular weight BisEMA, this may significantly restrict the mobility of the UDMA monomers, and thereby reduce its reactivity and degree of conversion value (Lempel et al., 2019).

Since this concerns an in vitro analysis, there is a need for clinical experiments to validate the methods used. The results of this study showed that it is imperative to conduct studies on the behavior of restorative materials when using different polymerization methods, because the compositions and chemical reactions are influenced by the light source, and this may lead to an unsatisfactory result of a restoration.

### CONCLUSION

Considering that the Filtek Bulk Fill resin had the highest microhardness values in the time interval of 14 days with use of the light Source VALO; and although its lowest degree of conversion value in the deep third occurred with the use of the Optilight Max light Source, it was concluded that with the methodology applied, the Valo unit showed the best results in comparison with the Tetric N-Ceram Bulk Fill e Opus Bulk Fill resins.

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RESUMEN: El presente estudio analizó la microdureza y el grado de conversión de tres resinas Bulk Fill (M1 - Filtek Bulk Fill; M2 - Tetric N-Ceram Bulk Fill y M3 - Opus Bulk Fill) polimerizadas por lámparas de curado de diodo emisor de luz de pico único y polionda. Se obtuvieron un total de 90 especímenes de prueba (n=10) utilizando una matriz de teflón con el propósito de probar la microdureza; y para grado de conversión: 135 especímenes (n=5) utilizando una matriz de 2 x 6 cm. Las muestras se fotopolimerizaron utilizando 3 fuentes de luz (L1 - Optilight Max, L2 - Bluephase, L3 - VALO). Se mantuvieron en saliva artificial en estufa a 37±1°C durante el experimento. El grado de conversión se midió por FTIR 24 h después de obtener cada muestra de prueba. Las lecturas de microdureza se realizaron con un microdurómetro en los intervalos de tiempo de 48 horas (T0), 7 días (T1), 14 días (T2) y 21 días (T3). Se encontró que M1L3 mostraba los valores más altos de microdureza en T2, y M1 mostraba el grado más bajo de conversión en el tercio profundo con L1. Se concluyó que la resina Filtek Bulk Fill mostró los mejores resultados en comparación con las demás resinas.

PALABRAS CLAVE: resinas compuestas, material dental, dureza, grado de conversión, LED.

### REFERENCES

- Al-Senan, D. & Al-Nahedh, H. The effect of different light curing units and tip distances on translucency parameters of bulk fill materials. *Saudi Dent. J.,* 34(5):362-8, 2022.
- Al-Zain, A. O.; Alboloshi, E. A.; Amir, W. A.; Alghilan, M. A. & Münchow, E. A. Effects of aging and light-curing unit type on the volume and internal porosity of bulk-fill resin composite restoration. *Saudi Dent. J.*, 34(3):243-8, 2022.
- Algamaiah, H.; Silikas, N. & Watts, D. C. Polymerization shrinkage and shrinkage stress development in ultra-rapid photopolymerized bulk fill resin composites. *Dent. Mater.*, 37(4):559-67, 2021.

- Alshali, R. Z.; Silikas, N. & Satterthwaite, J. D. Degree of conversion of bulk-fill compared to conventional resin-composites at two time intervals. *Dent. Mater.*, 29(9):e213-7, 2013.
- Arnez, M. M.; Castelo, R.; Ugarte, D.; Almeida, L. P. A.; Dotta, T. C. & Catirse, A. B. C. E. B. Microhardness and surface roughness of Biodentine exposed to mouthwashes. J. Conserv. Dent., 24(4):379-83, 2021
- Besegato, J. F.; Jussiani, E. I.; Andrello, A. C.; Fernandes, R. V.; Salomão, F. M.; Vicentin, B. L. S.; Dezan-Garbelini, C. C. & Hoeppner, M. G. Effect of light-curing protocols on the mechanical behavior of bulk-fill resin composites. *J. Mech. Behav. Biomed. Mater.*, 90:381-7, 2019.
- Boaro, L. C. C.; Pereira Lopes, D.; de Souza, A. S. C.; Lie Nakano, E.; Ayala Perez, M. D.; Pfeifer, C. S. & Gonçalves, F. Clinical performance and chemical-physical properties of bulk fill composites resin -a systematic review and meta-analysis. *Dent. Mater.*, 35(10):249-64, 2019.
- Daugherty, M. M.; Lien, W.; Mansell, M. R.; Risk, D. L.; Savett, D. A. & Vandewalle, K. S. Effect of high-intensity curing lights on the polymerization of bulk-fill composites. *Dent. Mater.*, 34(10):1531-41, 2018.
- de Araújo-Neto, V. G.; Sebold, M.; Fernandes de Castro, E.; Feitosa, V. P. & Giannini, M. Evaluation of physico-mechanical properties and filler particles characterization of conventional, bulk-fill, and bioactive resin-based composites. J. Mech. Behav. Biomed. Mater., 115:104288, 2021.
- Diab, R. A.; Yap, A. U.; Gonzalez, M. A. G. & Yahya, N. A. Impact of light-curing distance on the effectiveness of cure of bulk-fill resinbased composites. *Saudi Dent. J.*, 33(8):1184-9, 2021.
- Durner, J.; Schrickel, K.; Watts, D. C.; Becker, M. & Draenert, M. E. Direct and indirect eluates from bulk fill resin-based-composites. *Dent. Mater.*, 38(3):489-507, 2022.
- Erhardt, M. C. G.; Goulart, M.; Jacques, R. C.; Rodrigues, J. A. & Pfeifer, C. S. Effect of different composite modulation protocols on the conversion and polymerization stress profile of bulk-filled resin restorations. *Dent. Mater.*, 36(7):829-37, 2020.
- Flury, S.; Peutzfeldt, A. & Lussi, A. Influence of increment thickness on microhardness and dentin bond strength of bulk fill resin composites. *Dent. Mater.*, 30(10):1104-12, 2014.
- Fronza, B. M.; Rueggeberg, F. A.; Braga, R. R.; Mogilevych, B.; Soares, L. E. S.; Martin, A. A.; Ambrosano, G. & Giannini, M. Monomer conversion, microhardness, internal marginal adaptation, and shrinkage stress of bulk-fill resin composites. *Dent. Mater.*, *31(12)*:1542-51, 2015.
- Gonçalves, F.; de Paiva Campos, L. M.; Rodrigues-Júnior, E. C.; Costa, F. V.; Marques, P. A.; Francci, C. E.; Braga, R. R. & Boaro, L. C. C. A comparative study of bulk-fill composites: degree of conversion, post-gel shrinkage and cytotoxicity. *Braz. Oral Res.*, 32:e17, 2018.
- Guo, G.; Fan, Y.; Zhang, J. F.; Hagan, J. L. & Xu, X. Novel dental composites reinforced with zirconia-silica ceramic nanofibers. *Dent. Mater.*, 28(4):360-8, 2012.
- Habib, A. N. A. & Waly, G. H. The degree of conversion and class II cavity microleakage of different bulk fill composites placed with different restorative techniques. *Future Dent. J.*, 4(2):231-8, 2018.
- Harlow, J. E.; Rueggeberg, F. A.; Labrie, D.; Sullivan, B. & Price, R. B. Transmission of violet and blue light through conventional (layered) and bulk cured resin-based composites. *J. Dent.*, 53:44-50, 2016.
- Hayashi, J.; Tagami, J.; Chan, D. & Sadr, A. New bulk-fill composite system with high irradiance light polymerization: Integrity and degree of conversion. *Dent. Mater.*, *36*(*12*):1615-23, 2020.
- Hordones Ribeiro, M. T.; Felipe de Bragança, G.; Sales Oliveira, L. R.; Lourenço Braga, S. S.; Quirino de Oliveira, H. L.; Price, R. B. & Soares, C. J. Effect of pre-heating methods and devices on the mechanical properties, post-gel shrinkage, and shrinkage stress of bulk-fill materials. *J. Mech. Behav. Biomed. Mater.*, 138:105605, 2023.

ARNEZ, M. M.; ALMEIDA, L. P. A.; DOTTA, T. C.; CASTELO, R.; UGARTE, D. E.; RAMOS, A. P.; REIS, A. C.; & CATIRSE, A. B. C. E. B. Effects of LED lights on bulk fill resin polymerization. Int. J. Odontostomat., 17(4):438-445, 2023.

- Labrie, D.; Price, R. B.; Sullivan, B.; Salazar, A. M.; Gautam, D.; Stansbury, J. W. & Ferracane, J. L. Effect of thickness on the degree of conversion of two bulk-fill and one conventional posterior resin-based composites at high irradiance and high temporal resolution. *J. Mech. Behav. Biomed. Mater.*, 136:105489, 2022.
- Lempel, E.; O'ri, Z.; Kincses, D.; Lovász, B. V.; Kunsági-Máté, S. & Szalma, J. Degree of conversion and in vitro temperature rise of pulp chamber during polymerization of flowable and sculptable conventional, bulk-fill and short-fibre reinforced resin composites. *Dent. Mater.*, 37(6):983-97, 2021.
- Lempel, E.; O"ri, Z.; Szalma, J.; Lovász, B. V.; Kiss, A.; Tóth, Á. & Kunsági-Máté, S. Effect of exposure time and pre-heating on the conversion degree of conventional, bulk-fill, fiber reinforced and polyacid-modified resin composites. *Dent. Mater.*, 35(2):217-28, 2019.
- Lempel, E.; Szebeni, D.; O'ri, Z.; Kiss, T.; Szalma, J.; Lovász, B. V.; Kunsági-Máté, S. & Böddi, K. The effect of high-irradiance rapid polymerization on degree of conversion, monomer elution, polymerization shrinkage and porosity of bulk-fill resin composites. *Dent. Mater.*, 39(4):442-53, 2023.
- Maucoski, C.; Price, R. B.; Rocha, M. G.; Roulet, J. F. & Sullivan, B. Ability of short exposures from laser and quad-wave curing lights to photo-cure bulk-fill resin-based composites. *Dent. Mater.*, 39(3):275-92, 2023.
- Pirmoradian, M.; Hooshmand, T.; Jafari-Semnani, S. & Fadavi, F. Degree of conversion and microhardness of bulk-fill dental composites polymerized by LED and QTH light curing units. J. Oral Biosci., 62(1):107-13, 2020.
- Price, R. B. T.; Labrie, D.; Rueggeberg, F. A. & Felix, C. M. Irradiance differences in the violet (405 nm) and blue (460 nm) spectral ranges among dental light-curing units. *J. Esthet. Restor. Dent.*, 22(6):363-77, 2010.
- Sampaio, C. S.; Atria, P. J.; Rueggeberg, F. A.; Yamaguchi, S.; Giannini, M.; Coelho, P. G.; Hirata, R. & Puppin-Rontani, R. M. Effect of blue and violet light on polymerization shrinkage vectors of a CQ/TPO-containing composite. *Dent. Mater.*, 33(7):796-804, 2017.
- Shimokawa, C. A. K.; Sullivan, B.; Turbino, M. L.; Soares, C. J. & Price, R. B. Influence of emission spectrum and irradiance on light curing of resin-based composites. *Oper. Dent.*, 42(5):537-47, 2017.
- Tarle, Z.; Attin, T.; Marovic, D.; Andermatt, L.; Ristic, M. & Tauböck, T. T. Influence of irradiation time on subsurface degree of conversion and microhardness of high-viscosity bulk-fill resin composites. *Clin. Oral Investig.*, 19(4):831-40, 2015.
- Yu, P.; Yap, A. U. J. & Wang, X. Y. Degree of conversion and polymerization shrinkage of bulk-fill resin-based composites. *Oper. Dent.*, 42(1):82-9, 2017.

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