Original Article

Evaluation of the Polymerization Properties of Bulk-Fill Composite Resins

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Abstract

Introduction: The aim was to evaluate the polymerization properties of bulk-fill composite resins in terms of the degree of conversion (%DC), Vickers hardness (HV). SonicFill (SF, Kerr, CA, USA), Tetric Evo Ceram Bulk-Fill (TECBF, Ivoclar Vivadent Schaan, Lichtenstein), and X-trafil (XTF, Voco GmbH, Cuxhaven, Germany) were investigated. **Methods:** The samples prepared from composites (n = 10) were polymerized via three different curing modes (standard mode: 20 s, high-power mode: 12 s, extra power mode: 6 s) using a polywave LED. %DC was determined by FTIR-ATR-spectroscopy. HV was measured at the top and bottom of the specimens. Data were analyzed by two way ANOVA, and Bonferroni's post-hoc respectively (α=0.05). **Results:** Except TECBF (%41.10 ± 3.1) in three different curing modes, all materials showed no significant inferior %DC. In all curing modes, the highest hardness ratio was found in XTF (0.84 ± 0.03 GPa), and the lowest hardness ratio was found in the TECBF (0.78 ± 0.02GPa). In the cases where the extra power mode of the Valo Led was applied, the lowest degree of conversion and hardness were also measured. **Conclusion:** It has been observed that the examined properties of some tested materials change with the curing time and power of the light and however, the results are dependent on the material. Extra power mode of the Valo Led negatively affected high viscosity bulk-fill composites in terms of their polymerization properties.

Keywords: Bulk-Fill, Resin Composite, Light Curing, Degree of Conversion, Vickers Hardness

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Introduction

The general recommendation is that traditional composite materials be layered with a final total thickness of no more than 2 mm to ensure light transmittance and optimal polymerization of the restorations when undertaking extensive restoration of the material loss in teeth. However, performing restorations on teeth with deep cavities using this layered placing method is time-consuming for both patients and dentists and may result in moisture contamination and the presence of porosity between the layers. To resolve these problems, shorten the time patients must spend undergoing the procedure, and reduce costs, bulk-fill composite resins that can be used in a depth of 4–5 mm, have recently been developed (1).

Developers have modified the initiator dynamics of the bulk-fill composites and increased their translucence-related properties, ensuring that bulk-fill composites can be used in a thick layer (2). Increasing translucence facilitates light penetration and results in deeper polymerization (3). When bulk-fill composites such as X-trafil (XTF) are compared to traditional composites, it becomes clear that the filler rate is decreased and the particle size increased, achieving more advanced light transmittance. Accordingly, polymerization can be ensured within the deep points of restorations conducted with bulk-fill composites. As the amount of inorganic filler decreases but its size increases, the total area of the filler–matrix interface also decreases; thus, light scattering is decreased, facilitating light penetration into deeper points (4). As a composite with high viscosity, Sonicfill (SF) is a bulk-fill composite featuring a sonic activation system; it is implemented in cavities in the same manner as a liquid composite, enabling contouring and shaping on itself for modeling.

These recently developed high-viscosity bulk-fill composite resins eliminate the need for an upper-layer procedure to complete the restoration process (5). Bulk-fill composites have an initiator system and camphorquinone (CQ)-based systems; this is also the case for traditional composites. In contrast, Tetric Evo Ceram Bulk-Fill (TECBF) contains a normal CQ/amine initiator system as well as Ivocerin, a germanium-based initiator. This initiator system, the photoinitiator, is more sensitive to light and absorbs light better compared to CQ. Accordingly, it improves the TECBF system's photopolymerization activity (4). New illumination devices overcome the issues of insufficient polymerization and narrow bandwidth present in first- and second-generation LED illumination devices. Specifically, several manufacturers have developed third- and fourth-generation LED illumination devices that can generate light between the wavelengths of 405–410 nm (6). One third-generation LED illumination device (Valo, Ultradent Products Inc. South Jordan UT, USA) has an irradiation capacity of 3200 mW/cm2 and features safe polymerization for 3–6 s (7). Third-generation LED illumination devices contain blue diodes like second-generation devices, but they also include one or multiple diodes with a lower power, therefore forming a purple diode. LED devices with purple and blue diodes have the capacity to activate both CQ and other initiators (8). The rate of converting monomers to polymers in composite resins—in other words, the rate of conversion from carbon double bonds (C=C) to carbon single bonds (C-C) during the polymerization phase—is called the conversion or polymerization degree (9, 10).

Fourier transform infrared (FTIR) spectroscopy is a spectroscopic method that has been recently and frequently used to easily calculate the degree of conversion (DC%) from monomer to polymer in composite resins(11). In cases where it is challenging to use the principle of transmittance, the principle of reflection can be used, and the spectra can be recorded via the attenuated total reflectance-Fourier transform infrared (ATR-FTIR) unit. Small portions of solid and liquid samples are used to perform measurements before beginning the sampling process (12). In addition, according to a study, it was stated that the polymerization depth value of composite resins can be found by comparing the hardness values between the lower and upper surface of the sample. Further, the clinically acceptable polymerization depth value can be achieved if the hardness rate between the lower/upper surface is a minimum of 0.80 GPa(13).

The aim of this study is to evaluate the polymerization properties of bulk filled composite resins in terms of conversion degree (% DC) and Vickers hardness (HV). The first hypothesis is that there would be no differences among the materials' Vickers hardness and degree of conversion. The second hypothesis of the study is that the use of Valo Led curing unit in three different modes would be no differences on the conversion degree and Vickers hardness of the materials.

Materials and Methods

This study used three different modes (standard, high-power, and extra-power) of a Valo Led curing unit (Ultradent Products Inc, South Jordan, UT, USA) and three different bulk-fill composite resins. The composite resins are presented in Table I, and the illumination device is listed in Table II.

Table I. Composite resins used in the study and their contents

Name of the Material	Manufacturing Firm	Material Type	Matrix Type	Filler Type	Filler Volume (%)	Serial No:	Color
SonicFill	Kerr Orange, CA, USA	Bulk-Fill Composite	Bis-GMA, TEGDMA, BisEMA,	Inorganic filler	66	6383676	A2
Tetric EvoCeram Bulk Fill	Ivoclar Vivadent Schaan, Lichtenstein	Bulk-Fill Composite	Bis-GMA, Bis-EMA, UDMA	Ba–Al–Si glass, prepolymer filler (monomer, glass filler and YbF3), spherical mixed oxide	60-61	V19410	IVA
X-trafil	Voco Cuxhaven, Germany	Bulk-Fill Composite	Bis-GMA, UDMA, TEGDMA	Ba–B–Al–Si glass	70	1724335	universal

Table II. Illumination device used in the study

Illumination Device Name	Manufacturing Firm	Illumination Device Type	Serial No
Valo	Ultradent Products Inc., South Jordan, UT, USA	Polywave Standard mode: ~1000 mW/cm2 _ 10%, 20 s High power mode: ~1400 mW/cm2 _ 10%, 12 s Extra power mode: ~3200 mW/cm2 _ 10%, 6 s	V02640

Preparation of Test Samples

Specimens were prepared (d = 5mm, h = 4mm, n = 10 per product) by packing the material pastes in opaque plastic molds pressed between two glass slides (0.5mm thick) covered with polystyrene matrix strips to remove material excess and avoid oxygen inhibition upon setting. In the power analysis to determine sample size, it revealed that at least 10 samples were required for each group in order to obtain sufficient statistical power ($n>10$, $\alpha=0.05$, and $1-\beta=0.80$). The samples prepared from three different bulk-fill composite resins were polymerized using three different modes of Valo Led curing unit (standard, high-power, and extra-power). All specimens were stored for 24h in distilled water at 37 ◦C prior to %DC and HV measurements.

FT-IR Spectroscopy

ATR-FTIR spectra were recorded at a wave-number resolution of 4 cm-1 between the wave number range of 4000–400 cm-1 using the VERTEX 70v Diamond ATR unit/Spectra two system.

To eliminate the environment factor, “background spectrum” was obtained first; thereafter, ATR-FTIR spectra of non-polymerized (uncured) bulk-fill composite samples were recorded. Additionally, polymerized (cured) bulk-fill composite samples were

turned into powder using a mortar and pestle (90 polymerized samples, two spectra from each sample) and ATR-FTIR spectra were recorded.

A baseline was implemented for the area between the 1660–1590 cm-1 within the infrared spectra of all samples to ensure that the vibration bands used to measure the monomer degree of conversion did not affect the band absorption values. The quantitative determination of %DC was based on the two-band technique. The peak of the aliphatic C=C bond stretching vibrations (1637 cm−1), which are consumed during polymerization, was chosen as the analytical absorption band, whereas the peak of the aromatic C-C bond stretching vibrations (1608 cm−1), which are not affected by the curing process, was selected as the reference absorption. The monomer/polymer conversion rate was calculated using the following formula:

DC(%) = [1- (A1637/A1608) polymerized / (A1637/A1608) non-polymerized] x 100

where, A is the net peak absorbance height of cured (p) and uncured (m) materials at the specific wavenumbers (14).

Vickers Hardness Test (VH)

Micro-hardness tests were performed on both lower and upper surfaces of the samples in each group. Hardness measurement was performed by applying 100 grams of force (grf) (2.9 newtons) for ten seconds in each measurement(15) and leaving marks on three points from the lower and upper surfaces using a micro-diamond tip(16). Then, the ratio of depth and side length within the marks was automatically analyzed by the tip of the device to find the Vickers hardness number (VHN). The hardness rate of each sample was found using the following formula:

V.Kmean = V.Klower surface / V.Kupper surface

Samples with a hardness degree of 0.80 or higher were deemed to have a sufficient degree of conversion.

Statistical Analysis

Statistical analysis was performed by two-way ANOVA (independent parameters: bulk-fill composite resins and light-curing modes). For multiple comparisons, the Bonferroni post-test was used. The statistical significance level was set at $P < 0.05$.(Table III-IV)

Table III. The mean values, standard deviations and Bonferroni's multiple comparison test results of the measurement data obtained in different light modes of the conversion degree (%DC) of composites in FTIR-ATR.

Composite Type / Light Mode	Standard mode	High power mode	Extra power mode	Total
SonicFill	55.93±1.1	58.10±1.5	56.16±1.5	56.7±1.7^{A}
Tetric Evo Ceram	40.5±3.4	41.64±3.0	41.18±3.1	41.10±3.1^{C}
X-trafil	45.64±1.8	45.24±3.7	40.59±2.9	43.82±3.6^{B}
Total	47.35±6.9^{a}	48.32±7.7^{a}	45.98±7.6^{b}	47.22±7.4

Different letters indicate statistically different mean values for the light levels on the same line ($P<0.05$). Different capital letters on the same column indicate different statistically different mean values for the composite types on the same column ($P<0.05$).

Table IV. Upper, lower and hardness rates of bulk-fill composites, standard deviation figures, and Bonferroni's multiple comparison test results

	Composite Type	Standard mode	High power mode	Extra power mode	Total
Upper Surface	SonicFill	76.78±0.45	76.70±0.40	76.0±0.87	$76.50±0.7^{A}$
	Tetric Evo Ceram	67.22±0.6	66.41±0.7	65.80±0.8	$66.48±0.9^{B}$
	X-trafil	77.81±2.3	75.38±2.04	75.16±0.9	$76.11±2.1^{A}$
	Total	$73.94±5.0^{a}$	$72.83±4.8^{b}$	$72.32±4.7^{b}$	73.03±4.86
Lower Surface	SonicFill	62.20±0.62	65.28±1.25	61.70±0.83	$63.06±1.84^{N}$
	Tetric Evo Ceram	53.33±0.35	52.47±0.51	50.30±1.04	$52.03±1.46^{O}$
	X-trafil	67.80±2.27	62.56±1.70	61.70±0.70	$64.01±3.20^{M}$
	Total	$61.11±6.20^{m}$	$60.10±5.73^{n}$	$57.90±5.53^{o}$	59.70±5.91
Hardness rate	SonicFill	0.81±0.008	0.85±0.015	0.81±0.006	$0.82±0.022^{Y}$
	Tetric Evo Ceram	0.79±0.006	0.79±0.006	0.76±0.016	$0.78±0.016^{Z}$
	X-trafil	0.87±0.009	0.83±0.015	0.82±0.012	$0.84±0.025^{X}$
	Total	$0.82±0.034^{x}$	$0.82±0.028^{x}$	$0.79±0.027^{y}$	0.81±0.032

a, b, c, m, n, o, x, y, z; Different letters indicate statistically different mean values for the light levels on the same line (P<0.05). A, B, C, M, N, O, X, Y, Z; Different letters on the same column indicate different statistically different mean values for the composite types on the same column (P<0.05).

Results

Degree of Conversion from the ATR-FTIR Spectra

The data obtained were analyzed using a two-factor interactive model. Based on two-way ANOVA, the interaction effect of Valo Led light-curing modes on the degree of conversion of bulk-fill composite resins was significant (P=0.005). The degree of conversion and standard deviation values of the bulk-filled composite samples in FTIR-ATR are listed in Table 3. In all the light-curing modes, SF had the highest conversion (56.7 ± 1.7%), while TECBF had the lowest conversion (41.10 ± 3.1%). When standard mode and high power mode were used for polymerization of bulk-fill composite resins, no significant difference was noted in the degree of conversion of bulk-fill composite resins (P=0.154) while extra power mode showed significant differences (P=0.003). For all groups, the lowest degree of conversion was found when the extra power mode (6 sec) of the Valo Led was used.

Assessment of Micro-Hardness

These data were also analyzed using a two-factor interactive model; the upper and lower surface hardness and hardness rates of the samples are listed in Table 4. When the hardness ratio of the bulk-fill composites used in the study was examined, statistically significant differences were found between the bulk-fill composites used according to all curing modes.(P=0,000) The highest hardness ratio was found in the X-trafil groups (0.84±0.025), and the lowest hardness ratio was found in the TECBF groups (0.78±0.016).

When the effect of the Valo Led curing modes on the hardness ratio of the bulk-fill composites was examined, no significant difference was statistically found between the hardness rates of the sample groups in which the standard and high-power mode were applied (P=0.685) ; the hardness rates of the groups in which the extra strength mode was applied were lower (P=0.000).

Discussion

This study assessed the degree of conversion of three high-viscosity bulk-fill composites (SF, TECBF, XTF) using three different light modes of Valo Led curing (standard mode, high-power mode, extra-power mode) with an ATR-FTIR device; the micro-hardness of these composites was examined via the VMH.

The results of the current study showed that the %DC of SF was significantly higher than other bulk-fill composite resins. In addition, an adequate %DC could not be achieved in the samples where the extra power mode of the Valo LED light source was used.

Many factors are effective in achieving a sufficient polymerization in composite resins, such as the type and matrix structure of the composite resin, dimension, amount, distribution, and color of the filler particles, and the amount and type of the photo-initiators within the matrix structure. Moreover, the type of the light source as well as the power and intensity of the light, wavelength range, duration of the implementation light, size of the light tips, and the distance where the light is applied can influence the polymerization of composite resins (17, 18).

There is no consensus regarding the degree of conversion requirements for minimum FTIR analysis for the main restorative materials; however, a degree of conversion of 55% minimum is desirable(19). During the FTIR analysis, the SF composite displayed a sufficient degree of conversion under all the light modes, whereas the TECBF and XTF composites were not sufficient in terms of polymerization.

Replacing a certain portion of the Bis-GMA present in the SF with flexible siloxane-methacrylate copolymer (SIMA) with a low-viscosity ethoxyl analog (bis-EMA) can increase the molecular reactivity of crosslinked monomers. Additionally, sonic vibration may decrease the viscosity and increase the monomer activity during the process of placing through the particular hand tool of the system, thereby increasing the degree of conversion (20). The SF bulk-fill composite used in this study reached the highest degree of conversion in the FTIR analysis compared to the XTF and TECBF bulk-fill composite, which might be due to the aforementioned reasons.

Two approaches have been adopted thus far to manage the degree of conversion of bulk-fill composite restorations. The first is to increase the transparency. Maximizing the light transmittance properties is not possible, as this process affects the aesthetic appearance of many bulk-fill composites. However, the adaptation of the refractive index between the growing filler dimension, filler, and resin matrix is one potential way to increase the composite's transmittance (21). The second approach is to increase the photo-initiator activity. Most modern dental composite resins are based on the CQ-amine system with an efficient free radical. To increase this performance, TPO derivatives (Lucerin-TPOTM, Irgacure-819TM) or benzoyl-germanium compounds (IvocerinTM) have been included as the additional photo-initiators, ensuring a synergetic effect on the CQ. TECBF is based on Ivocerin technology (22).

Tarle et al. (23) conducted a study regarding the transparency of the composite resins (Tetric Evo Ceram Bulk-fill (TECBF), X-trafil (XTF), and QuiXTFil-QF). They reported that TECBF had a transparency of 15%, whereas the transparency rate of QF was 17% and the transparency rate of XTF was 23%. They found a better degree of conversion for XTF that was more transparent than that of TECBF. Accordingly, they stressed the impact of material transparency on polymerization depth.

Operating the additional photo-initiator system in ECBF (Ivocerin) did not compensate for its lower transparency rate, as the polymerization kinetic decreases more quickly in deeper layers (24). Zorzin et al. (25) indicated that although TECBF contained a dibenzoyl germanium-based photo-initiator stimulated by a light between 380–450 nm, a higher degree of conversion or greater hardening depth was not achieved for this composite. The authors posited that this lower degree of conversion might be related to the lower penetration of light due to the short- wavelength (purple) within the composite. As Zorzin et al. used the Valo Led curing unit, their results are coherent with the results of the present study.

FTIR was an effective method for determining composites' degree of conversion; it is safely and commonly used to detect direct C=C stretching vibrations(26). The micro-hardness tests—as well as the FTIR, which is used as a direct method to find the degree of conversion—are used as indirect methods to determine the physical properties of the composite material (27). The VH method uses the ratio of the lower surface hardness value of composite resin to the upper surface hardness value. Theoretically, the ratio of the lower surface hardness should be at least 80% of the upper surface hardness of the composite resin for the polymerization to be considered as successful (28). A ratio of 1 indicated full polymerization, whereas 80% was accepted as being sufficient (29).

The XTF and SF composites displayed a hardness rate higher than 80% in all the light modes within the VH test implemented on certain samples of the study, whereas the hardness rate of TECBF was less than 80%, indicating an insufficient degree of conversion. Thus, the first hypothesis that all the bulk-fill composites would show sufficient polymerization at a depth of 4 mm was partially rejected. Leprince et al. (30) examined the physical–mechanical properties of bulk-fill composites and assessed the lower–upper surface hardness rates of bulk-fill (SF, XTF, and TECBF) composites polymerized for 40 seconds using LED light sources. The rate was less than 80% for TECBF and greater than 80% for the XTF

and SF composites. The VH values of XTF and SF may be related to the differences in the chemical compounds within the monomers of the composite resins. Additionally, based on the increase in the filler size of the bulk-fill composites, greater transparency results in higher VH values (31). The VH value of XTF being higher than the same value of SF might arise from the difference within the monomer viscosity. One study reported that the liquid bulk-fill composites displayed lower micro-hardness values compared to the high-viscosity bulk-fill composites (32). The SF composite has a particular monomer content that reacts to the sonic energy quite effectively and contains a high rate of filler. Composite viscosity decreases when sonic energy is applied (to as low as 87%), which increases the liquidity of the composite resin. Such a decrease might be the cause of the VH value found in the study (33). El-Damanhoury et al. (34) conducted a study on bulk-fill composites and found that the TECBF composite micro-hardness rate (%79.73) was lower than the XTF composite micro-hardness rate (%86.43). The results of their study are coherent with the results of the present study.

One of the important factors affecting the polymerization properties of the resins is the type of light-curing unit(35). The extra-power mode of the Valo Led light curing unit, the third-generation LED light-curing (6 s), did not display sufficient polymerization activity for the bulk-fill composites used in both the FTIR analysis and VH, whereas the standard mode (20 s) and high-power mode (12 s) showed sufficient polymerization. Therefore, the second hypothesis was partially rejected.

Conclusion

Considering the limitations of this in vitro study, the SF composite (56.7%) and the XTF composite displayed sufficient degrees of conversion under all the light-curing modes of the Valo Led curing unit. The examined properties of certain studied materials have been found to change with the curing time and light strength, but the findings are material dependent. In terms of polymerization properties, the Valo Led's extra strength mode had a detrimental impact on high viscosity bulk-fill composites.

Conflict of Interest

The authors deny any conflicts of interest related to this study.

Acknowledgment

None declared.

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