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Original Article

Bulk-Fill versus Conventional Composite: A Comparative Analysis on Degree of Conversion

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Abstract

Aim: This study aimed to determine the degree of conversion (DC) of two resin-based composites: a conventional nanohybrid composite and a bulk-fill flowable composite (SureFil® SDR™ flow). **Materials and Methods:** DC was evaluated at 1-, 2-, 3-, and 4-mm depths at varying irradiation times (20 and 40 s) by Fourier-transform infrared spectroscopy. Disc-shaped specimens of varying depths were prepared and photoactivated with a third-generation light-emitting diode light-curing unit. The specimens were stored for 24 h in the dark at ambient temperature and pulverized into a fine powder which was mixed with potassium bromide. After homogenization, the mixture pressed to form a pellet. All pellets were evaluated by an infrared spectrometer equipped with a triglycine sulfate detector using diffuse reflectance. The percentage of monomer conversion was determined from the ratio of the absorbance intensities of the aliphatic carbon–carbon double bonds (C = C) and the internal standard before and after the curing of the composite, represented by the aromatic carbon–carbon single bonds (C-C). **Results:** The DC data for the bulk-fill resin showed no significant difference ($P > 0.05$) for different irradiation depths, whereas for the conventional nanohybrid resins, statistical analyses revealed a significant result of the variables time and depth as well as the interaction between them. **Conclusions:** The results indicated that SDR™ achieves a 4-mm depth of cure with both 20- and 40-s light exposures.

Keywords: Bulk-fill flowable, composite resins, degree of conversion

Introduction

Since the introduction of composites in the early 1960s, resin-based materials have shown a constant technical progress with regard to new types of filler particles.[1] However, despite the evolution of resin-based materials, conventional composite resins used at the beginning of the 21st century still present unfavorable aspects such as an insufficient degree of monomer conversion[2] and generation of stress by polymerization shrinkage upon curing.[3]

No material can be "ideal:" exhibiting a high degree of conversion (DC) and minimal polymerization shrinkage.[4] These features vary generally antagonistically because a high DC occurs when the chemical interlacing of the monomers is increased, which consequently produces a higher volume shrinkage.[5] The main problem of a higher volume shrinkage is the higher concentration of stress induced on the cavity walls,[6] which may result in cracks, gaps, secondary caries,[7] and postoperative sensitivity.[8] In turn, a lower DC may create problems such as release of unreacted monomers[9] or high water sorption.[10]

Manufacturers have claimed that bulk-fill resins reach a satisfactory DC, leading to a maximal increment thickness of 4–5 mm, depending on the trademark, which can dispose of the need for applying and curing composite resins in increments of limited thickness.[11] The bulk-filling of cavities, besides saving clinical time, also prevents the formation of air bubbles and contamination between these increments, thus favoring the homogeneity of the restorative material. Therefore, bulk-fill resins have been increasingly studied.

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It is well known that light energy transmission through a composite is attenuated drastically with increasing depths from the light-irradiated surface. Therefore, increments of conventional composite resins are limited to 2 mm. A decrease in monomer conversion can compromise physical properties and clinical performance of the restorations[5] because DC is a codetermining factor of the physical and chemical properties of restorative resins, such as hardness, wear resistance, compressive strength, flexural strength, dimensional stability, solubility, discoloration, and degradation reactions.[12] Studies have found that the release of unreacted monomers that remain in the material[5] may stimulate bacterial growth around the restoration, irritate soft tissues, and cause allergic reactions in some patients.[9,13]

Several methods have been used to assess the DC: infrared spectroscopy, calorimetry, Raman scattering,[14] and even indirect techniques such as measuring hardness in the top and bottom. Both infrared spectroscopy and Raman spectroscopy are classified as vibrational techniques because they are sensitive to the vibrational modes of molecules,[15] whereas calorimetry allows measurement of the methacrylate group conversion through an exothermic polymerization reaction.[16] Among the spectroscopic techniques commonly reported in the literature, Raman technique has the advantage of analyzing the material without any sample preparation; however, Fourier-transform infrared (FTIR) technique has traditionally provided more useful information on the monomer conversion of dental composites.

Although several techniques have been described in the literature to assess DC, the development of bulk-fill resins brings with it the need to adapt the existing techniques. This is especially true for the preparation of specimens as manufacturers advertise that the specimens are adequately photoactivated in increments up to 4 mm in depth. This specification is not in accordance with the International Organization for Standardization (ISO) 4049 standard for testing composites.[17]

Therefore, the aim of this study was to evaluate the DC of a bulk-fill flow composite through FTIR analysis in comparison to a nanohybrid composite resin containing a conventional matrix at different specimen depths and irradiation times. The following null hypotheses were tested: (1) depth has no significant influence on DC, (2) irradiation time has no significant influence on DC, and (3) the bulk-fill flow composite shows no difference in DC behavior when compared with the conventional material.

Materials and Methods

Table 1 shows the resin composites tested.

Specimen preparation

To determine DC at different depths, a set of four cylindrical molds was prepared according to the schematic shown in Figure 1. The molds (1-mm depth and 4-mm diameter) were successively filled and superposed on each other, with a polyester strip separating consecutive molds. The light-emitting diode light-curing unit (LED LCU) (Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein, Europe) tip was then placed on the upper surface of the set of molds through a glass slide, and light was activated in the high-power mode (approximately 1100 mW/cm^2) for either 20 or 40 s. Then, the specimens were carefully removed from the molds and identified according to the number of the samples and its depth (i. e., 1, 2, 3, and 4 mm). Five replicates (n = 5) were performed for each composite resin. The specimens were then stored for 24 h in the dark at 37 ± 1°C before analysis.

Degree of conversion (%) analysis

After storage, the composites were pulverized into a fine powder which was weighed (5 mg) and thoroughly mixed

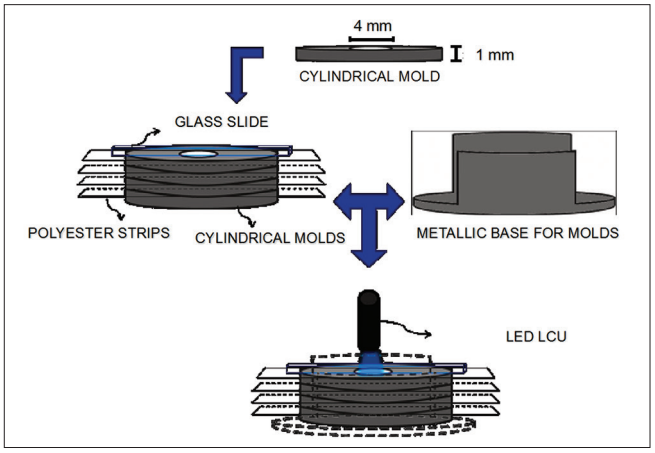

Figure 1: Schematic representation of sample preparation

Table 1: Summary of the dental resin composites investigated in this study

Brand name	Type	Composition	Filler loading (weight %)	Manufacturer	Shade
SureFil® SDR™ flow	Flowable bulk fill	Ba-Al-F-B silicate glass, Sr-A-F silicate glass, modified UDMA, EBPADMA, TEGDMA. CQ photoinitiator, BHT, UV stabilizer, titanium dioxide, iron oxide pigments	68	Dentsply	Universal
Esthet-X® HD	Conventional nanohybrid	Bis-GMA, Bis-EMA, TEGDMA, CQ photoinitiator, UV stabilizer, pigments. Combination of particulate fluoro-barium-borosilicate glass with a mean particle size below 1 μm and silica nanoparticles of 0.04 μm	60	Dentsply	A2

UDMA – Urethane dimethacrylate, EBPADMA – Ethoxylated Bisphenol A dimethacrylate, TEGDMA – Triethyleneglycol dimethacrylate, Bis-EMA – Bisphenol A polyethylene glycol diether dimethacrylate, Bis-GMA – Bisphenol A dimethacrylate, CQ – Camphorquinone, BHT – Butylated hydroxytoluene, UV – Ultraviolet

with powdered potassium bromide salt (100 mg). The obtained mixture was inserted into a pelleting device and pressed with a load of 10 t for 1 min to obtain a pellet.

The pellets were analyzed using a FTIR spectrophotometer (Nexus-470 FT-IR, Thermo Nicolet, EUA) to measure the DC of the composites. DC is defined by the number of carbon–carbon double bonds (C = C) that are converted into carbon–carbon single bonds. FTIR spectra of both uncured and cured samples were obtained through a triglycine sulfate (TGS) detector using diffuse reflectance coupled to a computer. The spectra were recorded in the absorbance mode with the following predetermined settings: 32 scans, 4 cm^{-1} resolution, and 300–4000 cm^{-1} wavenumber.

Following Ribeiro *et al*.,[18] the percentage of unreacted carbon double bonds (% C = C) is obtained as the ratio between the absorbance intensities of the aliphatic C = C (peak at 1638 cm^{-1}) and the internal standard before and after composite curing, represented by the aromatic C–C (peak at 1608 cm^{-1}). DC was determined according to the following formula:

$$DC(\%) = \frac{\text{cm}^{-1} \quad \text{cm}^{-1} \text{ cured}}{\text{cm}^{-1} \quad \text{cm}^{-1} \text{ uncured}}$$

Statistical analysis

Statistical analysis was performed for the evaluation of data normality (Shapiro–Wilk test) using SPSS software version 13.0 (SPSS Inc., Tulsa, OK, USA). The assessment of DC was carried out by two-way analysis of variance (ANOVA). Multiple comparisons of DC means by Bonferroni test followed the analysis. All statistical analyses were performed at 5% significance level.

Results

Results are listed in Table 2. Two-way ANOVA of the DC data for the SDR™ resin showed no significant difference ($P > 0.05$) for the various irradiation depths. The mean DC of SDR™ varied from a maximum of 78.24 (±3.46)% at an irradiation depth of 2 mm (irradiated for 40 s) to a minimum of 73.20 (±2.92)% at an irradiation depth of 4 mm (irradiated for 20 s). For Esthet-X™, two-way ANOVA and Bonferroni test revealed a significant effect of the variables time and depth as well as the interaction between them. DC varied from a maximum of 74.79 (±2.63)% (2 mm, irradiated for 40 s) to a minimum of 3.94 (±9.50)% (4 mm, irradiated for 20 s).

Considering DC as a function of irradiation time, a difference could be observed only for Esthet-X™ at 4-mm depth, where 40 s of irradiation exhibited a significantly better DC than 20 s of irradiation. However, for Esthet-X™, the DCs of 1 mm and 2 mm (for both 20 and 40 s of irradiations) are statistically similar, decreased linearly for 3 mm and 4 mm. SDR™ exhibited no differences between irradiation times for all depths. Both composite resins performed similarly for depths of 1 mm and 2 mm.

Discussion

It is well known that light energy emitted from an LCU decreases when transmitted through composites.[19] Hence, more the distance from the irradiated surface, the lower will be the DC of the resin-based material.[20] Therefore, we considered incremental techniques to apply and cure resin composites with increments of 2-mm maximal thickness.[2,20] However, in contrast to these principles, the use of bulk-fill composites with increments of 4-mm maximal thickness is advocated nowadays, allowing a less time-consuming and safer restoration technique than before, especially for deep cavities. The results of this study show that 4-mm SDR™ samples photoactivated for 20 s meet the manufacturer's expectations. Thus, a molar with a Class II mesio-occlusal-distal cavity of about 6-mm deep in proximal boxes restored with a bulk-fill resin can save less than half the clinical time it would take when restored by the conventional incremental technique. This is possible because a single increment of 4 mm can fill the two proximal boxes and part of the occlusal box and polymerize it in just 20 s and the other 2 mm if left can be filled in three conventional increments (increments of no more than 2 mm with a conventional resin) polymerized for at least 20 s each. Thus, it adds to 80 s of photoactivation, whereas for conventional techniques, this would need approximately 10 increments using a conventional resin for filling the same cavity, each being cured for at least 20 s, totaling 200 s in all.

More than 25 years ago, the ISO introduced a method for defining the depth of cure of resin composites, the well-known "ISO 4049; Depth of cure."[21] According to this method, tube-shaped specimens of the tested resin are light-cured, and the uncured part of the resin is then scraped away. The depth

Table 2: Mean and standard deviation of degree of conversion evaluated on specimens of variable depths after different irradiation times

Material	Time (s)	DC 1 mm (%)	DC 2 mm (%)	DC 3 mm (%)	DC 4 mm (%)
Esthet-X*,α	20	70.74±4.97^{aA}	62.40±3.99^{aA}	44.46±23.90^{bA}	3.94±9.50^{cB}
	40	73.66±4.36^{aA}	74.79±2.63^{aA}	42.14±7.70^{bA}	29.81±5.23^{bA}
SDR**,α	20	76.66±3.48^{dC}	76.88±1.24^{dC}	76.72±3.69^{dC}	73.20±2.92^{dC}
	40	75.62±1.95^{dC}	78.24±3.46^{dC}	76.24±4.46^{dC}	74.64±7.07^{dC}

*The influence of effects and interaction between time and curing depth was considered statistically significant ($P<0.05$), **The influence of effects and interaction between time and curing depth was considered not statistically significant ($P>0.05$), αEqual lowercase letters in the same row represent no statistical difference; equal capital letters in the same column represent no statistical difference ($P<0.05$). DC – Degree of conversion

of cure is then given by the total length of the residual hard tube divided by the factor 2. The resulting value determines the maximum increment thickness of the tested resin. Because resin composites are in continuous development with regard to their composition and properties, studies have tested new ways to evaluate the depth of cure of new resins, especially for bulk-fill ones.[17,20,22] In a study published in 2012,[20] the authors verified the accuracy of the ISO 4049 method when used for bulk-fill materials and concluded that the method overestimates the depth of cure. Based on these findings, an alternative method was used in this study to determine the depth of cure through the DC assessment of each millimeter of resin samples.

Many studies have assessed the DC of the external surface of a restoration using simple techniques; however, the evaluation of monomer conversion in the inner layers of resins is not simple to assess.[7] In the current study, a matrix has been synthesized specifically for the assessment of the DC of the resins at different depths. A similar device was used by Finan *et al*.,[17] who assessed the DC in specimens that are up to 8-mm deep and 11-mm diameter. In the previously mentioned study, the authors observed a maximum DC of 59% for SDR™ at an irradiation depth of 1 mm and at least 45% at a depth of 8 mm. In the present study, using a similar methodology, SDR™ resin achieved a maximum DC of 76% considering the same irradiation time of 20 s. The difference between these results can probably be due to the photoactivation unit. We used a high-intensity LED, whereas the other study used a quartz-tungsten-halogen LCU (operated at an output intensity of 650 ± 26 mW/cm^2). Rueggeberg *et al*.[23] also reported the variation when the infrared spectroscopy technique was used, more specifically to the standard baseline technique that was used when C = C peak intensities are determined. They pointed out that baselines can be drawn in a number of locations, and not all researchers draw baselines in a similar manner. Thus, results for DC may be imprecise when interlaboratory results are compared.[23]

The evaluation of DC permits characterization of the conversion of monomers into polymers. Several methodologies can be used to assess the DC of composite resins.[16] FTIR spectroscopy is a technique that may be applied for the evaluation of the conversion of monomers into polymers by calculating the ratio of the aliphatic C = C absorption at 1638 cm^{-1} to the aromatic C = C absorption at 1608 cm^{-1}.[23] This technique is well reported in the literature; however, recent studies[7,17,22,24] have used infrared spectroscopy with attenuated total reflection (ATR) because of its simplified sample preparation when compared to the TGS detector. Considering the technique used in this study, Shin *et al*.[15] highlighted the fact that preparing samples as thin and fragile sections is one of the disadvantages of the technique. However, we consider that by crushing the specimens, we are using a portion of the specimen's constituent for analysis and not just its surface of it, as it happens when the ATR crystal is used.

The efficiency of polymerization is completely dependent on the characteristics of the light produced.[16] Thus, a third-generation LED LCU was chosen because its peak emission coincides with the maximum absorption of the camphorquinone, which is the photoinitiator present in both tested resins[25] to achieve best results for the tested materials, without any adverse influence of LCU on the results. In addition, two different curing times were used (20 and 40 s). Although manufacturer's recommendation indicates only 20 s, this study evaluated a longer time aiming to verify any potentiating effect on DC.

In this *in vitro* study, depth influences the DC of the Esthet-X™ resin but not SDR™. Therefore, the first null hypothesis was partially accepted. The same is true for the second null hypothesis because the irradiation time influenced only the Esthet-X™ resin. However, up to a depth of 2 mm (the maximum increment recommended by the manufacturer), irradiation time did not have any influence because, for both times tested, the DC values were statistically similar. The third null hypothesis was rejected because the materials tested behaved differently with regard to the DC.

This study evaluated only one bulk-fill material, which is a limitation to generalizing the conclusions. More studies need to be carried out to assess the behavior of other brands of bulk-fill composites.

Conclusions

- This study indicates that SDR™ achieves a 4-mm depth of cure with both 20- and 40-s light exposures
- Depth has a significant influence on the DC of Esthet-X™ but not of SDR™
- Irradiation time does not have any significant influence on the DC of SDR™, while it influences that of Esthet-X™.

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Conflicts of interest

There are no conflicts of interest.

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