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Extensive Assessment of the Physical, Mechanical, and Adhesion Behavior of a Low-viscosity Bulk Fill Composite and a Traditional Resin Composite in Tooth Cavities

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Clinical Relevance

Integrated analysis of the physical, mechanical, and adhesion behavior of low-viscosity bulk fill resin composite confirms that it presented equal or superior properties when compared with a traditional resin composite, which has been known to be clinically successful.

SUMMARY

Objectives: To compare the degree of conversion (DC), depth of polymerization (DP), shrinkage stress (SS), flexural strength (FS), elastic modulus (EM), and bond strength (BS) of a low-viscosity bulk fill resin composite and a paste-like traditional composite.

Methods: Tetric Evo-Flow Bulk Fill (TBF) and Empress Direct (ED; Ivoclar Vivadent) composites were used. DC (%) and FS/EM (MPa/GPa) were evaluated in bar specimens (7×2×1 mm; n=10) using Fourier-transform infrared spectroscopy and a three-point bending test in a universal testing machine (UTM), respectively. For DP and BS tests, conical cavities (n=10) were prepared in bovine dentin and restored with the composites. DP was analyzed by calculating the bottom-to-top surface microhardness ratio (BTHR), and BS (MPa) was determined by push-out testing in the UTM. SS (MPa) was measured for one increment of

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TBF and two increments of ED in a UTM attached to an extensometer (n=5). Data were analyzed using Student *t*-test and analysis of variance (α=0.05).

Results: TBF presented higher values than ED for DC (85.7±6.6% vs 54.2±4.9%) and BS (0.95±0.70 MPa vs 0.35±0.15 MPa). TBF values were lower than ED values for FS (76.6±16.8 MPa vs 144.9±24.1 MPa) and maximum SS (0.77±0.07 MPa vs 1.07±0.15 MPa). TBF and ED values were similar for BTHR (0.83±0.16 vs 0.84±0.08) and EM (11.5±2.8 GPa vs 12.5±2.6 GPa).

Conclusions: The physical and mechanical properties of TBF, a bulk fill resin composite, were similar or superior to those of ED, a conventional composite, with the exception of FS measurements.

INTRODUCTION

A major objective in restorative dentistry is the development of resin composites that allow rapid filling of cavities and provide acceptable clinical longevity. Current clinical protocols recommend the application of traditional resin composites in increments of no more than 2-mm thickness.[1] However, this technique increases chair time, favors the incorporation of voids in the restoration body, increases the risk of contamination by moisture between composite layers, and makes working in small cavities more difficult.[2,3]

Recent advances in resin composites have led to the emergence of bulk fill materials,[4] which can be inserted in a dental cavity in a single 4-5–mm layer[5,6] and are commercially available in low and medium viscosity (paste-like consistency).[7] Both of them generally have decreased filler loads and increased filler sizes to enhance the depth of cure. They also may contain photoinitiators able to provide effective depth of cure at 4-5 mm, as well as monomers with low double-bond concentrations and, in some cases, monomers that cleave during polymerization.[8,9]

Most traditional flowable composites have reduced percentages of inorganic filler particles (44%-55% of total volume) and greater amounts of resinous components, in comparison with traditional paste-like composites. Flowable composites have low elastic moduli that compete with stress development, potentially helping to maintain the marginal seal of the restoration.[10] Moreover, flowable composites are readily workable and adaptable to cavity walls, and their use can reduce marginal defects in restorations.[11]

Many studies have demonstrated the favorable properties of low-viscosity bulk fill composites, such as a suitable degree of conversion (DC)[2,6,12] and bond strength similar to that of traditional paste-like composites.[13] However, a limited number of studies have evaluated the physical, mechanical, and adhesion behaviors of low-viscosity bulk fill composites using methods capable of reproducing clinical conditions, such as the filling of dentin cavities that are deep (4 mm) and have high C-factor.

The aim of this study was to compare the DC, depth of cure, shrinkage stress, flexural strength, elastic modulus, and bond strength of a low-viscosity bulk fill composite and a conventional resin composite in dentin cavities with high C-factor. The null hypothesis was that there is no significant difference between materials tested.

METHODS AND MATERIALS

Experimental Design

This laboratory study was conducted to examine the response variables DC, depth of polymerization, maximum shrinkage stress, flexural strength, elastic modulus, and bond strength in a low-viscosity bulk fill resin composite (Tetric Evo-Flow Bulk Fill [TBF]; Ivoclar Vivadent, Schaan, Liechtenstein) and a conventional resin composite (Empress Direct [ED]; Ivoclar Vivadent). For DC, depth of cure, flexural strength, elastic modulus, and bond strength, the factor under study was resin composite (TBF vs ED). For the evaluation of maximum shrinkage stress, resin composite (TBF vs ED) and increment (one 4-mm increment of TBF, first and second 2-mm increments of ED, and sum of first and second ED increments) were the factors under study. The compositions of the materials are listed in Table 1.

Degree of Conversion, Flexural Strength, and Elastic Modulus Tests

For these tests, bars (7×2×1 mm) of the two resin materials (n=10 each) were prepared using a polyvinyl siloxane mold (Express XT; 3M ESPE, St Paul, MN, USA).[14] Both resin materials were inserted in single increments. A polyester strip and a 1-mm glass plate covered each specimen, and final photoactivation was performed for 20 seconds using a polywave light-emitting diode (LED) light-curing unit (Bluephase G2; Ivoclar Vivadent) at 1150 mW/cm^2 (measured by using a computer-controlled

Table 1: *Materials Used in this Study*[a]

Material	Manufacturer	Chemical Composition (wt%)	Lot No.
Tetric Evo Flow Bulk Fill (TBF)	Ivoclar Vivadent	Ethoxylated bisphenol A dimethacrylate (10-25%), Bis-GMA (3-7%), ytterbium trifluoride (3-5%), tricyclodecane dimethanol dimethacrylate (1-3%)	TM0056
Empress Direct (ED)	Ivoclar Vivadent	urethane dimethacrylate (10-25%), Bis-GMA (2,5-3%), ytterbium trifluoride (3-10%), tricyclodecane dimethanol dimethacrylate (3-10%)	T28435
Stae	SDI	Acetone (50-55%), acrylic monomers (20-40%)	141141

Abbreviation: Bis-GMA, bisphenol A diglycidyl ether dimethacrylate.
[a] *Data obtained from Material Safety Data Sheet (MSDS).*

spectrometer - USB2000, Ocean Optics, Dunedin, FL, USA). The samples were stored in an oven at 37°C for 24 hours before testing.

First, DC was measured using Fourier-transform infrared spectroscopy (IRTracer-100; Shimadzu, Kyoto, Japan) coupled to a total attenuated reflectance device. The absorption spectra of nonpolymerized and polymerized materials were determined between 1800 and 1500 cm^{-1}, with 32 scans taken at 4-cm^{-1} intervals. As both composites contain aromatic vinyl bonds of bisphenol and aliphatic bonds of the methacrylate functional group, the aliphatic carbon-to-carbon double-bond absorbance peak intensity (located at 1638 cm^{-1}) and that of the aromatic component (located at 1608 cm^{-1}; reference peak) were measured. The DC was calculated using the following equation: $DC\ (\%) = 100 \times [1-(R_{polymerized}/R_{nonpolymerized})]$, where R represents the ratio between the absorbance peaks at 1638 cm^{-1} and 1608 cm^{-1}.

Flexural strength (in MPa) and elastic modulus (in GPa) were then evaluated using a universal testing machine (model 4111; Instron Corporation, Dayton, OH, USA) with a three-point bending design, a crosshead speed of 0.5 mm/min, and a 50 N load cell until fracture. The distance between supports was 5 mm. Bluehill 2 software (Illinois Tool Works Inc, Glenview, IL, USA) was used to calculate flexural strength and elastic modulus, considering the dimensions of the specimens.

Depth of Polymerization and Push-out Bond Strength Tests

Figure 1 is a schematic representation of the specimen preparation and methods used to evaluate the depth of polymerization and push-out bond strength. Twenty bovine incisors with no enamel cracks or structural defects were selected and decontaminated in an aqueous solution of thymol (0.1%) at 4°C for one week. The roots were removed at the cementoenamel junction (CEJ) with a water-cooled diamond saw using a precise cutting machine (Isomet 1000; Buehler, Lake Forest, IL, USA). A transverse mesiodistal cut was made 4 mm from the CEJ to obtain a 4-mm-thick slice with a central void corresponding to the pulp cavity. Standardized conical cavities (4.8-mm top diameter×2.8-mm bottom diameter×4-mm height) were prepared with Maxicut burs (Komet Inc, Lemgo, Germany) using a hand piece under air-water cooling. The bur was replaced every 10 preparations. The cavities had Configuration-factors of 2.5.

After cavity preparation, bonding procedures were performed. Dentin was acid-etched for 15 seconds with phosphoric acid (Super Etch; SDI, Victoria, Australia) and rinsed thoroughly with water for 15 seconds. Excess water was blotted with absorbent paper, leaving the dentin surface visibly moist (wet bonding). The Stae adhesive system (SDI) was applied; photoactivation was performed for 10 seconds with an LED unit (Bluephase G2; Ivoclar Vivadent). Each specimen was placed over a glass slide, and the composite was inserted into the cavity. TBF was inserted in one increment, completely filling the cavity; a polyester strip was placed on the top surface and photoactivated for 20 seconds. ED was inserted incrementally; the first 2-mm increment was inserted and photoactivated for 20 seconds, then the second 2-mm increment was inserted and photoactivated for 20 seconds under a polyester strip. After 24 hours, the restorations were finished/polished with Sof-Lex Pop On aluminum oxide discs (3M ESPE) and stored in water for 24 hours at 37°C before evaluation of the depth of polymerization and bond strength.

Depth of polymerization was analyzed using the bottom-to-top hardness ratio.[15] Knoop indentations were made on the top and bottom surfaces of each restoration with an indenter (HMV-2; Shimadzu), using a 50-*g* load for 30 seconds. Three readings were taken on each surface, and the mean Knoop hardness numbers and the bottom-to-top hardness ratio were calculated.

Then, push-out bond strength was evaluated. An acrylic device with a central hole was adapted to the

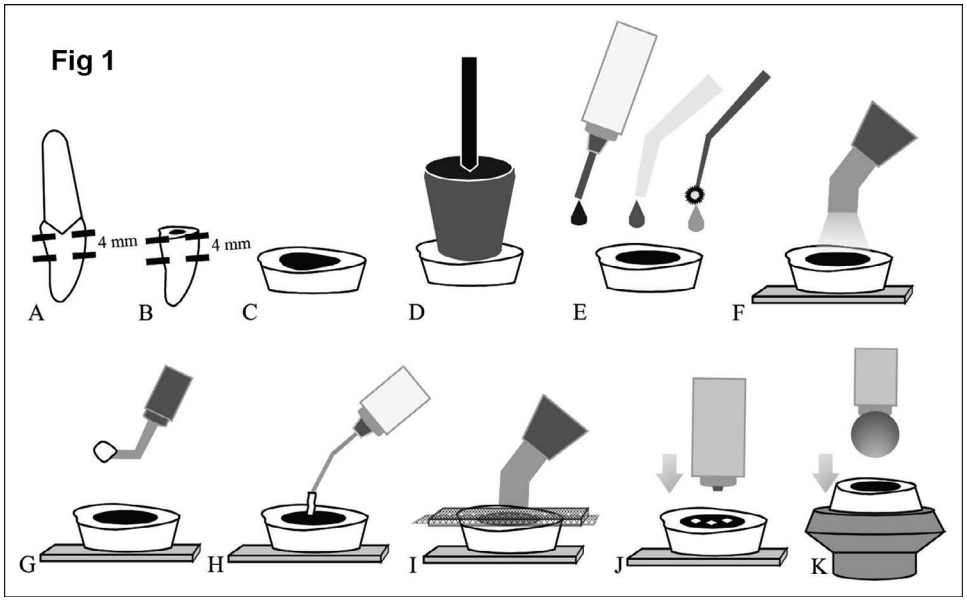

Figure 1. *Schematic representation of the polymerization depth and push-out bond strength analyses. Crowns of bovine incisors were removed from the roots at the cementoenamel junction (CEJ) and then cut 4 mm from the CEJ (A,B) to create three-dimensional 4-mm dentin cavities (C). Cavities were prepared using a Maxicut bur (D). Cavities were acid-etched and washed with water, and adhesive systems were applied to moistened dentin (E), which was photoactivated (F). Two increments of conventional composite (G) and a single increment of flowable bulk fill composite (H) were applied and photoactivated (I) under a Mylar strip and glass plate. Knoop hardness was measured on the top and bottom surfaces (J), and push-out bond strength was evaluated (K).*

base of a universal testing machine (model 4411; Instron, Buckinghamshire, UK). The central hole was used to position each specimen with the preparation bottom (the end with the smallest diameter) facing up. A rounded probe was adapted to the testing machine and a compressive force was applied with a crosshead speed of 0.5 mm/min to the bottom surface of the restoration to induce rupture of the tooth composite bonding area. The results (in Kgf) were divided by the bonded area (22.65 mm^2) and transformed to MPa. After the test, failure mode was examined using a dissecting microscope (Stereozoom; Bausch & Lomb, Rochester, NY, USA), using the following classification: adhesive (type 1), adhesive/cohesive in composite (mixed 1; type 2), adhesive/cohesive in composite and dentin (mixed 2; type 3), cohesive in composite (type 4), and cohesive in dentin (type 5).

Shrinkage Stress

Shrinkage stress was characterized as described in a previous study.[16] Briefly, the flat surfaces of poly(-methyl methacrylate) rods (6-mm diameter, 13- and 28-mm lengths) were sandblasted to produce a layer of methyl methacrylate, followed by two coats of a bonding agent (ScotchBond Multi-purpose Plus Adhesive; 3M ESPE). The unfilled resins were light-cured with an LED unit (Bluephase G2; Ivoclar Vivadent) for 10 seconds. The rods were attached to the opposing clamps of a universal testing machine (model 3342; Instron) with the treated surfaces facing each other at distances of 4 mm for TBF and 2 mm for ED. The composite was inserted into the empty space and shaped into a cylinder according to the rod perimeters. An extensometer (model 2630-101, 0.1-mm resolution; Instron) was attached to the rods to monitor specimen height and to provide feedback to the testing machine to keep the height constant. The load cell provided the corresponding force necessary to counteract the polymerization shrinkage force to maintain the specimen's initial height. The short rod was attached to the testing machine through a hollow stainless-steel fixture with a lateral slot that allowed the tip of the LED guide to be positioned in contact with the polished end surface of the rod. Force development was monitored for 20 minutes from the beginning of photoactivation, and nominal stress was calculated by dividing the maximum force value by the cross-sectional area of the rod. For TBF, stress was calculated only for 4-mm bulk increments. For ED, stress was calculated for 2-mm thicknesses placed directly between the rods (first increment), 2-mm thicknesses placed between the rod and a prepolymerized 2-mm increment (second increment), and for the sum of the first and second increments (n=5). The maximum shrinkage stress of photoactivation (expressed in MPa) was obtained from stress vs time curves.

Statistical Analysis

The Student *t*-test was used to analyze the DC, flexural strength, elastic modulus, depth of cure, and push-out bond strength (α=0.05). Maximum shrinkage stress was evaluated using one-way analysis of variance (ANOVA) with Tukey tests ($p<0.05$). IBM

Table 2: *Means ± Standard Deviations of Degree of Conversion (DC; %), Flexural Strength (FS; MPa), Elastic Modulus (EM; GPa), Bond Strength (BS; MPa), and Hardness Bottom-to-top Ratio (HB/T) According to the Composite Resins Analyzed*[a]

	DC	FS	EM	BS	HB/T
TBF	85.7 ± 6.6 A	76.6 ± 16.8 B	11.5 ± 2.8 A	0.94 ± 0.70 A	0.83 ± 0.16 A
ED	54.2 ± 4.9 B	144.9 ± 24.1 A	12.5 ± 2.6 A	0.35 ± 0.15 B	0.84 ± 0.08 A
p-value	0.001	0.001	0.415	0.026	0.944

Abbreviations: ED, Empress Direct; TBF, Tetric Evo Flow Bulk Fill.
[a] *Different letters indicate statistically significant differences among the resins according to T test, considering each evaluated property (each column).*

SPSS Statistics for Windows software (version 20.0; IBM Corporation, Armonk, NY, USA) was used to perform statistical analyses.

RESULTS

The DC, flexural strength, elastic modulus, depth of polymerization, and bond strength results are presented in Table 2. The DC and bond strength were significantly greater for TBF than for ED. Flexural strength was significantly greater for ED (144.9±24.1 MPa) than for TBF (76.61±16.8 MPa). TBF and ED had similar elastic moduli (11.5±2.8 GPa and 12.5±2.6 GPa, respectively) and depths of cure/bottom-to-top hardness ratio (0.83±0.16 and 0.84±0.08, respectively). All samples showed adhesive (type 1) failures between the adhesive and dentin.

The shrinkage stress results are presented in Table 3. ANOVA showed significant differences in maximum shrinkage stress according to material and increment (all p=0.001). The highest maximum shrinkage stress mean was obtained for the sum of the first and second increments of ED, while the lowest maximum shrinkage stress mean was obtained for the second increment of ED.

DISCUSSION

The null hypothesis that there was no significant difference between materials was rejected, since the resin composites showed only similar elastic moduli and depths of polymerization. The conventional material showed greater flexural strength than did the bulk fill, and the latter showed less shrinkage stress and greater bond strength and DC than did the former.

Intrinsic characteristics of the chemical components of resin composites, such as the amount of filler particles, type and concentration of the photoinitiator system, type and amount of monomers, color, and translucency, are related directly to the DC.[10,17] The bulk fill material tested has a comparable amount of filler particles to the conventional one. However, the former contains a germanium-based photoinitiator, Ivocerin, which can absorb more light in the range of 400-450 nm. Photoactivation of Ivocerin forms at least two radicals that initiate radical polymerization; in contrast, only one radical is formed in camphorquinone/amine systems, present in the conventional material, resulting in less efficient initiation of polymerization.[18] In addition, the bulk fill material has more low-viscosity monomers and is more translucent than is the conventional one. Low-viscosity monomers increase the DC of resin-based materials as a result of their greater mobility during polymerization;[19] indeed, translucent materials favor higher light transmittance and photoinitiator excitation.[20] These properties explain the higher DC observed for the bulk fill in comparison with conventional material.

A uniform DC along the composite thickness can guarantee a satisfactory depth of polymerization. In this study, the top-to-bottom hardness ratio was used to determine the polymerization depth of resin composites.[15] Bulk fill and conventional materials had similar bottom-to-top hardness ratios, despite differences in incremental application and photoactivation. The presence of a more reactive photoinitiator system, more low-viscosity monomers, and increased translucency in the bulk fill material explain this similarity in polymerization depth, despite the thickness of its increment. The high

Table 3: *Means ± Standard Deviations of Maximum Shrinkage Stress (MPa) in Accordance with the Type of Increment/Resin Composite Analyzed*[a]

Type of Increment/Resin Composite	Maximum
4-mm increment of TBF	0.77 ± 0.07 B
First 2-mm increment of ED	0.69 ± 0.03 B
Second 2-mm increment of ED	0.37 ± 0.13 C
Sum of first and second increments of ED	1.07 ± 0.15 A
p-value	0.001

Abbreviations: ED, Empress Direct; TBF, Tetric Evo Flow Bulk Fill.
[a] *Different letters indicate statistically significant differences among the resins.*

radiance emitted by the curing light used in this study likely favored satisfactory excitation of photoinitiators from the tops to the bottoms of the specimens, resulting in bottom-to-top hardness ratios > 0.8, considered to be satisfactory.[21] As cavity filling with a single 4-mm composite resin increment, instead of two 2-mm increments, reduces chair time, the use of a low-viscosity bulk fill composite can simplify restorative procedures under clinical conditions.

The DC, presence of crosslinking high-molecular-weight monomers, and percentage, size, and shape of filler particles are related to the elastic moduli of resin composites.[22] Increased amounts of filler particles and crosslinking high-molecular-weight monomers decrease the DC, as they promote light scattering and decreased mobility during polymerization, respectively, although they favor an increased elastic modulus of resin composites.[1] The bulk fill material presented a higher DC, whereas the conventional one contains more crosslinking high-molecular-weight monomers and filler particles, explaining the similar elastic moduli observed for these two materials in this investigation. On the other hand, the conventional material had greater flexural strength than did the bulk fill. However, the latter is a flowable material that requires a covering layer of high-viscosity resin composite in tooth restorations;[22,23] in this way, when the manufacturer's recommendations are followed, it is possible that the reduced flexural strength of the bulk fill material should not be a problem since the covering material would have increased flexural strength. However, further analysis should be performed to confirm this assumption.

Recent research has focused on the influence of resin matrix chemistry on the mechanical properties of resin composites. Factors such as the type and amount of filler particles, transference of stress between the filler particles and resin matrix, adhesion between these components, and the chemical composition of the matrix have been associated with differences in flexural strength.[22-25] The greater flexural strength of the conventional material in this study can be explained by the high percentage of inorganic fillers in this composite.[26]

Shrinkage stress occurs as a result of the development of covalent bonds between the polymer chains already formed, causing contraction, and is defined by a combination of factors such as matrix composition, amount of charge, photoactivation standard, DC, elastic modulus, and shape of the cavity into which the composite is inserted.[27] In this study, shrinkage stress of the conventional material was measured in two separate steps to better simulate the incremental insertion protocols of conventional resin composites. Shrinkage stress was significantly higher for the conventional than for the bulk fill material, meaning that the latter was better able to dissipate the generated tension. This can be appointed as significant advantage of the low-viscosity bulk fill resin composite, because even with application in a single 4-mm increment, this material showed less shrinkage stress than did a traditional paste-like composite.

The flow of stresses may be associated with the pre-gelation phase time, and this can be attributed to the modified polymer chains of bulk fill flowables, which are very flexible in the pre-gelation phase. This highly stress-relieving internal monomer may delay the gel point, which could allow more time to compensate for shrinkage; consequently, shrinkage stress would be reduced. This may have been a factor that caused a better dissipation of tensions by the bulk fill material.[28,29]

Another strategy that may be related to decreased shrinkage is the replacement of high-molecular-weight with lower-weight monomers.[28] The bulk fill material contains urethane dimethacrylate (UDMA), and the conventional one contains ethoxylated bisphenol A dimethacrylate (Bis-EMA); although both are considered to be high-weight monomers, Bis-EMA has a lower weight (362.41 g/mol) than does UDMA (470.56 g/mol); this difference may explain the lower tension generated by this material.

In general, shrinkage stress was greater for the first increment of the conventional composite than for the second increment. However, shrinkage stress may have been reduced in the second increment because it had to be placed over a previously applied 2-mm increment because of methodological aspects, which can absorb stress.

In push-out bond strength tests, stress generated by composite polymerization is transferred directly to the adhesive interface, as the composite shrinks into the cavity. Although the bond strength of bulk fill composites can also be analyzed using microtensile tests after the filling of Class I and Class II cavities,[30,31] specimen preparation using diamond saws can apply external stress to the tooth/composite interface, leading to underestimation of bond strength values. In contrast, the push-out method allows the measurement of bond strength without this external stress, so that differences between materials are related to their intrinsic characteris-

tics. Thus, the lower maximum shrinkage stress of the bulk fill material in comparison with the conventional one explains the greater bond strength of the former.

Further studies involving physical, mechanical, and adhesion analyses of other low-viscosity and paste-like bulk fill composites in high–C-factor dentin cavities should be performed in an attempt to obtain information regarding materials from other manufacturers. Moreover, dentin bond strength analysis after aging methods should also be investigated further to evaluate dentin bond durability of low-viscosity bulk fill and conventional resin composites in high–C-factor dentin cavities.

CONCLUSIONS

The physical and mechanical properties of Tetric Evo Flow Bulk Fill, a low-viscosity bulk fill resin composite, were similar or superior to those of Empress Direct, a conventional resin composite, with the exception of flexural strength.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Federal University of Rio Grande do Norte (UFRN).

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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