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Depth-dependence of Degree of Conversion and Microhardness for Dual-cure and Light-cure Composites

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Clinical Relevance

New dual-cure bulk-fill composites show promise for uniform degree of conversion and microhardness throughout the entire depth of direct restorations.

SUMMARY

Objective: The aim of this study was to evaluate the degree of conversion (DC) through micro-Raman spectroscopy and surface microhardness in Vickers hardness (VHN) of three new dual-cure bulk-fill resin-based composites (RBCs) compared with light-cure bulk-fill and incremental RBCs at two clinically relevant depths and for two light irradiation times.

Methods: Three commercially available restorative dual-cure bulk-fill RBCs (BulkEZ, HyperFIL, and Injectafil) were evaluated and compared with three light-cure RBCs (Filtek Bulk Fill Flowable, Filtek One Bulk Fill, and incremental Filtek Z250) as controls. Specimens were prepared in two different depths (0.5 mm and 5 mm) and were light irradiated for 20 seconds or 40 seconds. Self-cure was also evaluated for the three dual-cure bulk-fill RBCs. Micro-Raman spectroscopic measurements and VHN tests (n=5) were made after 24 hours of dry storage in the dark at room temperature for all test conditions. Data were analyzed using one-way and two-way analyses of variance (α=0.05).

Results: All tested RBCs showed significantly higher DC and VHN values at 0.5-mm depth than at 5-mm depth, with the exception of BulkEZ, which showed similar DC and VHN values at two depths. The three dual-cure bulk-fill RBCs showed significantly higher DC than the three light-cure RBCs under the same curing condition. The three dual-cure RBCs showed much smaller differences in VHN values between the two depths than the three light-cure RBCs. Twenty seconds and 40 seconds of light irradiation did not generate significant difference in DC and VHN values for the three dual-cure bulk-fill RBCs at either depth or for the three light-cure RBCs at the 0.5-mm depth; however, 40 seconds of light irradiation generated significantly higher DC and VHN values for One Bulk Fill and Z250 at the 5-mm depth compared with 20 seconds of light irradiation.

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INTRODUCTION

Most dental resin-based composites (RBCs) are based on methacrylate resins, and the polymerization process is usually activated by visible light. A key way to quantify the effectiveness of polymerization is the degree of conversion (DC), which measures the percentage of carbon-carbon double bond (C=C) to carbon-carbon single bond (C-C) conversion to form the polymeric network. A high DC is vital to enhance the mechanical properties, chemical stability, and longevity of the restoration.[1,2] Incomplete monomer conversion not only causes premature failure of restorations and secondary caries[3–5] but also results in monomer elution, which is suggested to be responsible for undesirable biological responses, such as cytotoxicity and pulp tissue inflammation.[6–8] It has been shown that DC and other properties of light-cured RBCs depend on several factors: power and intensity of the light source, time and distance of irradiation, amount and particle size of the filler, ratio between the organic and inorganic components, and initiator systems.[9–13] Clinically, the most common strategy to maximize DC is to provide sufficient energy to the system by increasing curing time.[14] Several studies focusing on commercially available composites have emphasized the need to apply at least 20 seconds, but more likely 40 seconds, of light irradiation to minimize the amount of eluted monomers, even with the use of high irradiance light-emitting diode (LED) lights.[15–17]

Conventional dental RBCs are applied in increments of 2-mm thickness to allow sufficient photopolymerization.[18,19] However, this is time consuming and inconvenient, especially for deep posterior cavities. The demand for a faster and simpler restorative procedure led to the development of bulk-fill RBCs that can be placed in a single layer of up to 4-5 mm.[20] The improved depth of cure is usually achieved through greater translucency of the material, increased photoinitiator content, or an additional photoinitiator type.[13,21] Even with improved formulations, these light-cure bulk-fill RBCs can still suffer from unsatisfactory polymerization at deep layers due to impeded access or light attenuation,[22] especially for deep cavities where the distance from the top of the highest cusp to the cavity floor can reach up to 8 mm. The presence of very deep cavities has led to the development of chemical-cure RBCs that use chemical initiators to activate the polymerization reaction by mixing two components together.[23] The chemical-curing process avoids the problem of insufficient photopolymerization in deep layers and enables bulk filling in one layer. One of the disadvantages of chemical-cure materials is their fixed working time intervals: a very short one does not allow the practitioner to perfectly place the RBC material in the cavity; conversely, a longer working time interval is more time consuming and not clinically feasible.

To solve these problems, manufacturers introduced dual-cure resins. These products contain both light-cure and chemical-cure components: the light-cure component provides rapid, initial hardening of the top layers of the resins, stabilizing the restorations; then, the deeper layers that receive insufficient light irradiation are polymerized by the slower chemical-curing reaction. Some advantages of dual-cure resins as restorative materials include the possibility of a bulk insertion, clinical time saving, the achievement of polymerization in deep areas due to chemical curing, and the development of lower shrinkage stresses.[24]

There has been great interest in the curing processes of dual-cure resins. The impact of light irradiation on the chemical-curing mechanism of dual-cure resins has not been well established. Previous studies have suggested that it was necessary to light cure dual-cure resins in order to obtain an optimal polymerization of the materials, and when limited to chemical curing or inadequate light exposure, dual-cure resins may not obtain maximum mechanical properties.[25–29]

Manufacturers recently launched new dual-cure bulk-fill RBC products designed for direct restorations, such as BulkEZ (Zest Dental Solutions, Carlsbad, CA, USA), HyperFIL (Parkell Inc, Brentwood, NY, USA), and Injectafil DC (Apex Dental Materials Inc, Lake Zurich, IL, USA). Manufacturers claim that these products have unlimited depth of cure and other desirable properties, such as elimination of flowable liners, better cavity adaptation, and reduced shrinkage stress due to slower chemical curing. Very limited research is available assessing the polymerization and mechanical properties of these dual-cure bulk-fill RBCs. The aim of the present study was to evaluate the DC and Vickers hardness (VHN) values of the three new dual-cure bulk-fill RBCs in comparison with three light-cure RBCs Filtek Bulk Fill Flowable (FBF) , Filtek One Bulk Fill (FOBF) and incremental Filtek Z250 (Z250), all from 3M ESPE Dental Products (St. Paul, MN, USA), at two different depths (0.5 mm and 5 mm) for two different light irradiation times (20 seconds and 40 seconds). The null hypotheses were that 1) there would be no difference in the DC and VHN values among the investigated RBCs, 2)

Table 1: *Chemical Compositions of the Six RBCs*

RBC (Manufacturer)	Shade	Classification	Resin	Filler Content	Particle Size
BulkEZ (Danville Materials)	A2	Dual-cure, bulk-fill RBC	BisEMA, TEGDMA, BisGMA, and UDMA	Barium glass, 50-70 wt%; YbF3, 1-20 wt%	Proprietary
HyperFIL (Parkell, Inc)	universal	Dual-cure, bulk-fill RBC	BisEMA, UDMA, and other dimethacrylate monomers.	Barium glass/silica, 75 wt%	15 nm-3.5 μm
Injectafil (Apex Dental Materials, Inc)	A2	Dual-cure, bulk-fill, RBC	Bis-GMA and other methacrylate resins	Silica glass, 75 wt%	Submicron to 5 μm
FBF (3M ESPE)	A2	Light-cure, bulk-fill RBC	Bis-GMA, UDMA, Bis-EMA, and Procrylat resin	YbF3 and zirconia/silica, 64.5 wt%	0.1-5.0 μm
FOBF (3M ESPE)	A2	Light-cure, bulk-fill RBC	AUDMA, UDMA, diurethane-DMA, and DDDMA	YbF3 and zirconia/silica, 76.5 wt%	4-100 nm and clusters
Z250 (3M ESPE)	A2	Light-cure, incremental filling RBC	Bis-GMA, UDMA, and Bis-EMA resin	Zirconia/silica, 82 wt%	0.01-3.5 μm, Average 0.6 μm

Abbreviations: AUDMA, aromatic dimethacrylate; Bis-GMA, bisphenol A-glycidyl methacrylate ; Bis-EMA, ethoxylated bisphenol-A dimethacrylate; DDDMA, 1,12-dodecanediol dimethacrylate; DMA, dimethacrylate ; FBF, Filtek Bulk Fill Flowable; FOBF, Filtek One Bulk Fill; RBC, resin-based composite; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; YbF3, ytterbium trifluoride; Z250, incremental Filtek Z250.

specimen depth would have no effect on the DC and VHN values of each individual RBC, and 3) light irradiation time would have no effect on the DC and VHN values of each individual RBC.

METHODS AND MATERIALS

Six commercially available restorative dental RBCs were evaluated, including three dual-cure bulk-fill RBCs (BulkEZ, HyperFIL, and Injectafil) and three light-cure RBCs (FBF, FOBF and Z250). The chemical compositions of the six RBCs are listed in Table 1.

Specimen Preparation

Specimens were prepared in two depths (0.5 mm and 5 mm). Commercially available spacers were used as molds for specimen preparation. The 5 mm specimens were prepared in 5 mm × 6.3 mm black spacers (Model 13ME069, Grainger, Lake Forest, IL, USA), and the 0.5 mm specimens were prepared in 0.5 mm × 24 mm black spacers (Model BB-24MM-0.5, Walmart, Bentonville, AR, USA). All the specimens were prepared according to the manufacturers' instructions in a dark room at room temperature. An LED light-curing unit (Elipar S10, 3M ESPE, Seefeld/Germany) with manufacturer rated power density of 1200 mW/cm^2 was used for specimen irradiation. Uncured RBCs were filled into the molds in one increment. The specimen was immediately covered with an unbreakable plastic cover slip (Catalog No. 12-547, Fisher Scientific, Pittsburgh, PA, USA) and light-cured for 20 seconds or 40 seconds with the tip of the curing light in contact with the cover slip. The actual irradiant power density across the cover slip was measured to be 960 mW/cm^2. No light irradiation was used for the self-cure condition. All specimens were stored in the dark at room temperature for 24 hours before measurements. Five specimen replicates were used for each test condition (n=5) and three random locations were measured on each specimen surface.

Micro-Raman Spectroscopy

A LabRam HR 800 Raman spectrometer (Horiba Jobin Yvon, Edison, NJ, USA) with a monochromatic He-Ne laser (632.8 nm) and operating at excitation power of 20 mW was used to collect Raman spectra of the specimens. It was equipped with a confocal microscope (Olympus BX41, Tokyo, Japan), a piezoelectric XYZ stage with a minimum step width of 50 nm, and an air-cooled charge-coupled device detector of 1024 × 256 pixels. The following parameters were used for the Raman spectroscopy: 1800 grating, 400-μm confocal hole, and 150-μm slit width. Spectra were Raman-shift-frequency calibrated with the known peak of silicon at 520.7 cm^{-1}.

Specimen surfaces were scanned through a 100× Olympus objective. To obtain spectra of the uncured materials, the RBCs were placed on the surface of a microscope slide and were immediately scanned before polymerization happened. Raman spectra were acquired over the spectral region of 1350 to 1800 cm^{-1}

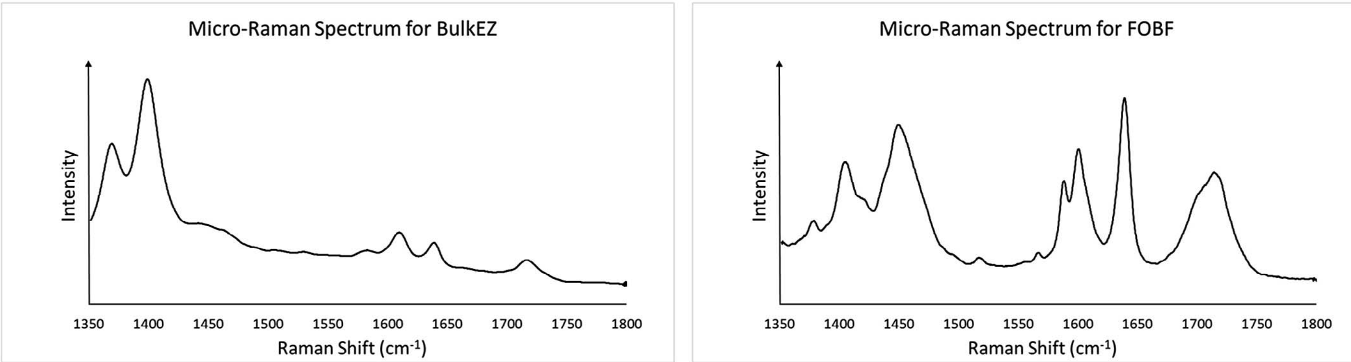


Figure 1. *Representative micro-Raman spectra for BulkEZ (left) and FOBF (right).*

with an acquisition time of 100 seconds. Five specimens with three random locations on each specimen surface were measured for each test condition. Labspec 5 software (Horiba Jobin Yvon) was used to analyze the acquired Raman spectral data. The Raman spectra were processed through cosmic spike removal, spectral smoothing, and two-point baseline adjustment. The Raman peaks at 1450, 1609, and 1637 cm^{-1} were fitted with Gaussian-Lorentzian shapes, and the maximum peak heights at those positions were measured for DC calculation.

Degree of Conversion Calculation

For RBCs containing bisphenol A-glycidyl methacrylate (Bis-GMA)/ethoxylated bisphenol A dimethacrylate (Bis-EMA) (all but FOBF), the peak of 1609 cm^{-1} (aromatic carbon double bonds) was used as an internal reference for normalization. The resin monomer content of FOBF does not contain Bis-GMA or Bis-EMA and therefore lacks the 1609 cm^{-1} peak. For FOBF, the alternative peak of 1450 cm^{-1} (C-H) was used as the internal reference for normalization (Figure 1).

The DC% was calculated by the reduction of normalized peak height at 1637 cm^{-1} (aliphatic carbon=carbon) between uncured and cured material using the following Equation (1):

$$\mathrm{DC\%} = \left\{1 - \frac{(1637\ \mathrm{cm}^{-1}/1609\ \text{or}\ 1450\ \mathrm{cm}^{-1})_{\text{Peak height before cure}}}{(1637\ \mathrm{cm}^{-1}/1609\ \text{or}\ 1450\ \mathrm{cm}^{-1})_{\text{Peak height after cure}}}\right\} \times 100.$$

The DC values from a total of 15 measurements of five specimens were used to calculate the mean and standard deviation of DC% for each test condition.

Vickers Hardness Test

The same specimen surfaces analyzed using confocal Raman spectroscopy were also used to determine their VHN values. A microhardness tester (MO Tukon, Wilson Instrument Division, Bridgeport, CT, USA) with a pyramidal diamond indenter was used to indent the specimen surface with a constant load of 100 gf for 15 seconds of dwell time. Once the load was removed, the length of the two indentation diagonals were measured using the microscope of the tester, and the VHN values were calculated automatically by the tester using the following formula:

$$\mathrm{VHN} = 0.102\frac{2F\sin\left(\frac{\alpha}{2}\right)}{d^2},$$

where F is the applied load (N), α is apex angle of the indenter (136°), and d is the average length of the diagonal (mm). Three random measurements were made for each specimen surface. The VHNs from a total of 15 measurements of five specimens were used to calculate the mean and standard deviation of VHN for each test condition.

Scanning Electron Microscopy (SEM)

Following VHN testing, specimens were polished using a 0.25-μm diamond polishing compound (Metadi, Buehler USA, Lake Bluff, IL, USA) on a spinning polisher (Buehler USA). Then the polished specimens were coated with a Pd-Au alloy in a sputtering machine (Leica EM SCD050, North Ryde, Australia). The coated specimens were placed inside a field emission scanning electron microscope (FEI-XL30, ESEM-FEG, FEI Company, Hillsboro, OR, USA) using a 15 kV electron beam at a working distance of 10 mm. SEM images were taken at 2000× magnification using a back-scatter detector to assess filler size and distribution for all six RBCs.

Table 2: *DC% Mean (Standard Deviation) for All Test Conditions*

	Composite and Irradiation Time											
	BulkEZ		HyperFIL		Injectafil		FBF		FOBF		Z250	
	20 s	40 s	20 s	40 s	20 s	40 s	20 s	40 s	20 s	40 s	20 s	40 s
Depth, mm												
0.5	74.39 (1.58)	73.03 (0.73)	79.93 (1.41)	78.29 (1.59)	76.35 (0.84)	77.18 (1.04)	60.18 (1.25)	61.21 (1.37)	55.03 (4.22)	55.45 (3.75)	62.06 (1.11)	62.37 (1.27)
5	73.50 (0.65)	72.42 (0.70)	75.14 (1.12)	74.87 (1.77)	70.23 (1.58)	68.05 (1.56)	55.54 (2.34)	56.79 (1.03)	41.34 (4.39)	49.03 (3.25)	44.83 (2.55)	51.95 (3.84)
Self-cure	72.91 (0.65)		74.69 (0.60)		69.4 (1.43)		N/A		N/A		N/A	

Abbreviations: FBF, Filtek Bulk Fill Flowable; FOBF, Filtek One Bulk Fill; Z250, incremental Filtek Z250; N/A, not available.

Statistical Analysis

Statistical analyses were performed using Microsoft Excel 2016 (Microsoft, Redmond, WA, USA) and GraphPad Prism 5 (version 5, GraphPad Software, Inc, San Diego, CA, USA). Normality of data distribution was verified by the Kolmogorov-Smirnov test. All DC and VHN data were analyzed using an unpaired Student *t*-test, and one-way or two-way analysis of variance (ANOVA) with Bonferroni posttests for multiple comparisons (α=0.05). Pearson correlation was used for DC and VHN correlation analysis.

RESULTS

Degree of Conversion

The numeric DC results for all test conditions are summarized in Table 2, and the bar chart is illustrated in Figure 2.

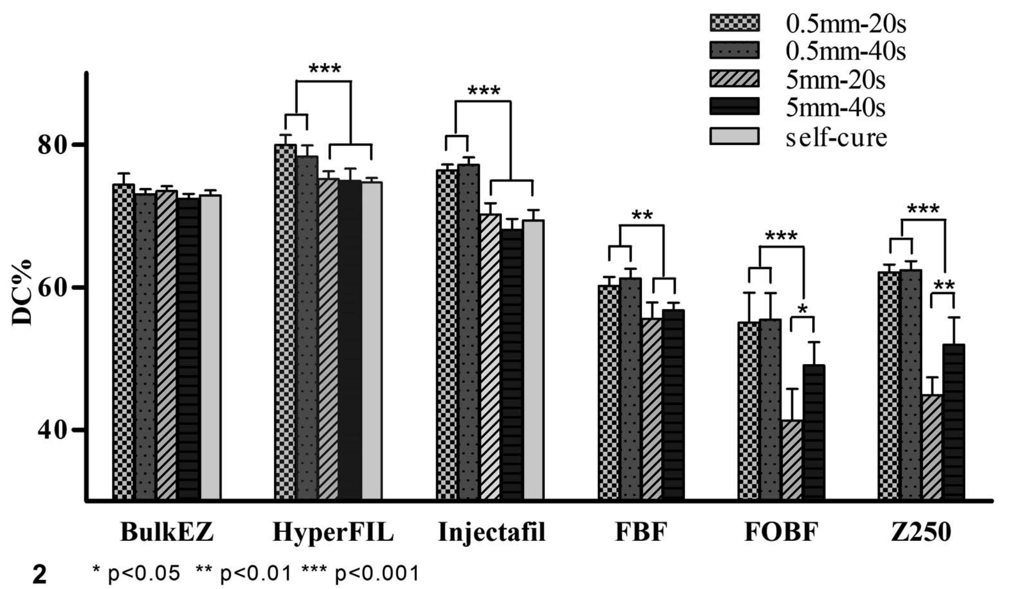
Analysis of the DC values of each individual dual-cure bulk-fill RBC at two depths and in the self-cure condition showed that for BulkEZ, the DC values were not significantly different among the two depths and self-cure conditions for either irradiation time ($p>0.05$); for HyperFIL and Injectafil, the DC values were not significantly different between 5 mm and self-cure ($p>0.05$), but they were both significantly lower than the DC values at 0.5 mm ($p<0.001$) for either irradiation time. For all light-cure RBCs, the DC values at 0.5 mm were significantly higher than those at 5 mm for either irradiation time ($p<0.005$). The ratio of mean DC value at 5 mm to mean DC value at 0.5 mm for 20 seconds of irradiation was 0.99 for BulkEZ, 0.94 for HyperFIL, 0.92 for Injectafil, 0.92 for FBF, 0.75 for FOBF, and 0.72 for Z250; the ratio for 40 seconds of irradiation was 0.99 for BulkEZ, 0.96 for HyperFIL, 0.88 for Injectafil, 0.93 for FBF, 0.88 for FOBF, and 0.83 for Z250.

Comparisons of the DC values between the 20 second and 40 second irradiation times at the same depth for each individual RBC using an unpaired Student *t*-test showed that the two different irradiation times did not generate significant differences in DC values for the three dual-cure bulk-fill RBCs at either depth or for the three light-cure RBCs at the 0.5-mm depth or for FBF at the 5-mm depth ($p>0.05$); however, 40 seconds of light irradiation generated significantly higher DC values compared with 20 seconds of light irradiation for FOBF ($p=0.0136$) and Z250 ($p=0.0086$) at the 5-mm depth. Comparisons of the DC values at the same depth for the same irradiation time among the six RBCs showed that the DC values of the three dual-cure bulk-fill RBCs were generally higher than the DC values of the three light-cure RBCs ($p<0.0001$).

Vickers Hardness

The numeric VHN results for all test conditions are summarized in Table 3, and the bar chart is illustrated in Figure 3.

Analysis of the VHN values of each individual dual-cure bulk-fill RBC at two depths and in the self-cure condition showed that for BulkEZ, the VHN values were not significantly different among the two depths and self-cure conditions for either irradiation time ($p>0.05$); for HyperFIL and Injectafil, the VHN values were not significantly different between 5 mm and self-cure ($p>0.05$), but they were both significantly lower than the VHN values at 0.5 mm ($p<0.01$) for either irradiation time. For the light-cure RBCs, the VHN values at 0.5 mm were significantly higher than the VHN values at 5 mm for either irradiation time ($p<0.001$). The ratios of mean VHN at 5 mm to mean VHN at 0.5 mm for 20 seconds of irradiation were 0.88 for HyperFIL, 0.90 for Injectafil, 0.96 for BulkEZ, 0.64 for FBF, 0.55 for FOBF, and 0.57 for Z250; the ratios for 40 seconds of irradiation were 0.86 for HyperFIL, 0.87 for Injecta-



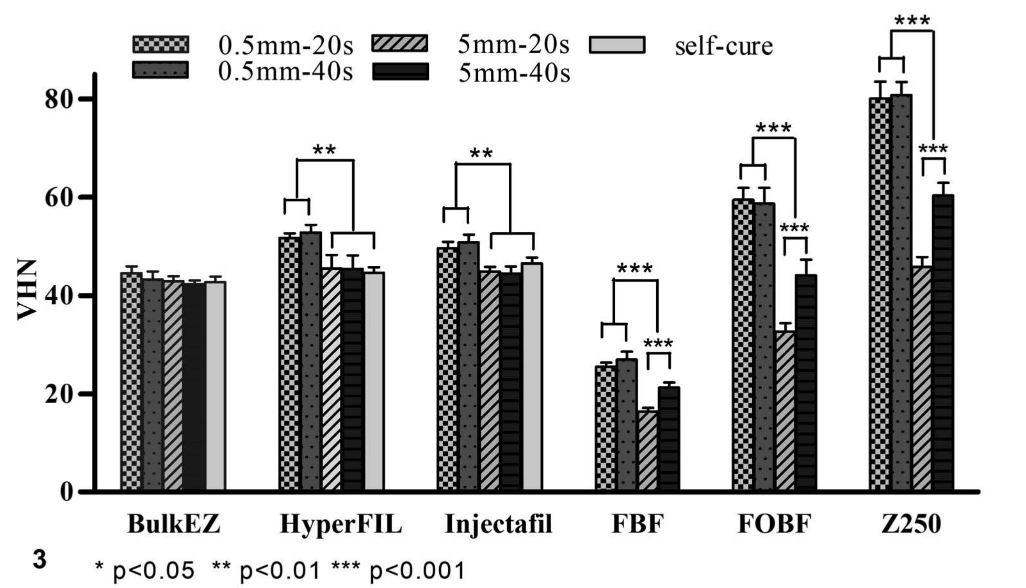


Figure 2. *DC% bar chart for all test conditions.*
Figure 3. *VHN bar chart for all test conditions.*

Table 3: *VHN Mean (Standard Deviation) for All Test Conditions*

	Composite and Irradiation Time											
	BulkEZ		HyperFIL		Injectafil		FBF		FOBF		Z250	
	20 s	40 s	20 s	40 s	20 s	40 s	20 s	40 s	20 s	40 s	20 s	40 s
Depth, mm												
0.5	44.53 (1.44)	43.25 (1.65)	51.72 (0.95)	52.87 (1.50	49.63 (1.31)	50.85 (1.61)	25.46 (0.87)	27.01 (1.59)	59.48 (2.45)	58.70 (3.20)	80.09 (3.50)	80.78 (2.70)
5	42.92 (1.01)	42.36 (0.77)	45.42 (2.73)	45.47 (2.76)	44.90 (1.00)	44.45 (1.52)	16.38 (0.76)	21.25 (1.02)	32.66 (1.69)	44.12 (3.21)	45.84 (1.99)	60.38 (2.60)
Self-cure	42.79 (1.12)		44.69 (1.09)		46.52 (1.24)		N/A		N/A		N/A	

Abbreviations: FBF, Filtek Bulk Fill Flowable; FOBF, Filtek One Bulk Fill; Z250, incremental Filtek Z250; N/A, not available.

Table 4: *Analysis Results for Pearson Correlation Between Mean DC and VHN for Six RBCs (α=0.05)*

	Composite					
	BulkEZ	HyperFIL	Injectafil	FBFF	FOBF	Z250
Pearson *r*	0.9257	0.9382	0.9596	0.9712	0.9912	0.9999
R square	0.857	0.8803	0.9208	0.9433	0.9825	0.9999
P value	0.012	0.0091	0.0048	0.0144	0.0044	< 0.0001
Correlation significant	Yes	Yes	Yes	Yes	Yes	Yes

Abbreviations: RBCs, resin-based composites; FBF, Filtek Bulk Fill Flowable; FOBF, Filtek One Bulk Fill; Z250, incremental Filtek Z250.

fil, 0.98 for BulkEZ, 0.77 for FBF, 0.75 for FOBF, and 0.75 for Z250.

Comparisons of the VHN values between 20 seconds and 40 seconds of irradiation times at the same depth for each individual RBC showed that the two different irradiation times did not generate a significant difference for the three dual-cure bulk-fill RBCs at either depth ($p>0.05$) or for the three light-cure RBCs at the 0.5-mm depth ($p>0.05$); however, 40 seconds of light irradiation generated significantly higher VHN values compared with 20 seconds of light irradiation for FBF, FOBF, and Z250 at the 5-mm depth ($p<0.0001$).

Correlations Between DC and VHN

The mean DC and VHN values for different test conditions for each individual RBC were subjected to Pearson correlation analysis. The results are summarized in Table 4. The DC-VHN correlations for all five test conditions of dual-cure bulk-fill RBCs and all four test conditions of light-cure RBCs are plotted in Figure 4.

All six RBCs showed good correlation between DC and VHN values with the Pearson correlation coefficient $r>0.9$. The three dual-cure bulk-fill RBCs showed similar DC-VHN correlation patterns–high DC range (68%-80%) and medium VHN range (40-53); the three light-cure RBCs showed similar lower DC ranges (40%-63%); Z250 showed the highest VHN range (45-81), FOBF showed a medium VHN range (32-60), and FBF showed the lowest VHN range (16-27).

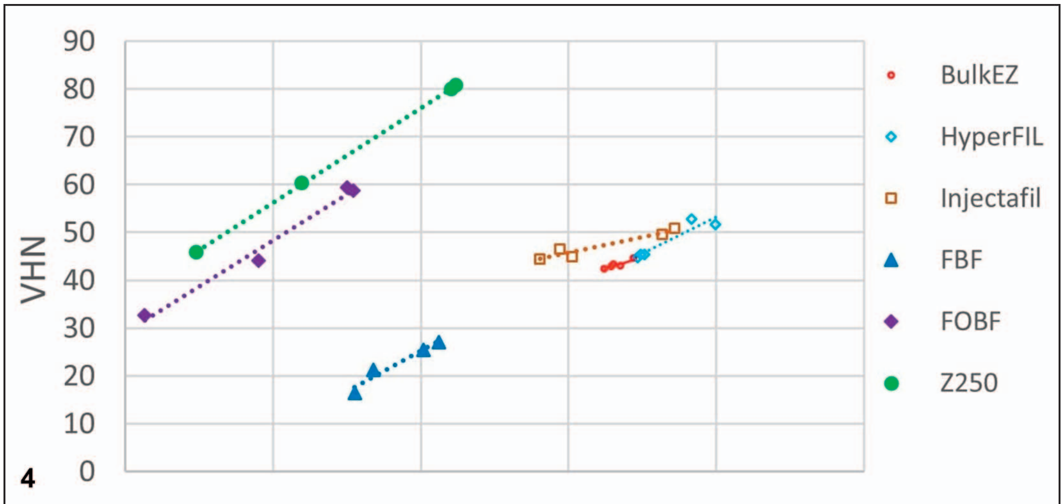
SEM Images

The SEM images of all six RBCs at 2000× magnification are shown in Figure 5: All three dual-cure bulk-fill RBCs contained irregularly shaped fillers of a wide range of sizes (from sub-micron to a few microns for HyperFIL and up to 10 μm or higher for Injectafil and BulkEZ). FBF and Z250 contained spherical-shaped fillers from sub-microns to a few microns. FOBF contained nanometer-sized fillers and filler clusters of a few microns.

DISCUSSION

Limited research data have been published on the polymerization and mechanical properties of the new dual-cure bulk-fill RBCs. In the present study, the DC and VHN values of three new dual-cure bulk-fill dental restorative RBCs (BulkEZ, HyperFIL, and Injectafil) were evaluated in comparison with three light-cure RBCs (bulk-fill RBCs FBF and FOBF and traditional incremental RBC Z250). The results showed that the DC and VHN values of the six RBCs were material related; therefore, our first null hypothesis was rejected. The DC and VHN values were significantly lower at the 5 mm depth than at the 0.5 mm depth for five RBCs (with the exception of BulkEZ); therefore, our second null hypothesis was largely rejected. Two different light irradiation times produced significantly different DC and VHN values for some test conditions (FOBF and Z250 at the 5 mm depth); therefore, our third null hypothesis was partially rejected.

A major difference between bulk-fill RBCs and conventional RBCs is their depth of cure. As a conventional RBC, Z250 has to be incrementally placed in layers of 2 mm in order to allow sufficient photo-polymerization. The manufacturers claim that the light-cure bulk-fill RBCs FBF and FOBF achieve an improved depth of cure of 4-5 mm. The manufacturers of the three dual-cure bulk-fill RBCs claim that they have unlimited depth of cure. A commonly accepted criteria for determining adequate depth of cure is the 80% bottom/top ratio for DC or hardness.[30–32] In the present study, the data at 0.5 mm could be treated as top surface measurements and the data at 5 mm could be treated as bottom surface measurements. The 5 mm/0.5 mm ratios for DC and VHN were calculated, and similar 80% ratio criteria were used to determine the efficiency of polymerization at the 5-mm depth. The results partially support the claims for the six RBCs. For Z250, the DC and VHN values at 5 mm were <80% of the values at 0.5



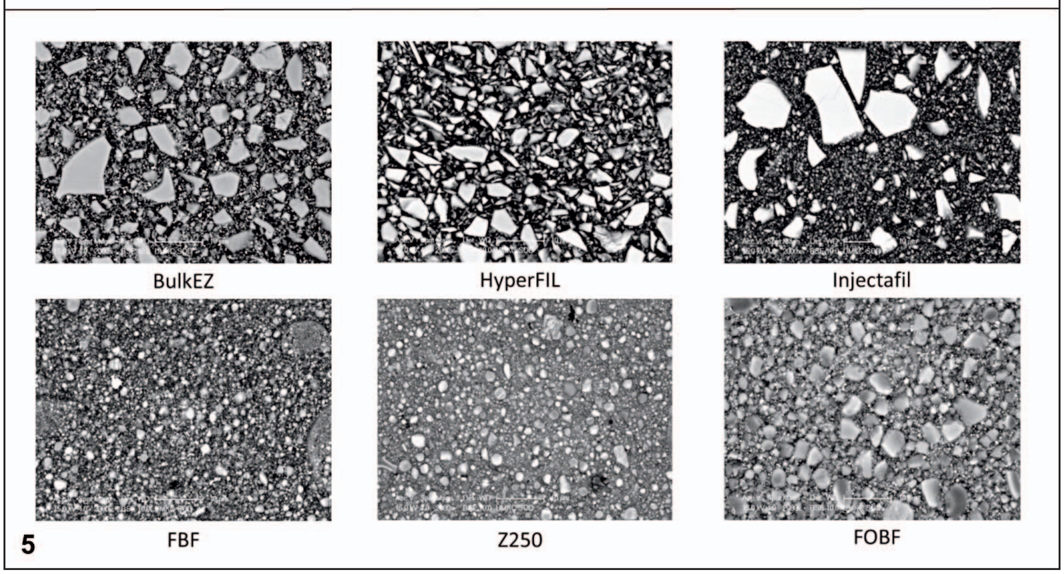


Figure 4. *DC%-VHN correlation chart for six RBCs.*
Figure 5. *SEM images of the six RBCs at 2000× magnification.*

mm for the manufacturer recommended 20 seconds of light irradiation, indicating insufficient polymerization at the 5 mm specimen depth. For FBF, the DC at 5 mm was >90% of the DC at 0.5 mm, but the VHN value at 5 mm was <80% of the values at 0.5 mm for the manufacturer recommended 20 seconds of light irradiation. Similar DC and VHN findings at the 4 mm depth for FBF were reported in previous studies.[33,34] For FOBF, the DC and VHN values at 5 mm were all <80% of the values at 0.5 mm, indicating insufficient polymerization and thus lower hardness at the 5 mm depth and raising questions about the manufacturer's claim of up to 5 mm depth of cure. For the three dual-cure bulk-fill RBCs, the DC and VHN values at the 5 mm depth were the same as the self-cure conditions; both were >5% of the DC and VHN values at the 0.5 mm depth, indicating sufficient polymerization in specimen depth up to 5 mm from chemical curing. Light curing slightly improved the DC and VHN values for HyperFIL and Injectafil in the top layers. BulkEZ showed the same DC and VHN values among all test conditions, indicating that chemical curing is its dominant polymerization process. The results suggest that the three dual-cure bulk-fill RBCs can achieve unlimited depth of cure thanks to their efficient and depth-independent chemical-curing process.

Two clinically relevant light irradiation times—20 seconds and 40 seconds—were assessed for the six RBCs. The results showed that an extended light irradiation time of 40 seconds did not generate any difference in DC or VHN values for the three dual-cure bulk-fill RBCs at either depth. The explanation could be that the three dual-cure bulk-fill RBCs mainly rely on chemical curing for polymerization and therefore light irradiation time did not affect their DC or VHN values. For the three light-cure RBCs, the two light irradiation times did not generate any difference in DC or VHN values at the 0.5-mm depth; however, 40 seconds of irradiation generated significantly higher DC values for FOBF and Z250 (but not for FBF) and significantly higher VHN values for FBF, FOBF, and Z250 at the 5 mm depth compared with 20 seconds of irradiation. Similar findings were reported by a previous study for FBF and Z250.[34] The explanation could be that in the top layers, where the curing light can reach with sufficient power, 20 seconds of irradiation time was enough to achieve maximum polymerization for those materials; while in the deep layers up to 5 mm, where the curing light was greatly attenuated, extended light irradiation time could help achieve better polymerization and greater hardness for some light-cure RBCs.

Previous studies have indicated that the loading, size and shape of filler particles of light-cure RBCs significantly affected their mechanical properties and light transmittance characteristics, which consequently affected the DC and depth of cure of the RBCs.[35,36] Reduced filler loading can help increase light transmission and depth of cure for bulk-fill light-cure RBCs but at the same time can also weaken their mechanical properties.[37,38] In the present study, FBF, which had the lowest filler loading of 64.5 wt%, showed good depth of cure (>90% 5 mm/0.5 mm DC ratio) but the lowest microhardness level (VHN<30), while the conventional Z250, which had the highest filler loading of 82 wt%, showed the highest microhardness level at 0.5 mm (VHN ~80) but poor depth of cure (<80% 5 mm/0.5 mm DC ratio).

On the other hand, dual-cure bulk-fill RBCs utilize chemical curing mechanisms to achieve depth of cure and therefore are not constrained by the low filler-loading design. With similar filler loading (>70 wt%), the three dual-cure bulk-fill RBCs showed much higher DC values than FOBF. FOBF showed the lowest DC, probably for two reasons: 1) FOBF contains high molecular weight monomers (aromatic dimethacrylate [AUDMA], urethane dimethacrylate [UDMA], and 1,12-dodecanediol dimethacrylate [DDDMA]) with decreased number of reactive carbon=carbon groups in its resin, which consequently results in lower DC[39]; 2) the high viscosity of FOBF could hinder the mobility of its reactive monomer species and reduce the conversion.[40] Compared with FOBF, the three dual-cure bulk-fill RBCs showed lower VHN values at 0.5 mm but higher VHN values at 5 mm, with a smaller difference in VHN values between the two depths. Material viscosity has been shown to be an important factor on the polymerization and clinical performance of RBCs.[41,42] Compared with high-viscosity packable RBCs, low-viscosity bulk-fill RBCs provide better adaptation to the cavity wall and potentially reduce marginal gaps and microleakage.[43,44] With relatively low viscosity and high flowability, the three dual-cure bulk-fill RBCs eliminate the need for flowable liners.

The results from our previous study showed that the polymerization kinetics of HyperFIL and Injectafil were depth dependent—the top layers were characterized by faster light-activated polymerization and the deep layers were characterized by slower chemical-activated polymerization, while the polymerization of BulkEZ was characterized by moderately paced polymerization across the entire depth.[45] These kinetics results are consistent with the DC and VHN results in the present study in which BulkEZ showed depth-independent properties while HyperFIL and Injectafil showed depth-dependent properties. It has been well documented that the rate of polymerization of a dental composite often affects its polymerization contraction stress.[46] The depth-independent, moderately paced polymerization kinetics of BulkEZ is likely to result in more uniform and moderate shrinkage stress; while the depth-dependent polymerization kinetics properties of HyperFIL and Injectafil are likely to result in higher shrinkage stress in the fast-cured top layers and lower shrinkage stress in the slowly cured deep layers. The difference in DC and VHN profiles from the present study and the difference in polymerization kinetics profiles from our previous study for the three dual-cure bulk-fill RBCs may have interesting implications for their physical properties and clinical performance.

Future studies are needed to experimentally measure other polymerization and mechanical properties, such as shrinkage volume and stress of these new dual-cure bulk-fill RBCs and to better understand their overall properties (eg, shrinkage, marginal gaps, microleakage). Research to evaluate the long-term clinical performance of these dual-cure bulk-fill RBCs is also desired.

CONCLUSION

The three dual-cure bulk-fill RBCs can achieve unlimited depth of cure with high degree of conversion and medium microhardness levels. Among them, BulkEZ showed uniformity in degree of conversion and microhardness levels across all depths, while HyperFIL and Injectafil showed slightly higher degree of conversion and microhardness levels in top layers than in deep layers. The difference may have interesting implications for their physical properties and clinical performances.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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