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BRUNA MARIN FRONZA

AVALIAÇÃO DE PROPRIEDADES FÍSICO-QUÍMICAS DE COMPÓSITOS RESINOSOS *BULK-FILL*

EVALUATION OF PHYSICOCHEMICAL PROPERTIES OF *BULK-FILL* COMPOSITE RESINS

Piracicaba
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Universidade Estadual de Campinas
Faculdade de Odontologia de Piracicaba

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Dissertação apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Mestra em Clínica Odontológica, na Área de Dentística.

Dissertation presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Master in Dental Clinic, in Operative Dentistry area.

Orientador: Prof. Dr. Marcelo Giannini

Este exemplar corresponde à versão final da dissertação defendida por
Bruna Marin Fronza e orientada pelo Prof. Dr. Marcelo Giannini

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Prof. Dr. FLÁVIO HENRIQUE BAGGIO AGUIAR

RESUMO

Os objetivos no presente estudo foram: (1) caracterizar o conteúdo inorgânico; (2) investigar a passagem de luz (PL) através de incrementos com diferentes espessuras; (3) avaliar a resistência à flexão biaxial (RFB) e módulo de elasticidade (MO) em função da profundidade de polimerização; (4) verificar o grau de conversão (GC) e microdureza (MD) e (5) correlacionar a tensão de polimerização (TP) com a formação de fenda marginal (FM) em cavidades classe I com materiais restauradores *bulk-fill*, comparando com um compósito resinoso tradicional. Quatro resinas *bulk-fill* foram avaliadas: Surefill SDR flow (SDR), Filtek Bulk Fill (FBF), Tetric EvoCeram Bulk Fill (TEC) e EverX Posterior (EXP). Herculite Classic (HER) foi utilizada como controle. Para a caracterização da morfologia e composição do conteúdo inorgânico, uma porção de cada material foi imersa em solventes para remoção da matriz resinosa e posteriormente analisada por EDX e MEV. A PL foi mensurada através de diferentes espessuras dos materiais (1, 2, 3 e 4 mm) utilizando radiômetro acoplado a esfera integrada (n=5). Para o teste de RFB e MF, discos de resina composta (0,5 mm de espessura) foram confeccionados em um conjunto de matrizes sobrepostas (n=8), simulando profundidade de polimerização de 4 mm. O GC obtido por espectroscopia Confocal Raman e MD Knoop foram determinados em cavidades classe I com 4 mm de profundidade, as quais foram restauradas com as resinas e respectivos adesivos (n=5). A partir dessas restaurações foram confeccionadas réplicas em resina epóxi para análise de formação de FM em MEV. A TP de cada material foi determinada por um extensômetro associado a uma máquina de ensaio universal (n=5). As partículas de carga das resinas *bulk-fill* apresentaram formato irregular, esferoidal ou cilíndrico de tamanhos variados, de 0,1 μm a 1 mm. Quanto a composição os principais elementos químicos identificados foram alumínio, bário e silício. Todos os materiais demonstraram menor passagem de luz com o aumento da espessura. Não houve diferença entre a PL para as resinas *bulk-fill*, porém apenas a SDR permitiu maior PL que HER. A atenuação da luz, entretanto, não teve influencia na RFB das resinas *bulk-fill*, enquanto para HER na maior profundidade houve redução da RFB. Embora, o MO ainda foi maior para HER. O GC foi uniforme em termos de profundidade para os materiais SDR, FBF e HER aplicado em incrementos. De modo contrário, os materiais TEC, EXP e HER em incremento único tiveram diminuição do GC com o aumento da profundidade. Para o teste de MD, resultados inferiores com o aumento da profundidade foram encontrados apenas para TEC, assim não houve correlação entre as análises de MD e GC. Com relação a TP, EXP apresentou os valores mais altos, enquanto TEC os menores. Correlação positiva foi detectada entre TP e formação de FM, as quais variaram entre os materiais. A técnica incremental demonstrou reduzir a FM para HER, SDR e TEC tiveram comportamento comparável a essa técnica convencional. As resinas *bulk-fill* apresentaram variações nos resultados, algumas demonstraram resultados semelhantes ou superiores à resina controle, podendo ser uma opção para os tradicionais procedimentos restauradores diretos.

Palavras-chave: resinas compostas, polimerização, resistência de materiais, dureza.

ABSTRACT

The aims of this study were: (1) to characterize the inorganic content; (2) investigate light transmission (LT) through different thicknesses increments; (3) assess biaxial flexural strength (BFS) and modulus (MO) according to depth of cure; (4) verify degree of conversion (DC) and microhardness (MH) and (5) correlate polymerization stress (PS) and gap formation (GF) in class I cavities of bulk-fill restorative materials, compared to a regular composite resin. Four bulk-fill composites were tested: Surefill SDR flow (SDR), Filtek Bulk Fill (FBF), Tetric EvoCeram Bulk Fill (TEC) and EverX Posterior (EXP). Herculite Classic (HER) was used as control. To morphological and composition characterization of inorganic content, a portion of each material was immersed in solvents to remove resin matrix and then analyzed by energy-dispersive X-ray and scanning electron microscopy. The LT was measured through different material thicknesses (1, 2, 3 and 4 mm) by a laboratory-grade spectral radiometer spectroradiometer (n=5). To BFS and MO tests, discs (0.5 mm thick) were fabricated using a set of eight molds, to simulate polymerization depth of 4 mm. DC obtained by Confocal Raman spectroscopy and Knoop MH were measured in 4 mm depth class I cavities, which were placed with the composites and it respective adhesives (n=5). PS was determined for each material by an extensometer attached to a universal testing machine (n=5). Filler particles of bulk-fill composites presented irregular, spherical or cylindrical shape, which size varied from 0.1 μm to 1 mm. Regarding composition analysis, aluminum, barium and silicon were identified in all composites. All materials demonstrated lower LT with increased increment thickness. No differences were found in translucency among bulk-fill composites, only SDR had higher light transmission than HER. However, light attenuation did not influence BFS of bulk-fill composites, while HER presented decreased BFS at deeper layers. Nevertheless, HER still had the highest MO. DC was uniform among depths for SDR, FBF and HER applied incrementally. Conversely, TEC, EXP and HER bulk-filling presented a decrease in DC with increased depth. To MH test, lower results with increased depth were detected only for TEC, indeed there was no correlation between DC and MH analyzes. Regarding PS, EXP had the highest values, whilst TEC the lowest ones. Positive correlation was detected between PS and GF, which varied according to materials. Incremental filling technique reduced GF percentage to HER, and only SDR and TEC had comparable results to this conventional technique. Some of the bulk-fill composites presented similar or superior outcomes compared to control, which may be an option for traditional direct restorative procedures.

Key words: composite resins, polymerization, material resistance, hardness.

SUMÁRIO

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INTRODUÇÃO

A busca pelo material restaurador ideal é constante na Odontologia. É desejável que este material seja duradouro e biocompatível no ambiente bucal, resistente às cargas oclusais, capaz de se aderir às estruturas dentais e apresente estética aceitável. Além disso, deve ser de fácil aplicação, preferencialmente de técnica direta com preparo minimamente invasivo que permita a preservação da estrutura dental remanescente. De todos os tipos de materiais restauradores, as resinas compostas associadas aos sistemas adesivos são os materiais que melhor atendem a essas exigências (Rueggeberg, 2002; Ferracane, 2011; Anusavice, 2013).

Os compósitos resinosos são formados, basicamente, pela combinação de diferentes tipos de monômeros, partículas de carga unidas à essa matriz orgânica por meio de um agente de união à base de silano, ativadores e iniciadores responsáveis por desencadear a reação de polimerização (Craig, 2012; Chen, 2009). As primeiras resinas compostas eram quimicamente ativadas e apresentavam razoável estética e durabilidade, entretanto demonstravam como desvantagem tempo de trabalho reduzido, baixa estabilidade de cor e resistência ao desgaste (Rueggeberg, 2002). O desenvolvimento de materiais ativados por luz facilitou a manipulação do compósito e melhorou a qualidade das restaurações. Porém, surgiram outros problemas inerentes ao procedimento, tais como a contração e tensão de polimerização e a subpolimerização dos compósitos (Tirtha *et al*., 1982; Rueggeberg, 1999; Stansbury, 2000).

Durante a polimerização, os monômeros se aproximam para estabelecer ligações covalentes com objetivo principal de produzir ligações cruzadas para formar uma rede polimérica com altas propriedades mecânicas (Stansbury, 2012). Essa aproximação resulta em contração volumétrica do material, que varia de 1 a 5% para as resinas compostas convencionais (Stansbury, 1990; Labella *et al*., 1999; Yamazaki et al., 2013). Quando essa contração ocorre sob confinamento em um preparo cavitário, devido a adesão às paredes da cavidade, uma tensão é gerada na interface entre o dente e a restauração. A magnitude dessa tensão é dependente não só da cinética de polimerização da resina composta, mas também do volume de material e do fator de configuração cavitário (Fator C), estimado pela área de superfícies aderidas em relação às superfícies livres (Carvalho *et al*., 1996; Braga *et al*., 2005). Como consequências clínicas da contração e tensão geradas durante a polimerização, são observadas trincas nas estruturas dentais, deflexão de cúspides e formação de fendas entre a restauração e o dente, que podem ocasionar sensibilidade pós-operatória, descoloração marginal, infiltração bacteriana e posterior desenvolvimento de cárie secundária (Ferracane e Mitchem, 2003; Calheiros *et al*., 2004; Ferracane, 2013).

A eficiência da polimerização está relacionada a importantes características das resinas compostas, como propriedades mecânicas, resistência ao desgaste e biocompatibilidade (Ferracane e Greener, 1986; Ferracane, 1994; Ferracane *et al*., 1997;). Com relação às propriedades mecânicas, a resistência do material restaurador deve ser maximizada, enquanto o módulo de elasticidade deve permanecer semelhante aos tecidos dentais adjacentes para evitar inadequada transferência de tensão durante a mastigação (Leprince, 2013). Em uma eficiente polimerização tem-se alta conversão monomérica que é dependente do sistema fotoiniciador, do aparelho fotoativador, das propriedades ópticas do material e sua capacidade de transmissão de luz em profundidade, além da viscosidade do material (Watts e Cash, 1994; Lovell *et al*.,1999; Rueggeberg, 2010). Em situações clínicas como cavidades profundas, grande volume de material restaurador e dificuldade de posicionamento do aparelho fotoativador, a luz pode não atingir o material em intensidade adequada para que ocorra uma polimerização uniforme e eficiente. Isso compromete as propriedades físicas do material e, consequentemente, a durabilidade das restaurações (Price e Felix, 2009; Rueggeberg, 2011).

Atualmente, as principais causas de falhas de restaurações de resina composta são a cárie secundária e a fratura do material (Sarret, 2005; Demarcoet al., 2012). Com a finalidade de minimizar os efeitos negativos, convencionou-se que esses materiais devem ser aplicados em incrementos que podem ser horizontais ou oblíquos com até 2 mm de espessura (Lutz *et al*., 1986; Pollack, 1987; Hilton e Ferracane, 1999). Dessa forma, seria possível assegurar um maior grau de conversão monomérico da resina e também controlar a tensão de polimerização, pela diminuição do volume de material e do fator C (Van Dijken, 2010; Borges *et al*., 2014). No entanto, essas técnicas são de difícil execução e demandam maior tempo clínico para os dentistas, que desejam materiais restauradores com maior facilidade de uso.

Modificações na formulação das resinas restauradoras têm sido feitas com a finalidade de superar essas deficiências, facilitar o procedimento restaurador e melhorar o desempenho clínico desses materiais. Os primeiros avanços foram com relação às partículas de carga, que tiveram tamanho reduzido para produzir materiais com polimento mais efetivo, melhor resistência ao desgaste e à fratura (Klapdohr e Moszner, 2005; Ferracane, 2011). Recentes tendências estão focadas em modificações da matriz resinosa, principalmente para desenvolver materiais com reduzida contração e tensão de polimerização (Ferracane, 2011; Stansbury, 2012). Entre essas novas formulações de compósitos resinosos, pode-se citar aquelas para aplicação em incremento único, também denominadas resinas *bulk-fill*. De acordo com os fabricantes, esses materiais permitem aplicação de incrementos de até 4 mm de espessura, com alegado grau de conversão uniforme por todo o incremento e reduzidas contração e tensão de polimerização. Diferentes mecanismos são utilizados para obter essas características, tais como modificações nas propriedades ópticas para aumentar a translucidez do material, alterações na cadeia dos monômeros para modular a reação de polimerização, adição de fotoiniciadores mais reativos e diferentes tipos de partículas de carga, como pré-polímeros capazes de absorver as tensões, e incorporação de fibras de vidro para aumentar a resistência mecânica.

O objetivo geral no presente estudo foi avaliar quatro sistemas resinosos *bulk-fill* quanto às suas propriedades físico-químicas, e comparar com uma resina composta convencional.

Os objetivos específicos neste estudo *in vitro* foram:

- Caracterizar a composição e a morfologia das partículas de carga das resinas compostas;
- Determinar a translucidez dos materiais através da avaliação de passagem de luz em incrementos de diferentes espessuras;
- Avaliar a resistência à flexão biaxial e módulo de elasticidade em diferentes profundidades de polimerização;
- Avaliar o grau de conversão e a microdureza Knoop em diferentes profundidades das restaurações de cavidades classe I com 4 mm de profundidade;
- Determinar a tensão de contração de polimerização de cada material;
- Investigar a formação de fendas internas em restaurações desses materiais aplicados em cavidades profundas classe I.

CAPÍTULO 1

Characterization of inorganic filler content, mechanical properties and light transmission of bulk-fill composite resins

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Abstract

Objectives: The aims of this study were to characterize the inorganic content, assess the light transmission (LT) and determine the biaxial flexural strength (BFS) and flexural modulus (FM) at different depths of one regular and four bulk-fill composites. Methods: The bulk-fill composites tested were Surefill SDR flow (SDR), Filtek Bulk Fill (FBF), Tetric EvoCeram Bulk Fill (TEC) and EverX Posterior (EXP). Herculite Classic (HER) was used as control. Energy dispersive X-ray and scanning electron microscopy were used to characterize filler particle compositions and morphologies. 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 composite discs (0.5 mm thick) were prepared simulating bulk filling of 4 mm (n = 8). Results: The SDR presented larger irregular particles than those observed for TEC and HER. The filler particles in FBF were spherical, while those in EXP were basically composed of fibreglass. The LT decreased as the composite thickness increased for all of the materials, and no significant differences in the LT were observed, although the SDR sample was more translucent than the HER composite. Furthermore, HER was a unique material with lower BFS values at greater depths. Conversely, HER exhibited a higher FM than SDR. Significance: the bulk-fill composites investigated exhibited high LT independent of the filler composition. Although an increase in the composite thickness reduced the LT, the BFS of the bulk-fill composites was not compromised despite the decreased irradiation at greater depths.

Keywords

Bulk-fill, dental composite, inorganic fillers, translucency, photopolymerization, biaxial flexural strength, flexural modulus.

1. Introduction

The development of dental resin-based composites began with the bisphenol-a glycidil methacrylate monomer (Bis-GMA) combined with glass filler particles by Bowen, in 1958 [1]. Since then, the composition of composite resins has evolved significantly. Current materials are composed of monomeric matrix and inorganic fillers bonded by a silane coupling agent, along with photoinitiator systems that promote polymerization when light activated to form crosslinked network with high mechanical properties [2-4]. Most of the changes have involved the inorganic fillers, which have been reduced in size to produce materials with greater polishing and wear resistance [3,5].

However, concerns related to the organic matrix remain such as the conversion of the monomers to polymers that provide materials with high modulus and strength [4,6] and polymerization shrinkage, caused by the monomers approximation during the reaction, which may negatively impact the clinical performance of bonded restorations [7,8]. Incremental filling techniques have been suggested to minimize polymerization shrinkage stress and ensure efficient polymerization of composites [9-11]. The maximum thickness recommended for each increment is 2 mm and depending on the composite resin formulation, light irradiation is necessary for 10 to 40 s. Thus, restorations for large cavity preparations are time consuming for the operator and inconvenient for the patient [12].

Bulk filling techniques have recently been introduced using new composite resin formulations. According to the manufacturers, these bulk-fill materials enable a depth of cure up to 4 mm with minimal polymerization shrinkage and stress. Successful resin composite restorations require an efficient polymerisation process, in order to enhance mechanical properties and biocompatibility [6]. One approach to improving bulk-fill composites is to increase their translucency to allow light to pass through the material, which induces to a uniform monomeric conversion [13]. The optical properties of resin composites and their light polymerization reaction are interdependent, i.e. a higher energy yields a higher degree of conversion [14]. Light transmission is affected by the composition of the material. Filler particles hinder light transmission (LT) due to scattering, which is dependent on the particle size and related to the incident wavelength of the curing light. The refractive indices of the fillers and resin matrix in a composite, as well as the mismatch between them, also influence light refraction. Other components, such as pigments and photoinitiators, absorb light and thus result in a decrease in the depth of cure [6,15,16].

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

The aims of this study were to characterize the morphology and composition of particles and their influence on the LT and the biaxial flexural strength (BFS) and flexural modulus (FM) of bulk-fill composites and one regular microhybrid composite at different depths. The following hypotheses were tested were: (1) there would be differences in filler particles characteristics between bulk-fill and conventional composites; (2) LT would be higher to bulk-fill composites compared to conventional composites and (3) there would be no difference in the mechanical properties at different depths for bulk-fill composites, while to conventional composite these properties would be reduced with increasing depth.

2. Materials and Methods

Five resin-based composites were investigated in the current study: one regular composite as a control (Herculite Classic), two high-viscosity bulk-fill composites (Tetric EvoCeram Bulk Fill and EverX Posterior) and two flowable bulk-fill composites (Surefil SDR and Filtek Bulk Fill). The compositions, lot numbers and manufacturers of the materials are presented in Table 1.

2.1. Inorganic content analysis and filler characterization

Unpolymerized composites (n = 5), ± 1 mg of material, were immersed in 6 mL of 99.5% acetone (Merck KGA, Darmstadt, Germany) and centrifuged (Excelsa, model 206, FANEM, São Paulo, SP, Brazil) for 5 min. This procedure was repeated until the entire organic matrix was dissolved [19]. Chloroform 99.8% (Merck KGA, Darmstadt, Germany) was then used in the same manner. The remaining content was then immersed in 6 mL of absolute ethanol (Merck KGA, Darmstadt, Germany) for 24 h, followed by drying at 37°C in an incubator (FANEM, São Paulo, SP, Brazil). The inorganic filler particles were then placed in plastic stubs and sputter-coated with carbon (MED 010 Baltec, Balzers, Liechtenstein) prior to energy-dispersive X-ray (EDX) spectrometry analysis or in metallic stubs and sputter-coated with gold (MED 010 Baltec, Balzers, Leichtenstein) prior scanning electron microscopy (SEM) observation.

EDX analyses (Vantage, NORAN Instruments, Middleton, WI, USA) coupled to a scanning electron microscope (JEOL, JSM-5600LV, Tokyo, Japan) were performed in order to identify the chemical compositions of the inorganic materials. Each spectrum was acquired for 100 s (voltage 15 kV, dead time 20–25%, working distance 20 mm). Images showing the identified chemical elements were obtained from five analyses of each material.

For morphological characterization of the filler particles, the specimens were observed using SEM (voltage 15 kV, beam width 25–30 nm, working distance 10–15 mm) at 50x, 1,000x and 5,000x magnifications. Representative images at different magnifications were obtained for each material for a qualitative analysis, and the particle sizes were determined using ImageJ Software (National Institute of Health, Bethesda, USA).

2.2. Light transmission through the composites

Composite cylinders (n = 5) of each material with four different thicknesses were fabricated in order to evaluate their LT. Silicon moulds with a diameter of 6 cm were used to manufacture discs with thicknesses of 1, 2, 3 and 4 mm. Each material was light activated using a polywave LED-curing unit (VALO, Ultradent Products Inc., South Jordan, UT, USA) for the exposure time recommended by the manufacturer.

The power density of the LED-curing unit and the light transmittance were determined using a laboratory-grade spectral radiometer (USB 4000, Ocean Optics, Dunedin, FL, USA) attached to a 7.62 cm diameter integrating sphere (DAS 2100, Labsphere Inc., Sutton, NH, USA), associated with specific software (Spectra Suite v5.1, Ocean Optics Inc., Dunedin, FL, USA). A mean value of 1153.4 mW/cm^2 was obtained for the total power density (100%) of the curing unit at wavelengths ranging from 350 to 550 nm. To measure the LT, each composite cylinder was positioned between the integrating sphere 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 five seconds of light irradiation. The transmittance value for each sample was calculated as a percentage by dividing the transmission measured through each specimen by the total light power density.

The data were identified as non-parametric using exploratory analyses. Specifically, the Kruskal Wallis and Dunn's test was used to analyse the material data ($\alpha = 0.05$), and Friedman's test was used for multiple comparisons of the results for different composite depths ($\alpha = 0.05$).

2.3. Biaxial flexural strength and modulus

Disc-shaped specimens (n = 8) with approximately 0.5 mm in thickness and 6.0 mm in diameter were fabricated using a set of eight Teflon moulds. A metal device was used to hold the eight Teflon moulds together. An acetate strip was positioned on the device with one mould on top. The mould was slight overfilled and then covered with a second acetate strip. A second mould was then placed on top of the acetate strip, and the procedure was repeated until all eight moulds were filled, resulting in a depth of 4 mm. A constant digital pressure was applied and the composite was light activated using the LED-curing unit. These procedures were executed in a light-proof room with a controlled temperature of 21°C.

After irradiation was complete, the specimens were removed from the Teflon moulds and their dimensions measured using a digital calliper (MDC-Lite, Mitukoyo Corporation, Kanagawa, Japan). The discs were stored in the dark in an incubator maintained at 37°C ± 1°C and a relative humidity of for one week prior to BFS determination.

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

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

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

$$X = (1 + v)ln(r_2/r_3)^2 + [(1 - v)/2](r_2/r_3)^2,$$

$$Y = (1 + v)[1 + ln(r_1/r_3)^2] + [(1 - v)(r_1/r_3)^2],$$

where v is Poisson’s ratio (0.25), r_1 is the radius of the support circle (mm), r_2 is the radius of the loaded area (mm) and r_3 is the radius of the disc (mm).

The BFS and FM values were calculated using SRS Biaxial Testing Software (Instron Corp., Canton, MA, USA) and were expressed in MPa and GPa, respectively. Data for both analyses were normal and homoscedastic. Split-plot two-way (material and depth) ANOVA and Tukey's post-hoc tests also were performed to detect differences among the groups ($\alpha = 0.05$).

3. Results

3.1. Inorganic content analysis and filler characterization

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

3.2. Light transmission through the composites

Table 2 presents the median values for the percentage of light passing through each composite. In general, the HER material had a lower LT ($p < 0.05$). However, all of the composites exhibited a similar trend for the light transmittance with respect to sample depth. No statistically significant differences were found between depths of 1 and 2 mm ($p > 0.05$), while statistically significant differences were observed for the light transmittance at 1 and 4 mm ($p < 0.05$).

3.3. Biaxial flexural strength and modulus

Average BFS and FM values for the composites are presented in Tables 3 and 4, respectively. Two-way ANOVA results indicated that the material ($p < 0.0001$) and depth ($p = 0.0007$) influenced the BFS results. The same analysis revealed that interactions between the factors was also significant ($p = 0.1456$) [20]. The BFS values at different depths for each of the bulk-fill composites (SDR, TEC, FBF and EXP) did not present any statistically significant differences ($p < 0.05$). Conversely, the regular composite (HER) had higher average values up to a thickness of 2 mm when compared to the values at a depth of 4 mm ($p > 0.05$). Differences were also found for the different materials. In general, HER, SDR and FBF exhibited higher BFS values, followed by EXP and TEC, which had the lowest values ($p > 0.05$).

Statistically significant differences in the FM values were also observed for the different materials ($p = 0.0001$) and depths ($p = 0.0129$). However, no interaction between the factors was identified as important ($p = 0.3258$). In general, the moduli of the top disc had a higher average value than that of the disc located at the bottom ($p < 0.05$). Furthermore, HER and SDR exhibited the highest and lowest modulus values independent of depth ($p < 0.05$). EXP, FBF and TEC exhibited intermediate values, with EXP possessing a higher modulus value than both FBF and TEC ($p > 0.05$), which had modulus values that were not statistically different ($p < 0.05$).

4. Discussion

The first hypothesis that the characteristics of the filler particles of the bulk-fill and conventional composites would be different was rejected. While the shapes of the filler particles in SDR, FBF and EXP were different and larger that the particles in the HER composite, the particles in the TEC composite were of similar shape and size to those in HER. Furthermore, LT through the composites was similar for all of the materials as a function of the composite depth, although SDR exhibited a slightly higher LT than HER, which had one of the lowest LT percentages of all of the materials. Thus, the second hypothesis that the LT would be higher for the bulk-fill composites than the regular composite was also rejected. The third hypothesis was only true for the BFS because BFS of the HER composite was higher at depths up to 2 mm than at a depth of 4 mm. However, the statistical analysis of the FM values indicated that there were no substantial differences at different depths for each of the materials.

LT 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 decrease with increasing filler size and shape irregularity. Studies have also shown that increasing the size of silica particles reduces the rate of polymerization at greater depths for experimental and commercial composites [6,22,23]. Furthermore, not only the size of the particles, but the amount of filler influences the LT. Higher filler content tends to reduce the LT, due to the increased probability of light refraction at the interfaces between the filler particles and the resin, due to the difference in their refractive indices [6,13].

The lower filler loading for the SDR composite explains its higher LT compared to that of the HER sample (Table 1), as well as larger particle size (approximately 20 μm) of the fillers (Figures 1 and 3). FBF also has low filler content, yet the LT through this composite was only different from that of the HER at a depth of 1 mm. In this case, the presence of zirconium in the FBF filler (Fig. 2) may influence the light transmission behaviour because it has a higher refractive index [24]. Both the TEC and EXP composites exhibited higher LT than the HER sample at a depth of 4 mm. Given that the inorganic content and morphological characteristics of the fillers in the TEC and HER composites 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 the EXP composite despite its high filler loading (57% by volume) may be due to the fiberglass filler, which may be effective in transmitting light inside the material [25].

A study compared light transmittance through nanohybrid, flowable and bulk-fill composites at 2, 4 and 6 mm incremental thickness [13]. Bulk-fill composites used in the study, including 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 that evaluated LT through pre-polymerized composite cylinders. The authors reported that light transmittance increased as the polymerization reaction progressed. As cross-linking starts, the density and refractive index of the polymer matrix increased, approaching the refractive index of the fillers and thus resulting in a reduction of scattering and an increase in the LT [13]. It is therefore possible that the LT values may be overestimated in the present study due to an increase in the LT of the pre-polymerised composites.

The relationship between the LT findings and the BFS results are very interesting because all of the bulk-fill resin composites exhibited uniform BFS values at depths ranging from 0.5 to 4 mm, while the BFS values of the HER composite decreased as the depth surpassed 2 mm (Table 3), although light attenuation was noted for all composites (Table 2). The polymerization and consequently the mechanical properties at a specific depth are not only dependent on the light reaching this particular layer, but also on the initiation of the polymerization process in the upper layers, which propagates in depth [13]. Depth of cure depends on the filler characteristics, monomer composition, initiator concentration, shade and translucency of the material, and the irradiance of the light source [25,26].

Resin composite viscosity has been shown to be an important parameter for determining the polymerization kinetics and final degree of conversion of dimethacrylate monomers because it influences monomer mobility and reactivity. In turn, the rheological properties of composite resins depend on the monomer composition and filler content [4,27]. In general, higher BFS values indicate higher monomer conversion. In the present study, the regular 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 the HER composite can be attributed to the high filler loading (approximately 59% by volume) [28].

SDR and FBF are flowable composites and theoretically should undergo a higher degree of conversion than composites with regular viscosities [29]. SDR contains TEGDMA, EBPDMA and 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). Bis-GMA, Bis-EMA, TEGDMA, UDMA and procrylat resin are present in the FBF formulation. Bis-EMA has a high molecular weight, but does not contain pendant hydroxyl groups, and thus has a lower viscosity than Bis-GMA [12]. Although the FM of composite resins may be affected by the mass fraction of [28,31], this behaviour was not observed for the EXP and TEC composites and was in agreement with results obtained in other studies [12,17,25,32], suggesting that a higher filler percentage 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 [32].

Although TEC composite has a higher filler loading (approximately 60%), it exhibited one of the lowest BFS values. The 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 into TEC in addition to the canforoquinone/amine initiator system. This initiator is excited by ultraviolet light (380–450 nm) and is a more efficient free radical generator than canforoquinone, leading to rapid polymerization and high monomer conversion [34]. Interestingly, although ultraviolet light has a low wavelength and consequently high dispersion effects and low penetration [26], TEC exhibited uniform BFS values from depths of 0.5 to 4 mm, suggesting a great depth of cure.

The intermediate mechanical performance of the EXP composite compared to the other materials was not expected because the use of fiberglass is known to provide material reinforcement [35]. Therefore, other factors, such as the volume of the fibers and their orientation and distribution must have contributed to this result. In the present study, thin specimens (0.5 mm) were used to test the mechanical properties (flexural strength and modulus), and it is likely that the fibres were aligned perpendicular to the applied load, which significantly reduced their reinforcement efficiency [36].

Various researchers have investigated the mechanical properties of bulk-fill composites, but most often the flexural strength has been evaluated using the three point bending test according to the ISO 4049 standard [17,28,30,32,37,38]. This method requires overlapping cure of the specimens to yield a higher degree of conversion, which directly affects the mechanical properties. Using this method, SDR was found to have a higher BFS than TEC [32,38] and a similar value to that of FBF [28,37] 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 [37,38] 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.

When the effects of polymerization characteristics on mechanical properties are studied, tests in which single-shot curing protocols are applicable, the biaxial flexure strength are indicated [6,12,38]. BFS of two bulk-fill composites (X-tra base, Voco; and SDR; Dentsply) was evaluated at different depths up to 8 mm. The values found for the SDR composite (178 MPa at 1 mm and 151 MPa at 4 mm) were in agreement with the results obtained in the present study, with no statistically significant difference at depths up to 4 mm. An increase in the depth to 8 mm did, however, result in a measurable difference in the BFS value compared to the valued obtained at 1 mm. These findings were also supported by a degree of conversion analysis conducted by the same authors, which revealed no difference in the degree of conversion at depths of 1 and 4 mm [12].

These 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]. It is important to emphasise that in clinical situations, the light can be easily attenuated by distance and 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 requires to ensure the adequate clinical performance of bulk-fill composites.

5. Conclusion

In general, 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. Furthermore, the LT decreased as the thickness increased for both the regular and bulk-fill composites. Notably, only SDR had a higher light transmittance than HER. However, light attenuation did not influence BFS of bulk-fill composites, while HER presented decreased BFS at greater depths.

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6. Appendices

Table 1. Materials evaluated and respective manufacturers' information.

Code	Composite Resin	Manufacturer (lot number)	Matrix composition	Filler type	Filler loading (%, by volume)	Shade
HER	Herculite Classic	Kerr Co, Orange, CA, USA (4009366)	Bis-GMA, TEGDMA	Borosilicate-aluminum glass	59	A2
SDR	Surefil 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 diglycidil ether dumethacrylate; Bis-EMA, ehoxylated bisphenol-A dimethacrylate; EPDMA, ethoxylated bisphenol-A dymethacrylate; PMMA, polymethyl methacrylate;TEGDMA, triethylenegycol dimethacrylate; UDMA, urethane dimethacrylate.

Table 2. Median (minimum and maximum values) for light transmission (%).

Depth (mm)	Material				
	HER	SDR	FBF	TEC	EXP
0	100 (100-100) Aa	100 (100-100) Aa	100 (100-100) Aa	100 (100-100) Aa	100 (100-100) Aa
1	21.2 (20.7-21.8) Bab	38.5 (37.5-40.1) Aab	31.3 (30.8-32.1) Aab	27.1 (26.8-28.3) ABab	28.6 (22.1-31.5) ABab
2	10.9 (10.7-11.5) Babc	23.3 (23.2-23.4) Aabc	16.0 (14.7-17.6) ABabc	15.2 (14.3-15.9) Babc	16.6 (15.0-17.3) ABabc
3	5.7 (5.2-5.8) Bbc	15.6 (15.0-15.6) Abc	8.7 (8.2-8.7) ABbc	8.9 (8.7-9.4) ABbc	8.8 (8.2-9.0) ABbc
4	2,5 (2.3-2.7) Bc	9.2 (8.7-9.5) Ac	4.2 (4.1-4.7) ABc	4.4 (4.4-4.4) Ac	4.9 (4.4-5.4) Ac

Means followed by the same letter (uppercase compare rows and lowercase compare columns) are statistically similar ($p > 0.05$)

Table 3. Means (standard deviation) for flexural strength (MPa).

Depth (mm)	Material				
	HER	SDR	FBF	TEC	EXP
0.5	173.8 (31.6) Aa	148.7 (12.3) Aa	171.2 (24.7) Aa	76.3 (9.5) Ca	103.4 (8.0) Ba
1	165.3 (35.8) Aa	149.4 (22.4) Aa	171.9 (17.1) Aa	77.2 (14.3) Ba	102.6 (14.0) Ba
1.5	174.9 (31.3) Aa	152.2 (14.7) Aa	170.5 (23.8) Aa	78.5 (12.7) Ca	106.7 (10.6) Ba
2	167.6 (31.8) Aa	148.9 (13.3) Aa	157.0 (28.2) Aa	79.6 (8.6) Ca	106.9 (7.6) Ba
2.5	141.5 (27.7) Aab	151.9 (25.1) Aa	150.9 (27.1) Aa	79.4 (3.8) Ba	103.5 (14.1) Ba
3	137.0 (26.4) ABab	148.4 (9.8) Aa	143.1 (22,6) Aa	77.1 (11.0) Ca	104.3 (9.8) Ba
3.5	147.3 (33,2) Aab	152.1 (15.6) Aa	148.7 (12.3) Aa	75.0 (8.1) Ca	103.9 (14.2) Ba
4	124.1 (22.8) ABb	145.9 (16.1) Aa	149.4 (22.4) Aa	76.2 (11.0) Ca	104.1 (11.9) Ba

Means followed by the same letter (uppercase compare rows and lowercase compare columns) are statistically similar ($p > 0.05$).

Table 4. Means (standard deviation) for flexural modulus (GPa).

Depth (mm)	Material					Tukey
	HER	SDR	FBF	TEC	EXP	
0.5	5.0 (1.3)	3.2 (0.4)	3.7 (0.7)	3.7 (0.4)	4.5 (0.9)	a
1	4.8 (0.5)	2.6 (0.3)	3.7 (0.7)	3.9 (0.9)	4.4 (1.1)	ab
1.5	5.0 (1.2)	2.5 (0.5)	3.4 (0.3)	4.2 (1.1)	4.3 (0.8)	ab
2	5.3 (1.2)	2.5 (0.4)	3.9 (0.9)	3.6 (0.1)	4.5 (0.5)	ab
2.5	4.8 (0.8)	2.4 (0.3)	3.0 (0.6)	3.5 (0.9)	4.7 (1.0)	ab
3	5.1 (0.7)	2.2 (0.2)	3.0 (1.3)	3.3 (0.8)	4,5 (1.0)	ab
3.5	5.3 (1.1)	2.2 (0.4)	2.8 (2.6)	3.1 (0.8)	4.6 (0.6)	ab
4	5.1 (0.5)	2.3 (0.2)	2.6 (0.2)	2.8 (0.5)	4.5 (1.1)	b
Tukey	A	D	C	C	B	

Means followed by the same letter (uppercase compare rows and lowercase compare columns) are statistically similar ($p > 0.05$).

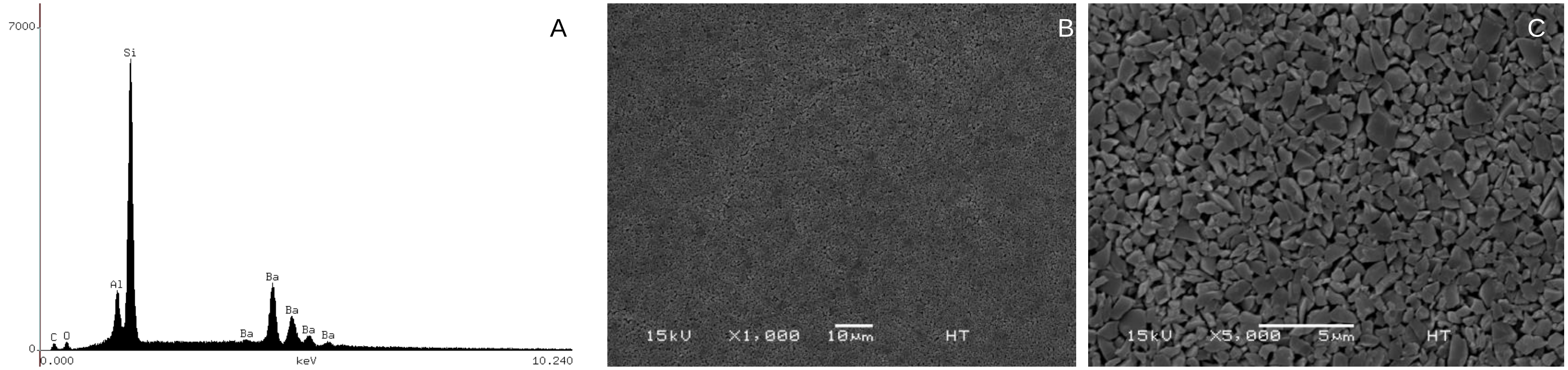

Figure 1. Elements identified by EDX analysis for HER (A) and SEM micrographs: original magnification 1000x (B) and 5000x (C).

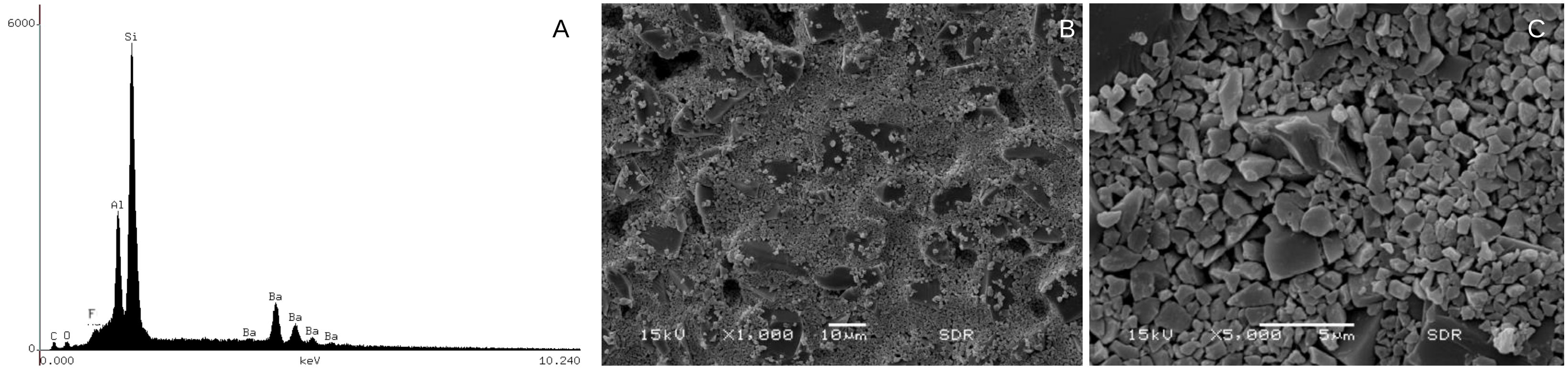

Figure 2. Elements identified by EDX analysis for SDR (A) and SEM micrographs: original magnification 1000x (B) and 5000x (C).

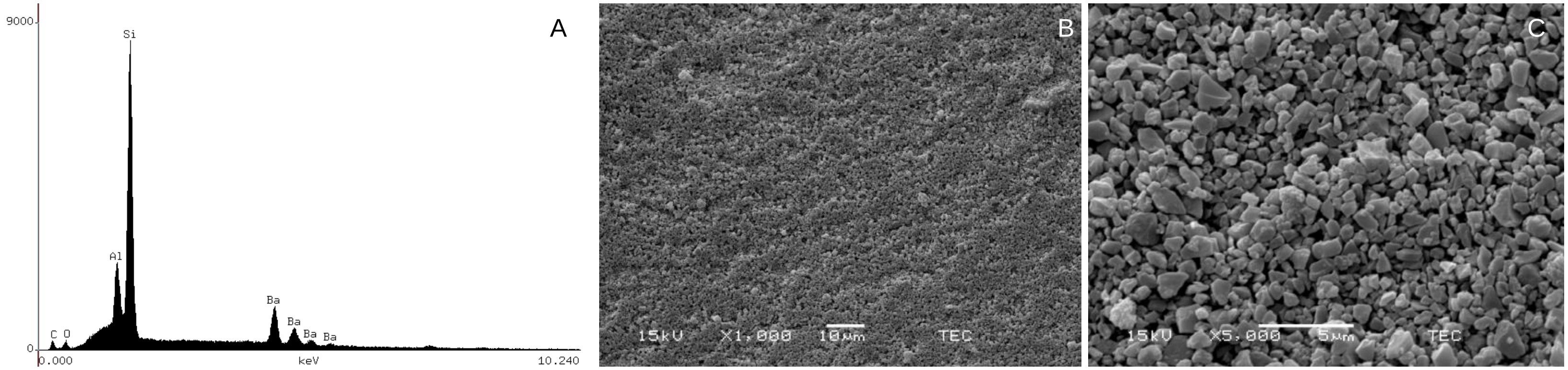

Figure 3. Elements identified by EDX analysis for TEC (A) and SEM micrographs: original magnification 1000x (B) and 5000x (C).

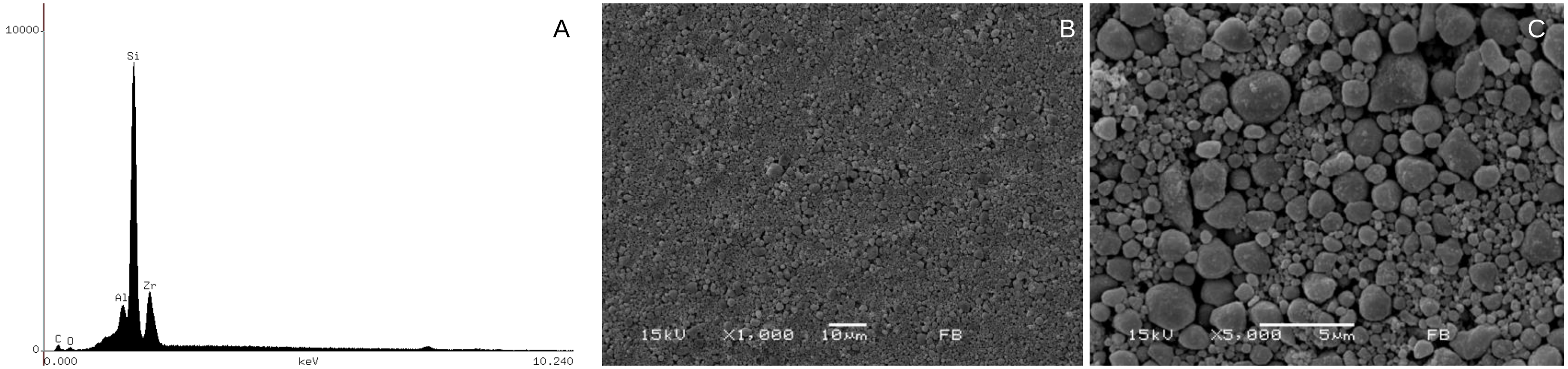

Figure 4. Elements identified by EDX analysis for FBF (A) and SEM micrographs: original magnification 1000x (B) and 5000x (C).

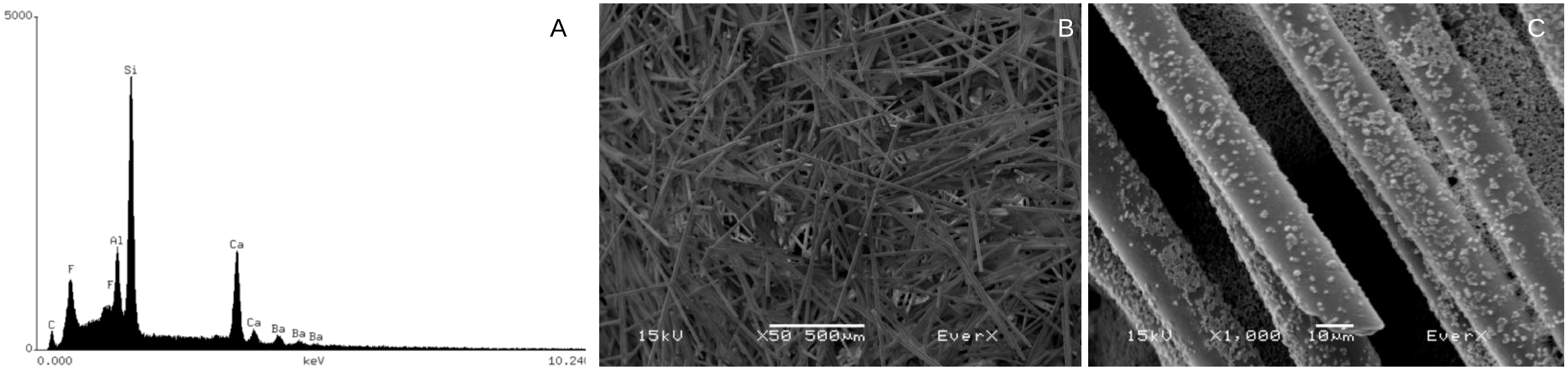

Figure 5. Elements identified by EDX analysis for EXP (A) and SEM micrographs: original magnification 50x (B) and 1000x (C).

7. Acknowledgements

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CAPÍTULO 2

Polymerization efficiency, marginal adaptation and shrinkage stress of bulk-fill resin composites

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Abstract

Objectives: To evaluate the degree of conversion (DC), Knoop microhardness (KHN), internal adaptation (IA) and polymerization shrinkage stress (PS) of one regular and four bulk-fill composites. Methods: Bulk-fill composites tested were Surefill SDR (SDR), Filtek Bulk-Fill (FBF), Tetric EvoCeram Bulk-Fill (TEC) and EverX Posterior (EXP). Herculite Classic (HER) was tested using both bulk and incremental insertion techniques. Standardized class I cavities (4-mm depth) were prepared in extracted molars and filled with restorative systems (n = 5). After one-week storage, restorations were cross-sectioned and the DC and KHN were evaluated at four depths using confocal Raman spectroscopy and a KHN tester, respectively. Epoxy resin replicas of the sectioned restorations were observed using scanning electron microscopy to evaluate the IA, and the PS was determined using acrylic rods attached to a universal testing machine (n = 5). Results: Only SDR and FBF composites exhibited similar DC values at all of the analysed depths. The KHN values did not statistically differ at any of the tested depths, except for TEC composite. The highest and lowest PS values were exhibited by the EXP and TEC composites, respectively. The incremental HER and bulk-fill SDR and TEC restorations exhibited a lower incidence of internal gaps. Significance: The DC values were not uniform with depth for all of the bulk-fill materials tested, while the KHN values did not significantly differ. Furthermore, a higher PS corresponded to a higher percentage of interfacial gaps and the incremental technique reduced gap formation.

Keywords

Bulk-fill, dental composite, Confocal Raman spectroscopy, degree of conversion, Knoop microhardness, polymerization stress, gap formation.

1. Introduction

The main reasons for clinical failure of resin composite restorations are secondary caries and fractures [1,2]. The former are related to early gap formation and degradation of the structures involved in restoration bonding: the dentin, adhesive and composite itself. Gap formation may be associated with volumetric changes in resin-based materials during polymerization reaction and polymerization shrinkage stress (PS) at the interfacial bond [3]. The bonding agent efficiency and quality of placement also influence the longevity of restorations [4]. Furthermore, the fracture strength of resin composite restorations is limited by the mechanical properties of the material which depend not only on the material composition but also on the extent of cure, amount of supportive tooth structure, cavity design and specific occlusion [5,6]. Incremental filling techniques have been proposed to ensure efficient composite polymerization and internal adaptation using 2-mm-thick oblique or horizontal increments [7-9].

These procedures are thought to reduce the final volumetric shrinkage of the material and thus the PS[5]. Clinically, however, incremental filling techniques are complex and require a long time for restoration placement. Recent advances in dental resin-based restorative materials have led to the development of composites for bulk placement [10]. Manufacturers claim that these composites have reduced volumetric shrinkages and PS and increased depths of cure, allowing for single placements in up to 4 mm increments. Bulk-fill composites thus have the advantages of a simplified process that takes less time. Furthermore, bulk placement prevents void incorporation and contamination between layers, leading to more compact fillings [11]. Such advantages are possible because of the increased translucency of the bulk-fill composites, which allows greater light transmission [12], modulation of the polymerization reaction and the use of more reactive photoinitiators and different types of fillers such as pre-polymer particles and fiberglass.

Several studies have demonstrated favourable outcomes for bulk-fill composites with respect to their depth of cure. These studies have used spectroscopic methods, such as Fourier transform infrared [13-15] and Raman spectroscopy [11,16-17], to determine the degree of conversion (DC). Microhardness measurements have also been used as an indirect method for evaluating the DC [14,17-19]. However, the effectiveness of this method for assessing the depth of cure of bulk-fill composites has been questioned because it overestimates the depth of cure [20] Experimental data for bulk-fill composite shrinkage are controversial because different methods and analytical techniques have yielded different results [18,21-22]. Few studies have focused on the PS of bulk-fill composites and its potential influence on clinical outcomes [19,21,23-24]. One clinical evaluation was found to compare a bulk-fill composite to a conventional 2-mm-thick resin composite after 3 years. The results indicated that the bulk-fill composite performed effectively [25]. However, before the change from the traditional incremental filling technique to bulk filling can occur, both additional clinical trials and laboratory studies designed to evaluate the characteristics of the polymerisation reaction and the physical properties in a simulated clinical setting are required.

The purposes of this study were to evaluate internal marginal adaptation of composite restorations, DC and MH at different depths of bulk-fill and conventional composites of restorations placed in high configuration factor cavities. And also, measure the PS of these materials. The null hypotheses tested were: (1) there would be no differences in DC or KHN of bulk-fill composites evaluated at different depths; (2) bulk-fill composites would not present similar PS and gap formation compared to a regular composite.

2. Materials and Methods

Five resin-based composites were investigated: one regular composite (Herculite Classic - HER) applied both incrementally (positive control) and in bulk (negative control), 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). For each material, the respective adhesive system was used in the restorative procedures. All material specifications are presented in Table 1.

A polywave light emitting diode curing unit (VALO, Ultradent Products Inc., South Jordan, UT, USA) was used for all of the light-curing procedures. A light irradiance of at least 1000 mW/cm^2 was applied as measured using a laboratory-grade spectral radiometer (DAS 2100, Labsphere, N. Sutton, NH, USA). The composites were light-cured as recommended by the manufacturers: 20 s for the bulk-fill composites and 40 s for the regular sample.

2.1. Cavity preparation and restoration placement

Thirty freshly extracted caries-free human third molars were collected and stored in an aqueous 0.2% thymol solution at 4°C for up to 1 month after extraction. The teeth were obtained and used in accordance with a protocol (#015/2014) approved by the Ethics Committee Research of Piracicaba Dental School, State University of Campinas.

First, the cusps were abraded with silicon carbon sandpaper (grit #320) in order to obtain a flat enamel occlusal surface. Standardized Class I cavities (mesio-distal width: 4.0 mm; bucco-lingual width: 3.0 mm; depth: 4.0 mm; C-factor: 4.5; volume: 48 mm^3) were then prepared using #3145 diamond burs of regular and fine granulation (KG Sorensen, Cotia, SP, Brazil) with a high-speed handpiece (Kavo, Joinville, SC, Brazil) with water cooling. The cavity margins were surrounded by enamel. The prepared teeth were divided randomly into 6 groups (n = 5) according to the restorative system and filling technique.

The adhesive systems were applied following the manufacturers' instructions. The restorative systems (composite/adhesive) consisting of Herculite Classic / OptiBond, Surefill SDR Flow / XP Bond, and Tetric EvoCeram Bulk Fill / Tetric N-Bond were applied using the total-etch bonding technique, while in the Filtek Bulk Fill / Scotchbond Universal and EverX Posterior / G-aenial Bond systems, the adhesives were self-etching systems. The Herculite Classic composite was tested using two filling techniques: incremental and bulk-filling. In the incremental oblique technique (HER incremental), the resin composite was applied in four increments (with a thickness of approximately 2.0 mm each), and each layer was individually light-cured for 40 s. For bulk-filling, 4 mm of the HER composite was light-cured for 40 s. For bulk-filling of the bulk-fill resins (SDR, FBF, TEC and EXP), the cavity was filled in one single increment and light-cured for 20 s.

After storage for one week at 37°C in distilled water, the restorations were cross-sectioned through their centres in the buccal-lingual direction using a diamond blade (Buehler Ltd., Lake Buff, IL, USA) with water cooling. Both halves were polished using silicon carbon sandpaper (grits #1000, #1200 and #2000, Norton Abrasivos, Vinhedo, SP, Brazil) and felt disks with 3, 2, 1 and 0.5 μm diamond pastes (Buehler Ltd., Lake Buff, IL, USA). Specimens were then sonicated (Thornton USC 1400, Unique Group, Indaiatuba, SP, Brazil) for 10 minutes in order to remove the polishing debris.

The restored teeth were divided for evaluation using different analytical techniques: one half was subjected to confocal Raman spectroscopic analysis and Knoop microhardness testing (non-destructive analyses), and the other half was used to evaluate the internal marginal adaptation of the restorations via scanning electron microscopy (SEM).

2.1.1. Confocal Raman spectroscopy

The composite resin restorations were analysed using confocal Raman spectroscopy in order to determine their DC values at different depths. Three measurements were performed at 1, 2, 3 and 4 mm. Raman spectra were collected using a spectrometer (Skin Analyzer–model 3510, River Diagnosis BV, Rotterdam, Netherlands) with a diode laser at a wavelength of 785 nm. The software (River Icon, River Diagnosis BV, Rotterdam, Netherlands) generated three spectra, each with an exposure of 10 s and laser penetration into the composite surface of 150 μm. To obtain spectra of the uncured materials, the composite resins were each placed in an aluminium rod sample holder and its spectrum collected following the same procedure.

Raman data were analysed over the wavelength range from 1570 and 1660 cm^{-1}. The spectra were processed (minimum-maximum normalisation, background correction and range selection) using Opus software (OPUS v. 4.2, Bruker Optik GmbH, Ettlingen, Germany). The Raman vibrational stretching modes at 1610 and 1640 cm^{-1} were fitted with Lorentzian shapes in order to obtain the height of the peaks using Microcal Origin software (Microcal Software Inc., Northampton, MA, USA). The DC values were calculated from the ratios of the peak heights for the aliphatic C=C bond (1640 cm^{-1}) and the aromatic C=C bond (1610 cm^{-1}) in the spectra of the cured and uncured specimens using the following equation: DC (%) = 100 × [1 − (Rcured /Runcured)], where: R = the peak height at 1640 cm^{-1}/peak height at 1610 cm^{-1}.

2.1.2. Knoop microhardness

The same specimen locations analysed using confocal Raman spectroscopy were also used to determine the KHN values. A microhardness tester (HMV 2000, Shimadzu, Tokyo, Japan) with a Knoop diamond indenter was used to apply a static load of 100 g (0.98 N) for 10 s to each composite surface. For each specimen, the averages of three indentations for each depth were used in the statistical analysis.

2.3. Interfacial gaps

Moulds of the polished half-restored surfaces were taken using polyvinyl siloxane with light and heavy consistency materials (Express XT, 3M ESPE, St. Paul, MN, USA) and impressions were poured with epoxy resin (Buehler Ltd., Lake Buff, IL, USA). The obtained replicas were sputter-coated with gold to a thickness of approximately 50 Å in a vacuum evaporator (SCD 050, Bal-Tec AG, Balzers, Liechtenstein) in order to identify any gaps that might be present using SEM (JEOL, JSM-5600LV, Tokyo, Japan).

For each specimen, approximately 40 images at a 200Å~ magnification were required to scan the entire perimeter of the restoration. ImageJ software (National Institute of Health, Bethesda, USA) was used to determine the length of the debonded segments at the composite margins and over the entire perimeter of the restoration. The scale bar of the SEM images was used for calibration. The initial values obtained in millimetres were converted to percentages based on the total specimen interface perimeter.

2.2. Polymerization stress

Polymethyl methacrylate rods with a diameter of 4 mm were sectioned into lengths of 13 and 28 mm. One surface of the 13 mm rods was polished using silicon sandpaper (grits #600, #800, #1000 and #1200, Norton Abrasivos, Vinhedo, SP, Brazil) and felt disks with 1 µm diamond paste (Buehler Ltd., Lake Buff, IL, USA) in order to allow the transmission of light through the rod during photoactivation. The opposite surfaces of the 13 mm rods and both the surfaces of the 28 mm rods were sandblasted using 100 µm aluminium oxide particles (BioArt, São Carlos, SP, Brazil). Methylmethacrylate monomer (JET Acrílico AutoPolimerizante, Artigos Odontológicos Clássico, SãoPaulo, SP, Brazil) was then applied on the sandblasted surfaces followed by two layers of unfilled resin (Scotchbond Multipurpose Plus, bottle 3, 3M ESPE, St. Paul, MN, USA) and light-cured for 10 s.

The rods were subsequently attached to a universal testing machine (model 5565, Instron Corp, Canton, MA, USA). The13 mm rods were attached to the lower clamp and the 28 mm rods to the upper clamp, with the space between them fixed at 0.8 mm, resulting in a composite volume of 6.8 mm^3 (C-Factor: 1.3). Each composite was inserted into this space and shaped as a cylinder following the perimeter of the rods. An extensometer was attached to the rods in order to monitor the specimen height and provide feedback to the testing machine (model 2630-101, Instron Corp, Canton, MA, USA) and move the actuator such that the specimen height was maintained within a minimum range. The tip of the light-curing unit was positioned in contact with the polished surface of the 13 mm rod and turned on. The values registered by the load cell corresponded to the force necessary to counteract the polymerization shrinkage force and maintain the initial specimen height. Development of the force was monitored for 10 min from the beginning of light-activation and the maximum nominal stress was calculated (MPa) by dividing the maximum force value recorded for the cross-section of the rods. Five specimens were tested for each material.

2.3. Statistical analyses

The DC and KHN data were analysed using the split-plot one-way ANOVA (factor: depth) method, while the interfacial gap and PS data were evaluated using one-way ANOVA (factor:material) tests. In both cases, Tukey post-hoc tests were applied in order to detect any differences among the groups ($\alpha = 0.05$).

Pearson’s test was also used to verify the presence of statistically significant correlations between the DC and KHN data and between the results for the internal gaps and polymerization stress, with a global significance level of 5%.

3. Results

3.1. Confocal Raman spectroscopy

The mean DC (%) values and standard deviations are presented in Table 2. Evaluation using a one-way ANOVA test demonstrated that the depth ($p < 0.0001$) influenced the DC results for the composites. Interestingly, the DC values were not influenced by the composite depth for the SDR and FBF bulk-fill composites and the HER composite applied incrementally, but were statistically lower at 4 mm than at 1 mm for the bulk-filled TEC, EXP and HER composites. Furthermore, the DC value for the TEC sample decreased significantly from the surface to a depth of 2 mm, while that of the EXP composite decreased when the depth changed from 1 to 3 mm.

3.2. Knoop microhardness

Table 3 presents the KHN results. One-way ANOVA testing revealed that the depth affected the KHN at least for the TEC composite ($p = 0.0010$), which exhibited low KHN values at depths of 3 and 4 mm compared to that at 1 mm. Conversely, the HER, FBF, SDR and EXP composites yielded KHN values with no statistically significant differences at all of the depths. Pearson's correlation analysis between the DC and KHN values also did not indicate any statistically significant interaction between these variables ($p = 0.0892$ and $r = -0.16$).

3.3. Polymerization stress

The PS results are presented in Table 4. One-way ANOVA testing revealed statistically significant differences among the materials ($p < 0.0001$). The EXP composite had the highest PS (4.3 MPa), while TEC exhibited the lowest value (2.6 MPa). The SDR, FBF and HER composites all exhibited intermediate average values that were statistically similar to each other.

3.4. Interfacial gaps

The percentages of discontinuous interfaces detected for each material are also presented in Table 4. Statistically significant differences among the materials were identified via one-way ANOVA testing ($p < 0.0001$). The highest incidence of gaps was observed for the EXP composite (65.9%); however, it did not differ significantly from the FBF and bulk-filled HER samples. The SDR and TEC composites had intermediate percentages of gaps. The HER composite placed incrementally has the lowest gap incidence (25.8%), which was significantly different from the results for the EXP, FBF and bulk-filled HER composites.

Differences in the gap patterns and sizes were also observed in the micrographs of the composites (Figure 1). In the HER composite formed incrementally, approximately 20 μm gaps were mainly located at the cavity angles, while in the bulk-filled HER, 100-μm-long gaps were located at the pulpal wall and

10-μm-long gaps were present in the surrounding surfaces, and in some cases the entire mesial or distal face was debonded. Gaps no larger than 15 μm were observed in all of the internal cavity walls of the EXP composite. The FBF sample also had gaps in all of the walls, but their lengths varied from 5 to 80 μm. In both the TEC and SDR composites, the gaps were at the pulpal walls and were approximately 30 and 50 μm in length, respectively. Linear regression (Pearson's correlation) revealed a direct relationship between PS and percentage of gaps, but only for the composites inserted using the bulk-fill mode (p = 0.0004, r = 0.6507).

4. Discussion

The first hypothesis that no differences would be observed in the DC or KHN values at the evaluated depths for the bulk-fill composites was rejected. In addition to the incrementally applied HER composite, only the SDR and FBF exhibited uniform DC values along the 4-mm deep restoration, while all of the materials had similar KHN values at all depths, including the HER placed in bulk, except for the TEC composite.

The polymerization process for resin-based composites is dependent on several factors. At a given light irradiation, the DC is defined by the monomer composition and ratio, filler content and photoinitiator type and concentration [26]. Because the curing light is attenuated by composite absorption and scattering, the depth of cure depends on the ability of the material to transmit light, as well as the reaction kinetics [10,12]. The DC of the conventional composite resin (HER) varied depending on the filling mode. When placed incrementally, no statistically significant difference was observed in the DC values at different depths. This result was expected since each increment received the same irradiance as that of the entire bulk-filled restoration of bulk filling, and higher light exposure times increase DC of resin composites [26]. Furthermore, the HER composite has a higher filler loading, leading to a greater number of particle/resin matrix interfaces, which may result in increased light scattering because of the difference in their refractive indices[12]. Therefore, fewer photons would reach the deep layers of the bulk-filled composite, and consequently, a lower DC value would be obtained.

Among the bulk-fill composites, only the SDR and FBF exhibited uniform polymerization over the entire 4 mm depth of the restorations. In part, the differences in the depth of cure and even the overall degree of conversion may be attributed to the viscosities of the composites in the uncured state. The viscosity is influenced by the monomer composition and the filler content and is an important parameter with respect to the reaction kinetics and final DC because it affects the mobility of the reactive species [27]. Both the SDR and FBF composites are flowable materials with modified monomers and relatively low filler loadings. The SDR composite also features a photoactive group embedded in urethane-based methacrylate monomers that are capable of interacting with camphoroquinone. The manufacturer claims that such an interaction helps modulate the curing and control the polymerisation stress because of its higher flexibility and also leads to more homogeneous network formation [15,18]. FBF, in turn, contains procrylat resins and Bis-EMA, which has a high molecular weight and low viscosity compared to Bis-GMA because of the absence of hydroxyl groups in its structure [26]. Dental polymers based on Bis-EMA and containing lower viscosity urethane monomers tend to exhibit higher DC values than typical Bis-GMA/TEGDMA resins [14].

Conversely, the lower DC value at the bottom of EXP and TEC restorations may be explained on the basis of their high filler contents, which decrease the translucency of these composites. A lower polymerization depth was obtained because the lower mobility of the reactive sites [11,28]. Moreover, although a photoinitiator system based on dibenzoyl germanium, which is excited by ultraviolet light (380–450 nm), was present in the TEC composition, it did not lead to a higher DC value or greater depth of cure for this material because ultraviolet light only weakly penetrated the interior of the composite [29].

No correlation was found between the DC and KHN values. It has been recently reported that the ISO 4049 and KHN tests overestimate the depth of cure [24]. As expected, in the present study, the KHN values were proportional to the filler content, except for the TEC composite. This result can be explained by the presence of pre-polymer particles in this composite, which have been shown to result in inferior hardness values [30,31]. These findings corroborate the results of a previous investigation of the DC, Vickers microhardness, filler fraction and flexural strength and modulus of bulk-fill composites, which demonstrated that the DC had a greater influence on the flexural strength, while the KHN showed a high correlation with the filler fraction [17]. It is important to mention, however, that the DC value does not completely characterize the network structure, as polymers with similar DC values may have different cross-link densities because of differences in the linearity of the chains, which is largely influenced by the reaction kinetics [32]. In fact, the actual mechanical properties of composites are more relevant to the clinical performance of restorations than their DC values. Therefore, the finding that not all of the bulk-fill composites exhibited homogeneous DC values up to 4 mm may be of less relevance than the results of the KHN tests.

It should also be noted that sample preparation for Raman analysis was a concern because any uncured monomers could have dissolved and leached out during sectioning and processing, which would result in falsely high DC and KHN values. In order to avoid such composite alterations, confocal Raman spectroscopy was chosen as the method for calculating the DC values since the spectra are obtained at a sample depth of 150 µm, and thus the analysis at the surface is avoided. Pilot tests were performed to ensure that the cut and polishing protocols used in the study would not interfere with the results.

The second hypothesis was also rejected, as not all of the bulk-fill composites had PS values similar to that of the regular composite. High PS values have been observed for composites with high volumetric shrinkage and high elastic moduli [6]. Stress development is also affected by the polymerisation reaction kinetics. As the resin composite plastic deformation is time-dependent, slower curing rates may provide extended periods during which the composite can be affected by contraction forces before a higher elastic modulus is realized [33]. The higher PS values of the EXP composite may be explained by its high in organic content and resultant high elastic modulus, as demonstrated previously [17,34]. The SDR and FBF composites exhibited intermediate PS values that were comparable to those of the HER composite. Flowable composites typically experience higher volumetric shrinkage in large part because of their reduced inorganic content. Furthermore, the use of low molecular weight diluent monomers increases the density of the polymerizable carbon bonds, which may lead to more shrinkage [33]. Furthermore, mobility in the reaction environment is increased due the lower viscosity, thus allowing more efficient conversion [35]. Considering these factors, it is reasonable to conclude that the approaches taken by the manufacturers to reduce the stress in these two bulk-fill composites are fairly successful.

Studies have demonstrated that bulk-fill composites have higher volumetric shrinkage than conventional composites, even though their PS values are lower [19,22]. When the PS values of bulk-fill and conventional composites are compared, it is important to ensure that the specimen dimensions are similar. However, bulk-fill composites were developed to be cured in thicker increments than regular composites. As there is evidence that the composite volume is directly related to the PS magnitude [36], ideally, bulk-fill composites should have low PS values in laboratory evaluations. A statistically significant positive correlation between the PS values and the percentage of gaps was observed in the present study, which is in agreement with the results of previous investigations [37,38]. High PS values are the main cause of gap formation along the interface, although a low-quality bonding system will also contribute to debonding. Interestingly, in the EXP restoration, despite the high percentage of debonding areas, the gaps were relatively small at approximately 15 μm because during polymerization the material was not able to shrink along the length of the fiber. It retains its original dimensions horizontally, although the resin matrix tried to shrink in the vertical direction [39]. The same behaviour was not observed for the TEC composite, despite its high inorganic content.

In fact, the TEC composite exhibited a lower PS value and a low percentage of gaps. According to the manufacturer, the formulation includes a shrinkage stress reliever, and as a result, the silanized filler features a lower modulus of elasticity that acts like a microscope spring, attenuating the forces generated during shrinkage [22]. Additionally, composite resins with pre-polymerized filler particles typically exhibit relatively low elastic moduli [30]. A moderate value for the elastic modulus of the TEC composite has been previously reported [17]. Furthermore, the lower DC of this material certainly influenced the PS. The DC of a polymeric matrix simultaneously affects the volumetric shrinkage and elastic modulus, thus materials with high a DC experience less deformation because enhanced polymer chain entanglement and a high crosslinking density hinder movement in the polymer network [33,39].

Few studies exist in the literature regarding the internal adaptation for bulk-fill composites and most have involved the evaluation of Class II restorations. When the environment is taken into consideration, the C-factor, restorative material volume, cavity geometry and angles formed at the cavosurface margin as well as their interactions influence the stress distribution [40].

It has been suggested that shrinkage vectors point towards the restoration occlusal surface, resulting in composite debonding at the pulpal wall [24,41-42]. Similar outcomes have been reported for the TEC and SDR composites when using the adhesive OptiBond FL [23]. In contrast, a third study [25] revealed a lower gap percentage for SDR compared to TEC. G-aenial Bond has also been used to evaluate one flowable and one packable conventional resin [43]. The strength of the bond between this adhesive and the SDR composite was also evaluated for cavities with different configuration factors, and it was found that the filling technique and composite type had a significant impact on the adhesion of the composite, particularly for high C-factor cavities. While the bulk-fill SDR provided satisfactory bond strength regardless of the filling technique and cavity depth, the adhesive failed when conventional composites were bulk-filled [43]. Thus, among the many factors influencing gap formation, the quality of the adhesive bond is important for maintaining contact between the restoration and the cavity walls [21,41]. Self-adhesive systems were employed for the EXP and FBF composites, while and etch-and-rinse system was used for the TEC, SDR and HER composites. Adequate bond strength to dentin is important for resisting the volumetric shrinkage of restorative composites during and after the polymerization. Therefore, the gap incidence reported here is the result of the interaction between the efficacy of the adhesive system and the composite PS. This interaction may have contributed to the relatively low correlation coefficient (r = 0.6507) observed in this study for the relationship between the PS and gap formation.

5. Conclusion

The depth of cure dependente on material, in which only SDR and FBF exhibited uniform DC values over the entire depth of 4 mm. Furthermore, no correlation was found between the DC and KHN values. Only the bulk-fill TEC composite exhibited a lower PS than the regular composite. For bulk-fill composites, a higher PS corresponded to a higher percentage of interfacial gaps. Notably, lower gap percentages were observed for the incrementally placed conventional composite was placed incrementally, as well as bulk-filling with TEC and SDR composites.

6. Appendices

Table 1. Materials evaluated and respective manufacturers' information.

Composite resin (lot number)	Adhesive system (lot number)	Manufacturer	Matrix composition	Filler type	Filler loading (% by volume)	Shade
Herculite Classic (400936)	Gel Etchant (31297) OptiBond FL (31297)	Kerr Co, Orange, CA, USA	Bis-GMA, TEGDMA	Borosilicate-aluminum glass	59	A2
Surefill SDR flow (08153)	Etching Dental Gel (671857E) XP Bond (793475F)	Dentsply Caulk, Mildford, DE, USA	Modified UDMA, TEGDMA, EBPDMA	Barium-aluminofluoro-borosilicate glass, strontium-aluminofluoro-borosilicate glass	44	Universal
Filtek Bulk Fill (402919)	Scotchbond Universal (482988)	3M ESPE, St. Paul, MN, USA	Bis-GMA, Bis-EMA, UDMA, TEGDMA, Procrylat resins	Zirconia/silica, ytterbium trifluoride	42.5	A2
Tetric EvoCeram Bulk Fill (R04686)	Total Etch Gel (R39845) Tetric N-Bond (R27602)	Ivoclar Vivadent AG, Schaan, Liechtenstein	Bis-GMA, UDMA	Barium glass, ytterbium trifluoride, oxides and prepolymers	60	IVA
EverX Posterior (1401152)	G-aenial Bond (1205231)	GC Corporation, Tokyo, Japan	Bis-GMA, TEGDMA, PMMA	Hybrid filler fractions and E-glass fibers	57	Universal

Abbreviations: Bis-GMA, bisphenol-A diglycidil ether dimethacrylate; Bis-EMA, ethoxylated bisphenol-A dimethacrylate; EPDMA, ethoxylated bisphenol-A dymethacrylate; PMMA, polymethyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.

Table 2. Means (standard deviation) for degree of conversion (%).

Material	Depth (mm)			
	1	2	3	4
HER Incremental	75.5 (0.4) Ac	75.5 (0.4) Abc	75.7 (0.6) Ab	77.2 (1.1) Ab
HER Bulk-fill	76.3 (0.9) Abc	75.8 (0.4) ABbc	75.0 (0.6) ABb	74.4 (1.7) Bbc
SDR	81.3 (0.8) Aa	80.4 (1.5) Aa	79.8 (1.4) Aa	81.2 (0.7) Aa
FBF	76.2 (1.1) Abc	75.4 (1.4) Abc	75.0 (1.2) Ab	75.3 (0.5) Abc
TEC	75.2 (2.1) Ac	73.2 (2.5) Bc	71.0 (3.0) Cc	72.2 (2.4) BCc
EXP	79.5 (0.8) Aab	78.3 (1.1) ABab	77.2 (0.7) BCab	76.1 (0.5) Cb

Means followed by the same letter (uppercase compare rows and lowercase compare columns) are statistically similar ($p > 0.05$).

Table 3. Means (standard deviation) for microhardness (KHN).

Material	Depth (mm)			
	1	2	3	4
HER Incremental	76.4 (3.1) Aa	76.5 (4.6) Aa	74.6 (3.9) Aa	76,8 (4.4) Aa
HER Bulk-fill	76.3 (3.0) Aa	77.5 (4.6) Aa	75.0 (4.0) Aa	77,6 (3.6) Aa
SDR	52.3 (2.1) Ac	51.8 (2.2) Ac	51.4 (2.1) Ac	52,9 (1.9) Ac
FBF	45.1 (2.2) Ac	45.9 (1.0) Ac	46.2 (1.2) Ac	46,2 (1.0) Ac
TEC	66.8 (2.0) Ab	64.6 (1.8) ABb	61.0 (1.2) Bb	61,0 (1.5) Bb
EXP	72.8 (5.3) Aab	74.0 (2.4) Aa	70.1 (4.2) Aa	72,4 (5.8) Aa

Means followed by the same letter (uppercase compare rows and lowercase compare columns) are statistically similar ($p > 0.05$).

Table 4. Means (standard deviation) for PS (MPa) and internal gap formation (%).

Material	PS	Gap
HER Incremental	-	25.8 (4.0) c
HER Bulk-fill	3.7 (0.2) b	52.5 (17.8) ab
SDR	3.3 (0.2) b	43.0 (4.0) bc
FBF	3.5 (0.2) b	49.0 (8.6) ab
TEC	2.6 (0.3) c	32.9 (10.1) bc
EXP	4.3 (0.4) a	65.8 (10.7) a

Means followed by the same letter (uppercase compare rows and lowercase compare columns) are statistically similar ($p > 0.05$).

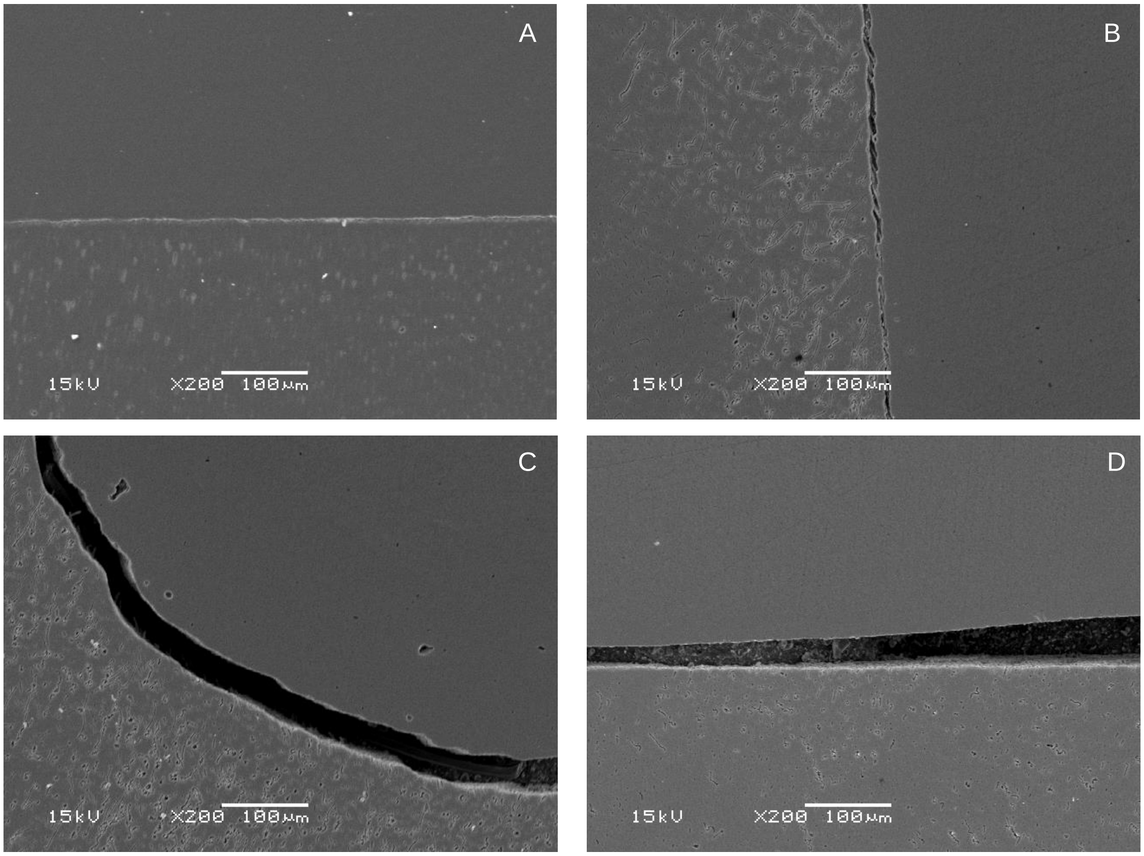

Figure 1. Examples of specimens showing the cavity wall adaptation of resin composite restorations. No gap formation at pulpal wall is observed at the dentin-composite interface for HER incremental (A). Small gaps and some areas where composite kept bond to dentin were noted in the surrounding walls for EXP (B). Gaps located at the internal angle of the cavity measuring approximately 40 µm for SDR (C). Presence of gap at pulpal wall with 30 to 50 µm size for TEC (D).

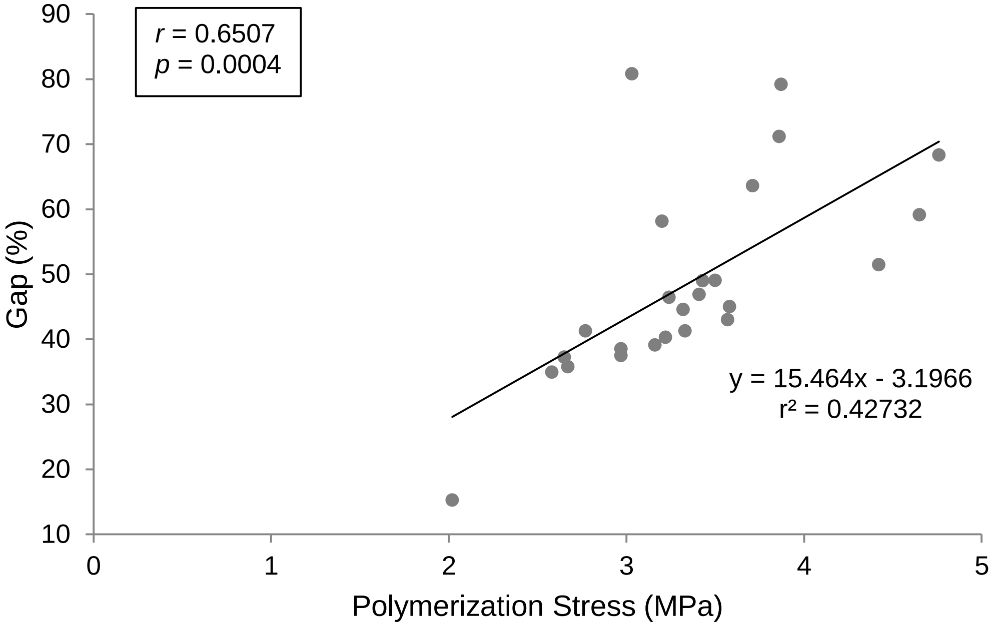

Figure 2. Regression analysis of polymerization stress vs. marginal gap.

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CONCLUSÃO

A partir dos resultados obtidos nos dois capítulos que compõe o presente estudo, é possível concluir que:

- As resinas compostas *bulk-fill* apresentaram partículas de carga de formato irregular, esferoidal ou cilíndrico, com tamanhos variando de 0,1 µm a 1 mm. Quanto à composição química, os principais elementos identificados foram alumínio, bário e silício. Algumas delas apresentaram características semelhantes à resina composta tradicional.
- O aumento da espessura do incremento de resina composta atenuou a transmissão de luz através do material. Não houve diferença de translucidez entre as resinas compostas *bulk-fill*, entretanto apenas Surefill SDR Flow permitiu maior passagem de luz que a resina composta tradicional para todas as espessuras de incremento avaliadas.
- A baixa irradiância nos incrementos mais profundos não comprometeu a resistência à flexão das resinas compostas *bulk-fill*, o que não ocorreu na resina composta tradicional. Herculite Classic demonstrou menor resistência à flexão com o aumento de profundidade de polimerização. Alguns materiais *bulk-fill* demonstraram resistência à flexão comparável à resina composta tradicional, embora essa ainda tenha apresentado o maior módulo de elasticidade.
- A profundidade de polimerização das restaurações foi dependente do compósito utilizado. Apenas Surefil SDR Flow e Filtek Bulk Fill demonstraram grau de conversão uniforme. Esses resultados não apresentaram correlação com os dados obtidos na análise de microdureza, uma vez que esta, de maneira geral, não detectou diferenças entre as profundidades.
- Quanto maior a tensão de contração de polimerização da resina composta, maior sua tendência de formar fendas entre o dente e a restauração, independente do sistema adesivo utilizado. A técnica

convencional de aplicação da resina em incrementos reduz a porcentagem de fenda marginal, entretanto SDR Surefil Flow e Tetric EvoCeram *bulk-fill* tiveram esse mesmo comportamento.

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ANEXOS

Anexo 1 – Certificado do Comitê de Ética em Pesquisa da FOP – UNICAMP

COMITÊ DE ÉTICA EM PESQUISA
FACULDADE DE ODONTOLOGIA DE PIRACICABA
UNIVERSIDADE ESTADUAL DE CAMPINAS

CERTIFICADO

O Comitê de Ética em Pesquisa da FOP-UNICAMP certifica que o projeto de pesquisa **"Avaliação das propriedades físicas e adaptação em cavidade classe I de sistemas restauradores indicados para aplicação em incremento único "**, protocolo nº 015/2014, dos pesquisadores Bruna Marin Fronza e Marcelo Giannini, satisfaz as exigências do Conselho Nacional de Saúde - Ministério da Saúde para as pesquisas em seres humanos e foi aprovado por este comitê em 02/04/2014.

The Ethics Committee in Research of the Piracicaba Dental School - University of Campinas, certify that the project **"Evaluation of physical properties and adaptation in class I cavities of bulk fill restoration systems"**, register number 015/2014, of Bruna Marin Fronza and Marcelo Giannini, comply with the recommendations of the National Health Council - Ministry of Health of Brazil for research in human subjects and therefore was approved by this committee on Apr 02, 2014.

Prof. Dr. Felippe Bevilacqua Prado
Secretário
CEP/FOP/UNICAMP

Livia M. A. Tenuta
Profa. Dra. Lívia Maria Andaló Tenuta
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