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Characterization of Inorganic Filler Content, Mechanical Properties, and Light Transmission of Bulk-fill Resin Composites

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Clinical Relevance

Light attenuation at greater depths did not influence flexural strength of bulk-fill composites, which can be applied in a 4-mm thickness. Some of these materials present inferior mechanical properties compared with conventional composite.

SUMMARY

Objectives: The aims of this study were to characterize inorganic content (IC), light transmission (LT), biaxial flexural strength (BFS), and flexural modulus (FM) of one conventional (layered) and four bulk-fill composites at different depths.

Methods: Bulk-fill composites tested were Surefil SDR flow (SDR), Filtek Bulk Fill (FBF), Tetric EvoCeram Bulk Fill (TEC), and EverX Posterior (EXP). Herculite Classic (HER) was used as a control. Energy dispersive x-ray analysis and scanning electron microscopy were used to characterize filler particle composition and morphology. The LT through different composite thicknesses (1, 2, 3, and 4 mm) was measured using a laboratory-grade spectral radiometer system (n=5). For the BFS and FM tests, sets of eight stacked composite discs (0.5-mm thick) were prepared simulating bulk filling of a 4-mm-thick increment (n=8).

Results: SDR demonstrated larger, irregular particles than those observed in TEC or HER. Filler particles in FBF were spherical, while those in EXP were composed of fiberglass strands. The LT decreased with increased composite thickness for all materials. Bulk-fill composites allowed higher LT than the HER. Furthermore, HER proved to be the unique material, having lower BFS values at deeper

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regions. SDR, FBF, and TEC bulk-fill composites presented reduced FM with increasing composite depth.

Conclusions: The bulk-fill composites investigated exhibited higher LT, independent of different filler content and characteristics. Although an increase in composite thickness reduced LT, the BFS of bulk-fill composites at deeper layers was not compromised.

INTRODUCTION

Development of dental resin-based composites began with use of a bisphenol-A glycidyl methacrylate monomer (Bis-GMA) combined with glass filler particles created by Bowen in 1958.[1] Since that time, the composition of composite resins has evolved significantly. The basic formulation of composite resins is methacrylate monomeric mixtures and inorganic fillers coated with a silane coupling agent, along with photoinitiator systems that promote polymerization when the material is light activated, to form a highly cross-linked network with high mechanical properties.[2-4] Most improvements to composites have involved the inorganic fillers that have been reduced in size to produce materials with greater surface gloss retention and wear resistance.[3,5]

However, concerns related to aspects of the organic resin matrix remain, such as the extent of monomer conversion into polymer, which provides materials with high modulus and strength.[4,6] Also, polymerization shrinkage, caused by monomer approximation during the curing reaction, and the stress generated along with modulus development may negatively impact the clinical performance of bonded restorations.[7,8] Incremental filling techniques for composites have been suggested to minimize polymerization shrinkage stress and ensure efficient polymerization.[9-11] The average maximum thickness recommended for each increment of regular composites is 2 mm, and depending on the composite resin formulation, light irradiation is recommended to last from 10 to 40 seconds. Thus, restorations involving large cavity preparation volumes are time consuming for the operator and inconvenient for the patient.[12]

Bulk-fill composites activated by light have recently been introduced, using new composite resin formulations. According to the manufacturers, these new materials enable depths of cure up to 4 or 5 mm with minimal polymerization shrinkage stress. Successful resin composite restorations require an efficient polymerization process to enhance mechanical properties and biocompatibility and offer the potential for long-term success.[6] One approach to improve curing depth of bulk-fill composites is to increase material translucency, thereby allowing more light to pass through to deeper levels of the material, which provides a more uniform monomer conversion with depth.[13]

The optical properties of resin composites and their light-activated polymerization reactions are interdependent: a higher radiant exposure yields a higher degree of conversion, which leads to enhanced physical properties.[14] Light transmission (LT) is affected by material composition. Filler particles hinder LT due to scattering, which is dependent on filler particle size and related to the incident wavelength of the curing light. The refractive indices of fillers and resin matrix in a composite, as well as the mismatch between them, also influence light refraction and, thus, light penetration depth. Other components, such as pigments and photoinitiators, absorb light and result in a decrease in depth of cure.[6,15,16]

There are differences among bulk-fill composites with respect to filler loading and resin matrix composition. Some products have a flowable consistency, while other materials have a high filler content or feature glass fibers for reinforcement and are thus more viscous.[17] Consequently, the mechanical properties of these materials are expected to present variations among brands. Because fracture of composite resin restorations remains a major cause of clinical failure,[18,19] laboratory evaluation of composite properties and the factors influencing their physical behaviour are needed to better predict the clinical outcomes of direct restorations. Specifically, flexural strength and modulus of composites have been shown to correlate with clinical performance.[20] Furthermore, LT through composites plays an important role in the polymerization process and, thus, in determining the final mechanical properties of restorations.[19]

The purposes of this study were to characterize the morphology and composition of filler particles and their influence on LT and on the biaxial flexural strength (BFS) and flexural modulus (FM) of a variety of commercially available bulk-fill composites at selected depths. One conventional, layered, microhybrid composite was used as a control. The following hypotheses were tested: (1) differences in filler particle characteristics will be observed between bulk-fill and conventional composites; (2) LT will be higher in bulk-fill composites compared with

Table 1: *Materials Evaluated and Respective Manufacturers' Information*

Abbreviation Used in Text	Brand Name	Manufacturer (Lot Number)	Matrix Composition*	Filler Type[a]	Filler Loading[a] (%, by Volume)	Shade
HER	Herculite Classic	Kerr Co, Orange, CA, USA (4009366)	Bis-GMA, TEGDMA	Borosilicate-aluminum glass	59	A2
SDR	Surefill SDR flow	Dentsply Caulk, Mildford, DE, USA (08153)	Modified UDMA, TEGDMA, EBPDMA	Barium-aluminofluoro-borosilicate glass, strontium-aluminofluoro-borosilicate glass	44	Universal
FBF	Filtek Bulk Fill	3M ESPE, St Paul, MN, USA (402919)	Bis-GMA, Bis-EMA, UDMA, TEGDMA, Procrylat resins	Zirconia/silica, ytterbium trifluoride	42.5	A2
TEC	Tetric EvoCeram Bulk Fill	Ivoclar Vivadent, AG, Schaan, Liechtenstein (R04686)	Bis-GMA, UDMA	Barium glass, ytterbium trifluoride, oxides and pre-polymers	60 (17% pre-polymers)	IVA
EXP	EverX Posterior	GC Corporation, Tokyo, Japan (1401152)	Bis-GMA, TEGDMA, PMMA	Hybrid filler fractions and E-glass fibers	57	Universal

Abbreviations: Bis-GMA, bisphenol-A diglycidyl ether dimethacrylate; Bis-EMA, ethoxylated bisphenol-A dimethacrylate; EPDMA, ethoxylated bisphenol-A dimethacrylate; PMMA, polymethyl methacrylate; TEGDMA, triethylenegycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] *Information supplied by manufacturer.*

a conventional, layered composite, and (3) there will be no significant difference in mechanical properties among the bulk-fill composites at similar depths, while properties will be reduced with increasing composite depth.

METHODS AND MATERIALS

Five resin-based composites were investigated: one conventional, incrementally layered material used as a control (Herculite Classic [HER]); two high-viscosity, bulk-fill composites (Tetric EvoCeram Bulk Fill [TEC] and EverX Posterior [EXP]); and two flowable bulk-fill composites (Surefil SDR flow [SDR] and Filtek Bulk Fill [FBF]). The compositions, lot numbers, and manufacturer information for these products are presented in Table 1.

Filler Content and Characterization

Approximately 1 g of unpolymerized composite was washed in 6 mL of acetone (99.5%, Merck KGA, Darmstadt, Germany) and centrifuged at 1000 rpm for 5 minutes (Excelsa, model 206, FANEM, São Paulo, Brazil). This procedure was repeated until the entire organic matrix was dissolved, as evidenced by clarity of the supernatant fluid.[19] Chloroform (99.8%, Merck KGA) was then used in the same manner. The remaining content of fillers was then immersed in 6 mL of absolute ethanol (Merck KGA) for 24 hours followed by drying at 37°C in an incubator (FANEM). The recovered filler particles were then placed on plastic stubs and sputter-coated with carbon (MED 010 Baltec, Balzers, Liechtenstein) before examination using energy-dispersive x-ray (EDX) spectrometry analysis, or placed on metallic stubs and sputter-coated with gold (MED 010 Baltec) before scanning electron microscopy (SEM) observation. EDX analysis (Vantage, NORAN Instruments, Middleton, WI, USA) coupled to a scanning electron microscope (JEOL, JSM-5600LV, Tokyo, Japan) was performed to identify the elemental composition of the recovered insoluble filler particles. Each spectrum was acquired for 100 seconds (voltage 15 kV, dead time 20% to 25%, working distance 20 mm). Images showing the identified chemical elements and their relative concentration were obtained from five different analyses of each material at different locations on a stub.

For filler particle morphologic characterization, specimens were examined using SEM (voltage 15 kV, beam width 25–30 nm, working distance 10–15 mm) at 50×, 1000×, and 5000× magnifications. Representative images at different magnifications were obtained for each material and were used for qualitative analysis and particle-size comparison using software analysis of the recorded images (ImageJ, 1.6.0_24, National Institutes of Health, Bethesda, MD, USA).

Light Transmission Through Cured Composite

Composite discs (n=5) of each material at four different thicknesses were fabricated to evaluate LT. Silicon molds (6 mm in diameter) were used to

fabricate discs with thicknesses of 1, 2, 3, and 4 mm. Each material was light-activated using a polywave light-emitting diode curing unit (LCU; VALO, Ultradent Products Inc, South Jordan, UT, USA) for the exposure duration recommended by the manufacturer (20 seconds for the bulk-fill composites and 40 seconds for the conventional composite) with the emitting end of the light source as close to the upper composite surface as possible without actually touching it.

The irradiance of the LCU and the light transmittance were determined using a laboratory-grade spectral radiometer (USB 2000, Ocean Optics, Dunedin, FL, USA) attached to a 7.62-cm-diameter integrating sphere (CTSM-LSM-60-SF, Labsphere Inc, Sutton, NH, USA) associated with specific software (Spectra Suite version 5.1, Ocean Optics Inc, Dunedin, FL, USA). A mean value of 1153 mW/cm^2 was obtained for the total irradiance of LCU (100%; without interposing disc) between 350 and 550 nm. To measure LT, each composite disc was positioned between the integrating sphere aperture and the curing unit tip. The light source was positioned such that the tip remained parallel to the specimen surface and just slightly touched it.

Each spectrum was obtained during the first 5 seconds of light irradiation. The transmittance value for each sample was calculated as a percentage by dividing the irradiance value measured through each specimen by the average curing light irradiance with no interposing disc.

The data demonstrated asymmetrical distribution. Adjustments were performed using a generalized linear model considering the gamma distribution (asymmetric mode) according to a two-way design. Software (GENMOD, SAS/STAT 9.3, Cary, NC, USA) was applied to analyze the data. Multiple comparisons of the results among the different composite depths were performed using a feature of that software (DIFF). All statistical testing was performed at a pre-set alpha of 0.05.

Biaxial Flexural Strength and Modulus

Disc-shaped specimens (n=8) approximately 0.5 mm thick and 6.0 mm in diameter were fabricated using a set of eight Teflon molds that were stacked on each other. A metal device was used to hold the eight composite-filled Teflon molds together. For each 0.5-mm-thick specimen, an acetate strip was positioned on the bench top, and the empty mold was placed on top. The mold was slightly overfilled with uncured composite paste and then covered with a second acetate strip. Vertical pressure was applied to force the material to conform to the confines of the mold dimensions and to extrude excess material. The filled Teflon mold was then placed into the holding jig, using the two vertical metal guides to precisely position it. A second mold was then placed on top of the acetate strip of the previously filled specimen and filled with composite; another acetate sheet was then placed, and vertical pressure was applied. This process was continued until a total of eight such molds had been stacked, with a total thickness of 4 mm, but such that the cylinder could be disassembled after light curing from the top into separate 0.5-mm-thick increments. Once all the wafers were stacked, the composite stack was photocured using the LCU with the distal end of the light guide touching the acetate-covered surface of the top-most wafer. This process is similar to that performed in other work.[21] Specimen fabrication was performed in a light-proof room with a controlled temperature: 21°C.

After irradiation, the composite specimens were removed from the molds and their dimensions were measured using a digital micrometer (six-digit precision, MDC-Lite, Mitutoyo Corporation, Kanagawa, Japan). The discs were stored in the dark, in an incubator maintained at 37°C ± 1°C at a relative humidity for one week prior to BFS determination.

For BFS testing, each disc was individually placed into a custom-made jig and subjected to the piston-on-ring biaxial test,[21,22] using a universal testing machine (Model 5844, Instron Corporation, Canton, MA, USA), at a crosshead speed of 1.27 mm/min, until failure. The maximum load at failure was recorded for each specimen, and the flexural modulus (FM) was determined from the linear portion of each stress/strain curve. The following formula was used to obtain the BFS data:

$$\mathrm{BFS} = -0.238 \times 7\mathrm{P}(\mathrm{X} - \mathrm{Y})/\mathrm{b}^2,$$

where BFS is the maximum tensile stress (MPa), P is the total load at fracture (N), b is the specimen thickness (mm) and

$$\mathrm{X} = (1+v)ln(r2/r3)^2 + [(1-v)/2](r2/r3)^2,$$

$$\mathrm{Y} = (1+v)[1+ln(r1/r3)^2] + [(1-v)(r1/r3)^2],$$

where v is Poisson's ratio (0.25), $r1$ is the radius of the support circle (mm), $r2$ is the radius of the loaded area (mm), and $r3$ is the radius of the disc (mm).

The BFS and FM values were calculated using software (SRS Biaxial Testing Software, Instron

Corp. and were expressed in megapascals and gigapascals, respectively. Exploratory analysis of data suggested logarithmic transformation with base 10 for both BFS and FM data. Data were subjected to split-plot two-way (material and depth) analysis of variance using SAS/STAT 9.3 software. Tukey post hoc tests were performed to detect significant differences among the groups using a preset alpha of 0.05.

RESULTS

Filler Content and Characterization

The elemental composition of HER revealed the presence of aluminium, silicon, and barium (Figure 1A). The SEM micrograph showed irregularly shaped particles ranging from 0.5 to 2.2 μm in diameter (Figures 1B,C). The inorganic elements in SDR were found to include aluminium, silicon, barium, and a minor amount of fluoride (Figure 2A). This material consisted mainly of irregular particles of two distinct sizes: larger particles approximately 20 μm and smaller particles ranging from 0.5 to 1 μm (Figures 2B,C). The TEC composite had a composition and morphology similar to that of HER, consisting of aluminium, silicon, and barium with particles ranging in size from 0.4 to 2.2 μm (Figures 3A through C). EDX analysis revealed that FBF contained aluminium, silicon, and zirconium (Figure 4A), consisting only of spherical particles with diameters ranging from 0.1 to 4.0 μm (Figures 4B,C). The filler particles in EXP were basically fiberglass consisting of aluminium, silicon, barium, fluoride, and calcium (Figure 5A) with lengths up to 1 mm and a diameter of approximately 15 μm (Figures 5B,C). However, small particles with a diameter of 1 μm were also observed.

Light Transmission Through Cured Composite

Table 2 presents the mean values for the percentage of light passing through each composite. Statistical results indicated that both material ($p<0.0001$) and depth ($p<0.0001$) significantly influenced LT. The interaction between factors was statistically significant ($p<0.0001$). In general, the HER material had a lower LT, while SDR presented higher LT for all depths evaluated. However, all of the composites exhibited a similar trend for light transmittance to decrease with respect to increase in sample depth.

Biaxial Flexural Strength and Modulus

Average BFS and FM values for the composites are presented in Tables 3 and 4, respectively. Statistical analyses indicated that both material ($p<0.0001$) and depth ($p=0.0022$) significantly influenced BFS results. Regarding factorial model, no significant interaction between factors was identified ($p=0.2741$). Considering this global analysis regarding the depths of all materials together, 0.5-mm, 1.5-mm, and 2.0-mm depths had significantly higher BFS than the 4.0-mm depth. Although the factorial model did not present significant interaction between factors (materials and depths), when an interaction slice was performed it detected a significant difference among depths only for HER ($p<0.0001$). For this conventional material, the BFS at a depth of 0.5 to 2 mm was higher compared with a depth 4 mm. The bulk-fill composites did not show significant difference among depths ($p<0.05$) (Table 3). When data obtained at different depths for a given material were pooled, significant differences were found among materials. In general, HER, SDR, and FBF demonstrated higher BFS values, followed by EXP and TEC, which had the lowest values.

For FM data, statistically significant differences were also observed among the materials ($p<0.0001$) and depths (p=0.0002), again with no significant interaction between them ($p=0.0865$). In this global factorial model and considering all materials together, FM was significantly higher at 0.5-mm and 2.0-mm depths compared with the 4.0-mm depth. Meanwhile, the slice of interaction demonstrated that there were significant differences in FM among depths for SDR, FBF, and TEC ($p=0.0004$, $p=0.0116$, and $p=0.0048$, respectively) (Table 4). Regarding the comparison of materials, HER and SDR demonstrated the highest and lowest moduli, respectively. The products EXP, FBF, and TEC showed intermediate FM values, with EXP demonstrating a higher value than both FBF and TEC, which had moduli that were not statistically different.

DISCUSSION

The first hypothesis, stating that there will be qualitative differences in filler particle characteristics between bulk-fill and conventional composites was observed in this investigation. While the shapes of the filler particles in SDR, FBF, and, EXP (Figures 2, 4, and 5) were different and larger than the particles in the HER composite (Figure 1), particles in the TEC composite (Figure 3) were of similar shape and size to those in HER. Furthermore, LT through the composites was similar for all materials for a given depth, although SDR exhibited higher LT, and HER had the lowest LT percentages of all materials (Table 2). Thus, the second hypoth-

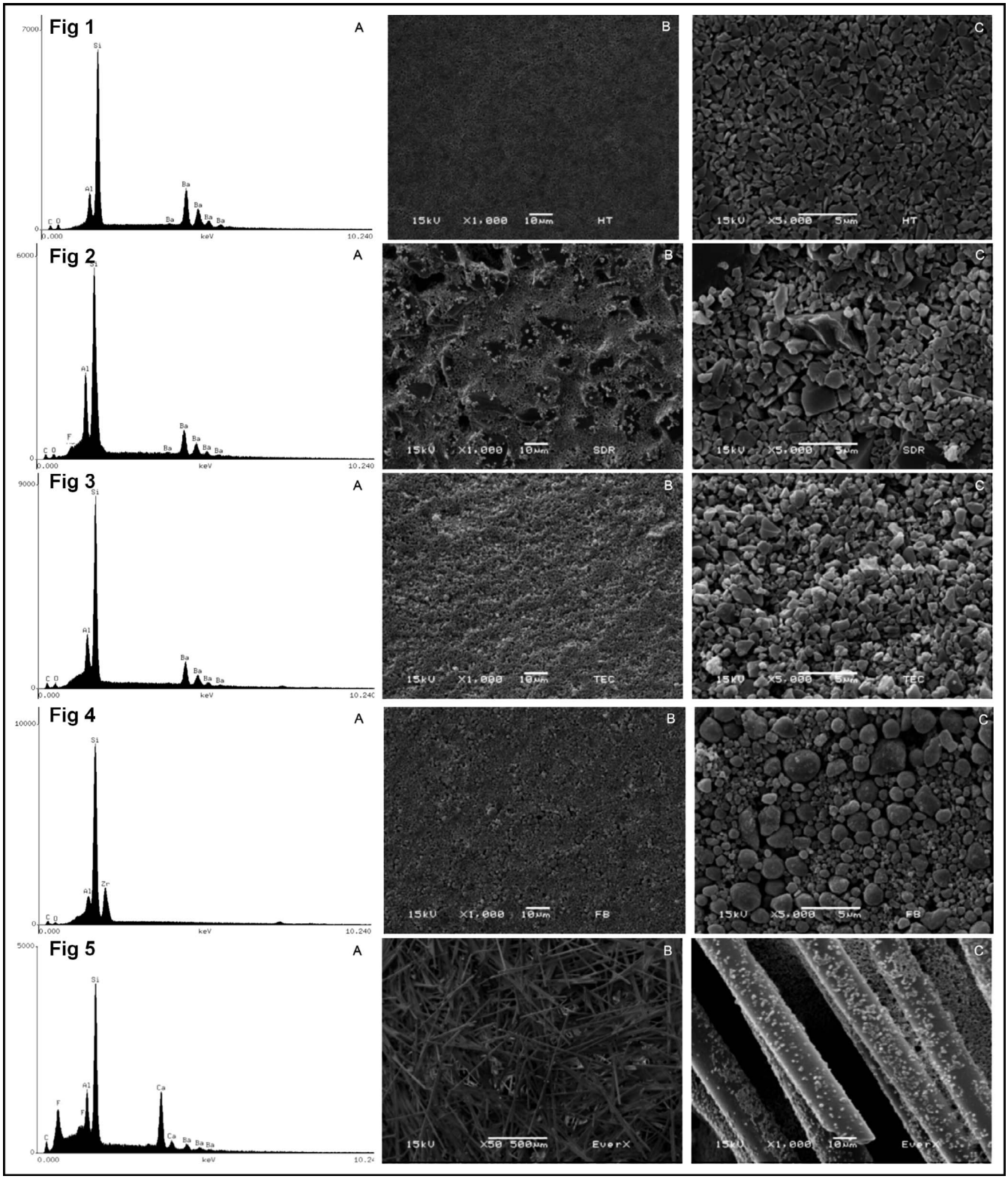

Figure 1. *Elements identified by EDX analysis for HER (A) and SEM micrographs: original magnification 1000× (B) and 5000× (C).*
Figure 2. *Elements identified by EDX analysis for SDR (A) and SEM micrographs: original magnification 1000× (B) and 5000× (C).*
Figure 3. *Elements identified by EDX analysis for TEC (A) and SEM micrographs: original magnification 1000× (B) and 5000× (C).*
Figure 4. *Elements identified by EDX analysis for FBF (A) and SEM micrographs: original magnification 1000× (B) and 5000× (C).*
Figure 5. *Elements identified by EDX analysis for EXP (A) and SEM micrographs: original magnification 50× (B) and 1000× (C).*

Table 2: *Mean (Standard Deviation) for Light Transmission (%)*[a]

Depth (mm)	Material				
	Layered	Bulk-Fill			
		Flowable		Viscous Paste	
	HER	SDR	FBF	TEC	EXP
1	21.1 (0.4) Da	38.6 (1.1) Aa	31.3 (0.5) Ba	27.3 (0.6) Ca	27.1 (4.7) Ca
2	11.1 (0.4) Db	23.3 (0.1) Ab	16.4 (1.2) Bb	15.2 (0.6) Cb	16.5 (0.9) Bb
3	5.5 (0.3) Cc	15.5 (0.3) Ac	8.5 (0.2) Bc	8.9 (0.3) Bc	8.7 (0.3) Bc
4	2.5 (0.1) Cd	9.1 (0.4) Ad	4.3 (0.3) Bd	4.4 (0.1) Bd	4.9 (0.5) Bd

Abbreviations: EXP, EverX Posterior; FBF, Filtek Bulk Fill; HER, Herculite Classic; SDR, Surefil SDR flow; TEC, Tetric EvoCeram Bulk Fill.
[a] Means (n=5) followed by the same letter (uppercase compare columns (materials), lowercase compare rows (depths)) are not statistically different (p>0.05).

Table 3: *Mean (Standard Deviation) Biaxial Flexural Strength (MPa) With Composite Depth* [a]

Depth (mm)	Material					Tukey
	Layered	Bulk-Fiill				
		Flowable		Viscous Paste		
	HER	SDR	FBF	TEC	EXP	
0.5	173.9 (31.7) *a*	148.8 (12.3) *a*	171.3 (24.8) *a*	76.3 (9.6) *a*	103.4 (8.0) *a*	**a**
1.0	165.3 (35.9) *ab*	149.5 (22.4) *a*	171.9 (17.1) *a*	77.3 (14.4) *a*	102.6 (14.0) *a*	**ab**
1.5	175 (31.4) *a*	152.3 (14.8) *a*	170.5 (23.8) *a*	78.5 (12.8) *a*	106.8 (10.7) *a*	**a**
2.0	167.6 (31.8) *ab*	148.9 (13.3) *a*	169.5 (22.5) *a*	79.7 (8.7) *a*	107.0 (7.6) *a*	**a**
2.5	141.5 (27.7) *abc*	151.9 (25.1) *a*	157.0 (28.3) *a*	79.4 (3.8) *a*	103.5 (14.2) *a*	**ab**
3.0	137.0 (26.4) *bc*	148.4 (9.9) *a*	157.8 (24.8) *a*	77.2 (11.0) *a*	104.3 (9.8) *a*	**ab**
3.5	147.7 (33.2) *abc*	152.2 (15.7) *a*	151.0 (27.2) *a*	75.1 (8.2) *a*	103.9 (14.3) *a*	**ab**
4.0	124.2 (22.9) *c*	146.0 (16.2) *a*	143.2 (22.7) *a*	76.2 (11.0) *a*	104.2 (12.0) *a*	**b**
Tukey	**A**	**A**	**A**	**C**	**B**	

Abbreviations: EXP, EverX Posterior; FBF, Filtek Bulk Fill; HER, Herculite Classic; SDR, Surefil SDR flow; TEC, Tetric EvoCeram Bulk Fill.
[a] Means (n=8) followed by the same bold uppercase letter (columns comparing materials) are not statistically different (p>0.05). Bold lowercase letters (outside the table) are related to global factorial model (all materials together) that compares depths (rows). The italic lowercase letters (inside the table) represent the slice of interaction and also compare depths, but within the same material.

Table 4: *Mean (Standard Deviation) Flexural Modulus (GPa) With Composite Depth* [a]

Depth (mm)	Material					Tukey
	Layered	Bulk-Fill				
		Flowable		Viscous Paste		
	HER	SDR	FBF	TEC	EXP	
0.5	5.0 (1.4) *a*	3.2 (0.5) *a*	3.8 (0.7) *a*	3.7 (0.5) *ab*	4.6 (1.0) *a*	**a**
1.0	4.9 (0.5) *a*	2.6 (0.3) *ab*	3.8 (0.7) *a*	3.9 (1.0) *ab*	4.5 (1.2) *a*	**abc**
1.5	5.1 (1.2) *a*	2.6 (0.5) *ab*	3.4 (0.3) *ab*	4.2 (1.1) *a*	4.4 (0.9) *a*	**abc**
2.0	5.3 (1.3) *a*	2.5 (0.5) *ab*	3.9 (0.9) *a*	3.7 (0.2) *ab*	4.5 (0.6) *a*	**ab**
2.5	4.9 (0.8) *a*	2.4 (0.3) *ab*	3.0 (0.7) *ab*	3.5 (1.0) *ab*	4.7 (1.0) *a*	**abc**
3.0	5.2 (0.8) *a*	2.2 (0.2) *b*	3.1 (1.4) *ab*	3.3 (0.8) *ab*	4.5 (1.1) *a*	**bc**
3.5	5.3 (1.1) *a*	2.2 (0.5) *b*	2.9 (0.8) *ab*	3.1 (0.9) *ab*	4.6 (0.7) *a*	**bc**
4.0	5.2 (0.5) *a*	2.3 (0.2) *ab*	2.7 (0.3) *b*	2.8 (0.5) *b*	4.5 (1.1) *a*	**c**
Tukey	**A**	**D**	**C**	**C**	**B**	

Abbreviations: EXP, EverX Posterior; FBF, Filtek Bulk Fill; HER, Herculite Classic; SDR, Surefil SDR flow; TEC, Tetric EvoCeram Bulk Fill.
[a] Means (n=8) followed by the same bold uppercase letter (columns comparing materials) are not statistically different (p>0.05). Bold lowercase letters (outside the table) are related to global factorial model (all materials together) that compares depths (rows). The italic lowercase letters (inside the table) represent the slice of interaction and also compare depths, but within the same material.

esis, that LT will be higher in bulk-fill composites compared with a conventional, layered composite was accepted.

Light transmission through a resin composite depends on light reflection, scattering, and absorption, which vary according to the material composition. Filler particles with diameters approaching half the wavelength of light used for curing increase light scattering, and thus light transmittance tends to increase with increasing filler size because scattering is decreased. Studies have also shown that increasing the size of silica particles reduces the extent of polymerization at greater depths for experimental and commercial composites.[6,23,24] Furthermore, not only the size of the particles but also the amount of filler loading influence LT. Higher filler content tends to reduce LT due to the increased probability of light refraction at the interfaces between the filler particles and the resin due to differences in their refractive indices.[6,13]

The lower filler loading for SDR explains its higher LT compared with that of HER (Table 1) as well as the larger particle size (approximately 20 μm) of the fillers (Figures 1 and 3). FBF also has low filler content, yet the presence of zirconium (Figure 2) may influence the LT behavior because this material has a higher refractive index, which explains the similar results compared with TEC and EXP.[25] Both TEC and EXP composites also exhibited higher LT than the conventional, layered control material: HER. Given that the inorganic content and morphologic characteristics of the fillers in TEC and HER were very similar, this finding may be attributed to the different compositions and shapes of the filler particles and the different monomers used in these composites.[6] Meanwhile, the higher transmittance of EXP, despite its high filler loading, may be due to the fiberglass filler, which may be effective in transmitting light inside the material.[26]

A study compared light transmittance through nanohybrid, flowable, and bulk-fill composites at 2-mm, 4-mm, and, 6-mm incremental thickness.[13] Bulk-fill composites used in that study, including several used in the current project (SDR, FBF, and TEC) demonstrated higher translucency than regular composites resin. In that study, measurements were made during real-time polymerization, different from this study, which evaluated LT through pre-polymerized composite cylinders. The authors reported that light transmittance increased as the polymerization reaction progressed. As polymer cross-linking starts, the density and refractive index of the polymer matrix increases, approaching the refractive index of the fillers, resulting in a reduction of scattering and an increase in LT.[13] It is therefore possible that LT values may be overestimated in the present study due to an increase in the effect of using pre-polymerized composites.

The third hypothesis, that there will be no significant difference in mechanical properties among bulk-fill composites at similar depths, while properties will be reduced with increasing composite depth, was partially accepted. For the BFS test, when the global analysis was considered, all materials had a decrease in flexural strength at the 4-mm layer. However, when interaction slice was performed, this effect was only observed in the HER conventional composite (Table 3). For the FM test, reduced values were also observed with increasing composite depth in global analysis. This was detected mainly in SDR, FBF and TEC, which also had significantly lower FM when materials were compared (Table 4).

Resin composite viscosity has been shown to be an important parameter affecting the polymerization kinetics and final degree of conversion of dimethacrylate monomers because it influences monomer mobility and reactivity. In turn, the rheologic properties of composite resins depend on monomer composition and filler content.[4,27] In general, higher BFS values indicate higher monomer conversion. In the present study, the conventional composite (HER) and two bulk-fill composites (SDR and FBF) exhibited higher BFS values despite differences in their filler contents. The good mechanical properties of HER can be attributed to the high filler loading (approximately 59% by volume).[28]

The products SDR and FBF are flowable composites and theoretically should undergo a higher degree of conversion than composites with regular viscosities.[29] SDR contains triethylenegycol dimethacrylate (TEGDMA), ethoxylated bisphenol-A dimethacrylate (EBPDMA), and urethane dimethacrylate (UDMA) modified by chain modulators: chemical moieties in the resin backbone that increase flexibility.[30] As a consequence, this material has the lowest FM values (Table 4). The monomers bisphenol-A diglycidyl ether dimethacrylate (Bis-GMA), ethoxylated bisphenol-A dimethacrylate (Bis-EMA), TEGDMA, UDMA, and procrylat are present in FBF. Bis-EMA has a high molecular weight but does not contain pendant hydroxyl groups and, thus, has a lower viscosity than does Bis-GMA.[12] Although the FM of composite resins may be affected by the mass fraction,[28,31] this behavior was not observed for EXP and TEC

composites and was in agreement with results obtained in other studies,[12,17,26,32] suggesting that a higher filler percentage only does not necessarily reflect superior mechanical properties. The increasing polymer network density, stress transfer between the filler particles and the resin matrix, and adhesion between these components also influence the polymerization reaction and final properties.[29]

Although the bulk-fill composite TEC has higher filler loading (approximately 60%), it exhibited one of the lowest BFS values. Use of pre-polymerized filler particles, such as in this material, has previously been shown to result in poorer mechanical properties.[33] Conversely, the photoinitiator Ivocerin, a derivative of dibenzoyl germanium, is incorporated in TEC, in addition to the camphorquinone/amine initiator system. Ivocerin is excited by shortwave visible light (380–450 nm) and is a more efficient free-radical generator than camphorquinone, leading to rapid polymerization and high monomer conversion.[34] Interestingly, although short wavelength visible light has a high dispersion effect and low penetration,[35] TEC exhibited uniform BFS values from depths of 0.5 to 4 mm, suggesting a great depth of cure, perhaps as a result of incorporation of Ivocerin.

The mechanical performance of EXP was intermediate compared with that of the other materials and was not expected because the use of fiberglass is known to provide material reinforcement.[36] Therefore, other factors, such as filler volume and their orientation and distribution may have contributed to this result. In the present study, thin specimens (0.5 mm) were used to test the mechanical properties, and it is likely that the fibers were aligned perpendicular to the applied load, which significantly reduced their reinforcement capability.[37]

Many researchers have investigated the mechanical properties of bulk-fill composites, but most often flexural strength has been evaluated using the three-point bending test, according to the ISO 4049 standard.[17,28,30,32,38,39] This method requires overlapping light exposures of the specimen to yield a specimen length greater than the tip diameter of the light guide. Use of such overlapping exposures may result in higher monomer conversion which may directly affect mechanical properties. Using this method, SDR was found to have a higher BFS than TEC[32,29] and a similar value to that of FBF,[28,38] which is in agreement with the results of the present study. Conversely, EXP has been reported to have a higher BFS than SDR,[17] and FBF and TEC were reported to have higher BFS values than SDR in one study,[29,38] while SDR was found to have a BFS similar to that of TEC in a different investigation.[28] Furthermore, in the present study, using the piston-ring biaxial test, the BFS of HER was determined to be higher than those of TEC and EXP but similar to those of the bulk-fill flowable composites SDR and FBF. Furthermore, the FM of the regular composite was found to be higher than those of the bulk-fill composites. The relationship between the LT findings and the BFS results are very interesting. All of the bulk-fill resin composites exhibited uniform BFS values at depths ranging from 0.5 mm to 4 mm, while the BFS values of HER composite decreased as the depth surpassed 2 mm (Table 3), although light attenuation was noted for all composites (Table 2). Monomer conversion, and consequently the mechanical properties, at a specific depth is not only dependent on the light reaching this particular layer but also on the initiation of the polymerization process of the layers above propagating in depth.[13] Depth of cure depends on filler characteristics, monomer composition, initiator concentration, shade and translucency of the material, and irradiance of the light source.[26,35]

When the effects of polymerization characteristics on mechanical properties are studied, tests in which single-shot curing protocols for making specimens are applicable, and biaxial flexure strength values are indicated.[6,12,39] BFS of two bulk-fill composites (X-tra base, Voco; and SDR, Dentsply) was evaluated at different depths up to 8 mm. Literature values found for SDR were in agreement with the results obtained in the present study because no statistically significant differences were found at depths up to 4 mm. At a depth of 8 mm, however, a measurable difference in the BFS value was noted compared with that obtained at 1 mm. These findings were also supported by monomer conversion analyses conducted by the same authors, which revealed no significant difference from depths of 1 mm to 4 mm.[12]

The present results suggest that the biaxial flexural test can be used as an indirect method for evaluating the depth of cure and comparing curing protocols for materials that may be used in a clinical setting. It must be noted, however, that the biaxial flexural test may not provide reliable data for elastic modulus determination.[6] In clinical situations, light can be easily attenuated by tip-to-target distance and by the angle between the tip of the light curing unit and the composite surface. In the present study, these factors were minimized by placement of the tip directly on the resin composite surface. Therefore, further studies that better simulate

clinical situations with different types of curing units and possibly clinical studies are required to ensure the adequate clinical performance of bulk-fill composites.

CONCLUSION

Within the limitations imposed by this in vitro study, the following conclusions may be made:

1. Different inorganic filler content characteristics were found among the composite resins. Irregular, spherical and cylindrical shapes were observed with sizes varying from 0.1 μm to 1 mm. Aluminium, barium, and silicon were present in all of the fillers.
2. LT decreased as composite thickness increased for both the regular and bulk-fill materials. However, the conventional composite HER demonstrated lower LT than the bulk-fill materials, among which SDR had the highest LT.
3. Light attenuation did not influence BFS of bulk-fill composites, while HER presented decreased BFS at greater depths. FM for SDR, FBF and TEC bulk-fill composites was reduced with increasing composite depth.

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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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