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Degree of Conversion in Bulk Fill and Conventional Composites Cured with Different Dental Curing Light Sources: An in vitro Study

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Abstract

Objective: This in vitro study aimed to compare the degree of conversion (DC) of bulk-fill and conventional composite resins cured by Quartz Tungsten Halogen (QTH) and poly wave Light-emitting diode (LED) curing units.

Materials and Methods: Thirty-two cylindrical specimens were fabricated in eight groups (n=4): (I) X-tra fil packable cured with the poly wave, (II) X-tra fil packable cured with QTH, (III) X-tra base cured with a poly wave, (IV) X-tra base flow cured with QTH, (V) Grandio packable cured with a poly wave, (VI) Grandio packable cured with QTH, (VII) Grandio flow cured with a poly wave, and (VIII) Grandio flow cured with QTH curing unit. Light-curing was performed with LED or QTH. Fourier-transform infrared spectroscopy (FTIR) was performed to assess the DC of the samples. Data were analyzed using one-way ANOVA and a post-hoc Tukey test.

Results: XtraBase flow bulk-fill composite/poly-wave was the only composite with no significant difference between the DC of its first and fourth layers. The highest depth of cure was noted in the first layer of Grandio flow/QTH, X-tra base/QTH, and X-tra base/poly-wave, and the fourth layer of Grandio flow/QTH, and Grandio flow/poly wave.

Conclusion: The degree of conversion decreased by an increase in depth. The poly-wave curing unit was more efficient for bulk-fill composites. QTH curing unit was more efficient for the conventional composites demonstrating a greater depth of cure. Composites with higher flow showed a higher depth of cure due to higher translucency.

Keywords: bulk-fill, composite resins, light curing, degree of conversion.

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Introduction Objective

Resin composite restorations have become an essential part of operative dentistry due to new adhesive technology, advances in the polymerization system, increasing patients' esthetic demands, and the importance of preserving more tooth structure (Kwon, Ferracane et al. 2012, Yancey, Lien et al. 2019). According to meta-analysis research conducted by Heintze and others (Heintze, Rousson et al. 2015), among 500 million direct restorations which are placed every year, almost 55% were composites or components. They are repairable and have improved esthetics. However, their application technique is challenging and time-consuming. Another disadvantage of photo-polymerized composites is their limited depth of cure (approximately 2 mm) which results in insufficient polymerization, especially in deep restorations (Yap, Pandya et al. 2016). Insufficient polymerization reduces the physical, mechanical, and biological properties and is one of the main causes of composite restorations failure, leading to secondary caries and postoperative sensitivity(Yoon, Lee et al. 2002, Savadamoorthi, Priyadharshini et al. 2017). To overcome this limitation, an incremental application technique is suggested, which also has complications including difficulty in the application of increments in a small cavity, debonding between the layers, void formation and contamination between the layers, and longer working time (Alrahlah, Silikas et al. 2014, Yap, Pandya et al. 2016).

The degree of polymerization is affected

by several factors such as the composite increment thickness, shade and translucency of composite, shape, and size of fillers, the intensity of curing light, duration of light-curing, and distance between the tip of the curing device and composite; In an ideal situation, the entire monomer content of composite should be converted to the polymer during the polymerization process (Alrahlah, Silikas et al. 2014).

With the onset of bulk-fill composites, the incremental technique could be replaced by the bulk incremental technique. Bulk-fill composites were introduced to the market due to their advantages and easy application. The advances made in the photo-initiator dynamics and the increased translucency, increase the depth of cure in bulk fill composites (approximately 4-5 mm). Thus, the clinicians can apply thicker composite increments, which decreases the chair time (Erhardt, Goulart et al. 2020). Bulk-fill composites are available in two types flowable and packable (Li, Pongprueksa et al. 2015).

bulk-fill composites have lower polymerization stress, and consequently lower polymerization shrinkage, which can prevent post-treatment hypersensitivity, secondary caries, deboning, marginal leakage, and debonding at the adhesivetooth interface (Ciucchi, Bouillaguet et al. 1997, Dickens, Stansbury et al. 2003).

The quartz-tungsten-halogen (QTH) curing unit is among the most commonly used light sources in dentistry to polymerize composites (Ritter 2017; Afnan L, et. al., 2022). It has a wavelength range of 390 to 520 nm. At present, light-emitting diode (LED) or mono-wave LED curing units are more commonly used in the clinical setting. These devices have a narrower wavelength range of 400-500 nm (Al Shaafi, Maawadh et al. 2011, Ilie, Keßler et al. 2013). The third-generation LED devices (poly-wave) are the most recent devices with a wavelength range of 380-515 nm. In poly-wave devices, in addition to the blue light used in the second generation of LED curing units, there is also a purple light, which There are different types of photoinitiators in bulk-fill composites to increase the depth of cure such as camphorquinone, phenylpropanedione, and Ivocerin, each with a different wavelength absorption (Menees, Lin et al. 2015). Camphorquinone generates free radicals by absorbing light in the range of 400 to 500 nm (Samir, Abdel-Fattah et al. 2020). The wavelength of mono wave LED devices is suitable for activation of camphorquinone the (Pirmoradian, Hooshmand et al. 2020) however it may not be suitable for curing composites that have other photoinitiators. QTH and third-generation LED (poly-wave) light curing units appear to be more suitable for the curing of bulk-fill composites, due to their higher wavelength range than the second-generation LED curing units (mono wave) (Lindberg, Peutzfeldt et al. 2005, Al Shaafi, Maawadh et al. 2011).

Fourier-transform infrared spectroscopy (FTIR) is one of the most useful methods to detect molecules and chemical reactions. Light passing through the sample can detect chemical bonds using an infrared bench detector. By comparing the number of double-carbon aliphatic and aromatic bonds in the cured and non-cured samples, the DC of the sample is determined (Obici, Sinhoreti et al. 2004).

the purpose of the present study was to compare the degree of conversion (DC) of bulk-fill and conventional composites cured by QTH and LED poly-wave curing units on their top and bottom surfaces, Considering the existing controversy and lack of sufficient

Materials and Methods

Four types of composite materials, bulk-fill X-tra fil packable, bulk-fill X-tra base flowable incrementalfill Grandio packable, and incremental-fill grandio flowable were used in this study. The characteristics of composites including their type, shade, composition, and manufacturer are listed in Table 1. Two different dental curing light sources were also used for the curing of the samples, shown in Table 2.

Specimen preparation

Plexiglass molds with an external dimension of 80 mm, an internal dimension of 70 mm, and a thickness of 1 mm were used (figure 1). The molds were black and opaque to prevent the transmission of light from one sample to another. Specimens were fabricated in 8 groups (n = 4) (Siagian, Dennis et al. 2020). The total number of samples was 32 the samples were cylindrical with sizes 5 mm in diameter and 4 mm in thickness for bulk fill material (groups 1,2,3, and 4), and 2 mm in thickness for incremental material (groups 5,6,7, and 8).

To fabricate the samples, the first mold (5 mm in diameter and 1 mm in height) was placed on a glass slab, composite was applied into the mold by spatula, and was covered by a transparent Mylar strip. the sample was given the pressure of 1 kg load to extrude excess composite and to ensure the 1-mm height of the composite sample inside the mold. Next, the second mold was placed on the matrix strip and after repeating all the above-mentioned steps, another transparent matrix strip was placed over it. This process was repeated using two other molds as well, and in the end, 1 kg of weight was placed on the top for two minutes to ensure that the total height of the sample did not exceed 4 mm.

The material was packed in bulk inside the mold for each bulk-fill composite until it was slightly overfilled (4 mm). Each sample was irradiated according to the manufacturer's instructions for 20 seconds with 1000 mW/cm² light intensity using a LED curing unit (Blue Phase Style LED; Ivoclar) or for 20 seconds with 500 mW/cm² light intensity with a QTH curing unit (Coltolux 75; Coltene). The output power of the curing unit was verified regularly after five exposures using a light radiometer (Coltolux; Coltene).

The distance between the light source and samples was standardized by the thickness of the glass slide and the Mylar strip which resulted in smooth surfaces of samples.

The incremental-fill composite samples were fabricated with the same method except that the material was packed inside the mold in two increments (each with 1-mm thickness) and each increment was cured from the top surface for 20 seconds, according to the manufacturer's instructions.

The samples were stored in lightproof boxes after the polymerization procedure to avoid further exposure to light. Next, the four molds were separated, and molds representing depths of 1 to 4 mm were placed in an 400 E412-0798, incubator (INB Memmert GmbH, Germany) at 37°C for 24 hours.

Degree of conversion analysis

To measure the DC of composite samples, FTIR was used. The samples were analyzed by FTIR (Nicolet 10; Termo scientific company, USA) operating at 400 to 4000 cm⁻¹.

two important peaks were measured: the absorption spectrum of the samples with a peak at 1635 cm⁻¹, related to aliphatic carbon double bonds, and a peak at 1608 cm⁻¹, related to the double bonds of aromatic carbon. The DC was determined according to the following equation: $\begin{bmatrix} \frac{Aliphatic}{Aromatic} \text{ polymerized} \\ \begin{bmatrix} \frac{Aliphatic}{Aromati} & \text{non-polymerized} \end{bmatrix} \times 100$

Statistical analysis

The Data was analyzed using one-way ANOVA to compare the DC between the groups. The paired comparison was tested by a posthoc Tukey test.

Results

The mean DC of the first and fourth layers of different composites cured with QTH and poly-wave curing units is shown in Table 3. There was a significant difference between the DC of the first and fourth layers in all groups (P<0.05) except between the DC of the first and fourth layers of the bulk-fill flow/poly-wave group (P = 0.316).

Figure 2 shows the difference between the DC of the first and fourth layers of the composites.

As shown in Table 3, a comparison of the curing depth of the first layer of different composites showed that the highest curing depth was related to the first layer of conventional flow composite cured with the QTH curing unit and bulk-fill flow composite cured with the poly-wave and QTH curing units. Also, the lowest DC among the first layers of different groups belonged to the conventional packable/poly-wave group and the conventional flow/poly-wave group, with no significant difference between them (P>0.05).

As shown in Table 3, a comparison of the depth of cure of the fourth layer of different composites showed that the highest DC was related to the conventional flowable composite cured with the QTH and bulk-fill flowable composite cured with the polywave LED curing unit, which did not differ significantly (P>0.05). Also, the lowest DC in the fourth layer was related to the bulk-fill packable composite cured with the QTH curing unit.

A comparison of the DC of the first and fourth layers of different composites cured with a poly-wave curing unit is shown in Figure 3. In the assessment of composites cured with poly-wave, the highest DC 1732

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was related to the bulk-fill flowable composite in the first and fourth layers and the conventional flowable composite (especially the fourth layer). But these groups were not significantly different, and the lowest DC was related to the fourth layer of the conventional packable composite.

The DC of the first and fourth layers of different composites cured with the QTH curing unit is shown in Figure 4. The highest DC was related to the conventional flowable composite and the lowest DC was related to the bulk-fill packable composite.

Discussion

There is a growing interest in using bulkfill resin-based composite materials because of their optimal esthetics and simplified application(Ilie, Keßler et al. 2013, Gaviria-Martinez, Castro-Ramirez 2022). According et al. to the manufacturers, the main advantages of bulk-fill composite materials are increased depth of cure and faster application, probably due to higher translucency (Alonso, de Souza-Júnior et al. 2013), and low polymerization shrinkage stress due to modifications in their filler content and organic matrix.

Adequate polymerization of composite resin restorations is one of the main factors that affect their clinical success. The DC is an important parameter to assess the physical, mechanical and biological properties of composite restorations (Cekic-Nagas, Egilmez et al. 2010, Galvão, Caldas et al. 2013). Higher DC leads to superior physical and mechanical properties (Cekic-Nagas, Egilmez et al. 2010). Inadequate polymerization might cause marginal microleakage (Kusgoz, Ülker et al. 2011), discoloration, and decreased bonding strength (Aguiar, Georgetto et al. 2011)of composite restorations.

In this study, the DC of the investigated composites was assessed by FTIR because it has been used as an appropriate and reliable test as it can detect the C=C stretching vibrations directly before and after the curing of composite resins (Acquaviva, Cerutti et al. 2009, Cekic-Nagas, Egilmez et al. 2010, Sgarbi, Pereira et al. 2010).

In addition to camphorquinone, bulk-fill composites contain other photo-initiators that are activated at different wavelengths, it appears that using curing devices with a wider wave length ranges, leads to a higher DC (Rocha, Roulet et al. 2021). Therefore, poly-wave and QTH curing units, which both have a wide light spectrum, were used for the curing of the samples in this study, and their effects on the conventional and bulk-fill composites were evaluated.

In this study, the DC of conventional Composites (Grandio, Germany) was compared with X-tra base and X-tra fill bulk-fill composites after curing with QTH and poly-wave curing units. The samples were kept in a completely dark room immediately after curing until the FTIR to prevent further unwanted curing. Also, the samples were kept at 37°C to simulate the oral environment (AlAwwad DA, et. al., 2022; Lee JH., 2022).

Many studies have shown that increasing the curing time can increase the DC and also the curing depth, and this can improve the mechanical properties of composites. To simulate the clinical setting (many dentists spend only 20 seconds on curing to save time) and since bulk fill composites are mainly used to decrease the chair time, curing was performed according to the manufacturer's instructions instead of increasing the radiation time (Musanje and Darvell 2003, Leprince, Palin et al. 2013).

According to the present study, the highest DC in the first layer was related to bulk-fill composites. Zorzin et al. (Zorzin, Maier et al. 2015) observed higher DC in areas far from the light source. This result is different from the present findings and can be due to variations in temperature rise at different thicknesses during polymerization, changes in the degree of crosslinking by an increase in depth, presence of an oxygen-inhibited layer at the surface, and higher light intensity at the upper surface of the specimens (Zorzin, Maier et al. 2015). In the present study, a Mylar strip was used to prevent the formation of an oxygeninhibited layer on the surface of the samples. Therefore, a high depth of cure was observed at the surface of the samples, which can justify this difference.

The main two characteristics of a monomer that influence the DC are the initial monomer viscosity and flexibility of its chemical structure (Dickens, Stansbury et al. 2003). In the present study, x-tra fil (bulk-fill composite) showed significantly higher DC than Grandio (incremental-fill composite) in the top layer, although they both have almost the same weight percentage of the organic matrix. This might be attributed to the different chemical compositions of their organic matrix. The organic matrix of x-tra fil consists of Bis-GMA, UDMA, and TEGDMA, while that of Grandio consists of Bis-GMA and TEGDMA. It was reported that the ultimate degree of conversion of different monomer systems increases in the following order: Bis-GMA < Bis-EMA < UDMA < TEGDMA (Sideridou, Tserki et al. 2002). UDMA is a viscous monomer due to the hydrogen bond intramolecular interaction between its amine (eNHe) and carbonyl (-CO) groups. However, the viscosity of

UDMA is much lower and its flexibility is higher than that of Bis-GMA because of the weak hydrogen bond of its amine group compared with that of hydroxyl groups (Khatri, Stansbury et al. 2003). Also, the presence of amine groups in the urethane structure of UDMA monomer is responsible for the characteristic chain transfer reactions that provide an alternative path for the continuation of polymerization. These reactions result in increased mobility of radical sites of the network and consequently enhanced polymerization and monomer conversion (Sideridou, Tserki et al. 2002). This explains the increase in reactivity and higher DC of UDMA-containing organic matrix of x-tra fil when compared with the Bis-GMA-containing organic matrix of Grandio.

According to the present study, the highest DC of the bottom layer was noted in the conventional flowable composite cured with QTH and bulk-fill flowable composite cured with a poly-wave LED curing unit. The higher depth of cure in the flowable composite is due to improved translucency. A simple approach to achieve this result is by decreasing the number of fillers since translucency is linearly correlated with the number of filler particles. Besides, the translucency of dental materials is influenced by the difference in the refractive indices between the filler particles and the resin matrix (M Primus, Chu et al. 2002, Shortall, Palin et al. 2008), which determines the pattern of light scatter within a material. Similar refractive indices of the components of a resin composite, as demonstrated for Bis-GMA and silica filler particles, were shown to improve translucency in experimental dental materials (Cicalău GI., et. al., 2022; Ragimov RM., et. al., 2022). Thus, light scattering at the filler-matrix interface is reduced, allowing light to penetrate the material and result in higher in-depth polymerization. Miletic et al. (Miletic, Pongprueksa et al. 2017) suggested that composites with higher flowability show higher translucency than viscose composites. This increase in translucency increases the depth of cure, which is consistent with our findings.

In a comparison of the groups cured with a poly-wave

curing unit, the highest DC was related to the bulk-fill composites, which indicates the higher compatibility of the primers in these composites with the wide spectrum of the poly-wave curing unit. In a study conducted by Alshahi et al, (Alshali, Silikas et al. 2013) it was concluded that poly-wave LED significantly increased the curing depth of bulk-fill composites, which was in line with the present results. In the present study, the third-generation LED had an intensity of 1000 mW/cm² with an irradiation time of 20 seconds. The mean total energy for the thirdgeneration LED group was 20 J/cm². For the QTH group, the intensity was 500 mW/cm^2 , and the irradiation time was 20 seconds; thus, it had lower total energy of 10 J/cm^2 . DC is affected by the total light energy. Energy is obtained from the intensity of light multiplied by the time of exposure to light (17). The main two characteristics of a monomer that influence the DC are the initial monomer viscosity and flexibility of its chemical structure (Abed, Sabry et al. 2015). The difference in DC of bulk-fill composites cured with third-generation LED compared with QTH curing units in this study may also be due to the differences in total energy transmitted by the lightcuring process.

Conclusion

The flowable composite showed more DC because of filler size and content. The bulk-fill composite showed more DC by poly-wave light curing unit than QTH, and X-tra fil showed higher DC than Grandio conventional composite.

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Table 1. Characteristics of composites used in this study

	Manufac			
Composite	turer,	Resin		Filler(W%)
	Color,	Matrix	Filler	
Grandio	Vocco,	Bis-	Bariumeboron-	86
(packable)	Germany,	GMA,	alumino-silicate	
	A2	TEGD	glass (0.1e2.5 mm),	
		MA	Silica: 20e60 nm	
Grandio(flowabl	VoccoGe	Bis-		65.6
e)	rmany,	GMA,		
	A2	TEGD		
		MA,		
		HEDM		
		А		

inar of Barvey in Tishertes Bere		10(1) 1/2/ 1/10		201
Bulk-fill X-tra	VoccoGe	Bis-	Bariumeboronealu	87
fil	rmany,	GMA,	minoesilicate glass	
	A2	UDMA	(2e3 mm)	
		, TEG-		
		DMA		
Bulk-fill X-tra	VoccoGe	Bis-	Si glass	75
base	rmany,	GMA,		
	A2	UDMA		
		,		

Bis-GMA: bisphenol A glycidyl methacrylate

UDMA: urethane dimethacrylate

TEGDMA: tri ethylene glycol dimethacrylate.

Table 2. Dental curing light sources used in this study

Device	Brand	Manufacturer	Power density
Quartz Tungsten Halogen	Coltolux75	Coltene	500 mW/cm^2
Light-emitting diode	Bluephase style	Ivoclar Vivadent	1000 mW/cm ²

Table 3: Mean degree of conversion of bulk-fill and conventional composites cured with QTH and poly-wave

light curing units

Type of composite	Type of light cure	Layers	Mean degree of conversion	P Value		
Bulk-fill Flowable X-tra base	Poly wave	First	69.0000	0.316		
		Fourth	66.7500			
	Quartz Tungsten Halogen	First	69.7500	0.013		
		Fourth	37.7500			
Bulk fill Packable X-tra fil	Poly wave	First	63.7500	0.019		
		Fourth	41.7500			
	Quartz Tungsten Halogen	First	55.0000	0.000		
		Fourth	24.7500			
Conventional Packable Grandio	Poly wave	First	50.2500	0.005		
		Fourth	27.2500			
	Quartz Tungsten Halogen	First	68.0000	0.015		
		Fourth	52.7500			
	Poly-wave	First	52.7500	0.009		
Conventional Flowable Grandio		Fourth	69.7500			
	Quartz Tungsten	First	75.5000	0.007		
	Halogen	Fourth	69.0000			