

Evaluation of Physical-Mechanical properties of different Bulk-Fill composites

Avaliação das propriedades Físico-Mecânicas de diferentes compósitos Bulk-Fill

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ABSTRACT

The aim of the study was to assess the degree of conversion (DC), microhardness, polymerization shrinkage stress (PSS) and volumetric contraction of two bulk-fill composites (SonicFill, Kerr Corporation, Orange, CA, USA - (SF) and Filtek BulkFill, 3M ESPE, St Paul, MN, USA - (FB)) and one conventional nanofiller resin composite (Z350XT – 3M ESPE, St Paul, MN, USA) inserted incrementally (Z350I) or in bulk (Z350B). Micro-Raman spectroscopy, Knoop microhardness, PSS test and micro-computed tomography technique (μ CT) were used to evaluate the resin composites. Data were analyzed with one-way ANOVA followed by Tukey's post hoc test ($\alpha = 0.05$). DC was not affected by the type of the composite, excepted when conventional resin composite was inserted in a single increment ($p = 0.002$). FB and Z350I showed higher microhardness values than Z350B ($p = 0,003$), while SF showed no statistically significant difference to the other resin composites. No statistical differences were found among the composites regarding to PSS ($p = 0.104$) and volumetric shrinkage ($p = 0.258$). Therefore, it can be concluded that bulk-fill and conventional resin composites tested present similar properties. Thus, shorter time required for the use associated with simplified operative technique are considered clinical advantages of bulk-fill composites.

Keywords: Resin composite, Bulk-fill technique, Degree of Conversion, Polymerization shrinkage, micro-computed tomography.

RESUMO

O objetivo do estudo foi avaliar o grau de conversão (GC), microdureza, tensão de contração de polimerização (TCP) e contração volumétrica de dois compósitos bulk-fill (SonicFill (SF) – Kerr Corporation, Orange, CA, USA e Filtek BulkFill (FB) – 3M ESPE, St Paul, MN, USA) e um convencional (Z350XT - 3M ESPE, St Paul, MN, USA) inserido de forma incremental ou em incremento único (Z350U). Espectroscopia micro-Raman, microdureza Knoop, teste de contração de polimerização e técnica de micro tomografia computadorizada (μ CT) foram usados para avaliar os compósitos resinosos. Os dados foram analisados com ANOVA one-way seguida do teste post hoc de Tukey ($\alpha = 0,05$). O GC não foi afetado pelo tipo de compósito, exceto quando o compósito convencional foi inserido em um único incremento ($p = 0,002$). FB e Z350I apresentou valores de microdureza maiores que Z350U ($p = 0,003$), enquanto SF não apresentou diferença estatisticamente significativa para as outras resinas compostas. Não foram encontradas diferenças estatísticas entre as resinas compostas quanto ao TCP ($p = 0,104$) e contração

volumétrica ($p = 0,258$). Portanto, pode-se concluir que os compósitos bulk-fill e convencional testados apresentaram propriedades semelhantes. Assim, o menor tempo necessário para o uso associado à técnica operatória simplificada são considerados vantagens clínicas dos compósitos bulk-fill.

Palavras-chave: Resina Composta, Técnica De Bulk-Fill, Grau De Conversão, Contração De Polimerização, Microtomografia Computadorizada.

1 INTRODUCTION

The direct light-cured resin composites are considered good material choice for esthetic restorations in posterior teeth. However, clinical failures could be related to the limitations of the mechanical properties of the resin composites such as the inherent volumetric shrinkage caused by the polymerization and the development of polymerization stress (1). The stress may develop marginal gaps at the tooth/restoration interface, which can result in adhesive defects, such as postoperative sensitivity and restoration fracture (2). The longevity of restorations could be also associate with operative technique, cavity configuration, quantity and quality of the tooth structure, patient's occlusion and parafunctional habits, such as bruxism, can lead to restoration failures over time (3).

The incremental technique recommends the insertion of 2-mm thickness of resin composite in order to achieve a proper polymerized restoration, minimizing the residual shrinkage stresses without reducing the mechanical properties of the composites (4). However, this method has some disadvantages such as the possibility of empty areas between the layers, contamination risk, difficulty in the placement of layers in small cavities and long time required to perform the procedure (5). Over time, different technologies have been developed to simplify the restorative method. Bulk-fill resin composites present an attractive alternative for posterior restorations, since they can be placed into teeth cavities in a single increment of 4–5 mm depth associated to low polymerization shrinkage (6). In addition, these composites require shorter restorative time compared to conventional resin composites (7).

Several strategies are used by different manufacturers to increase the depth of cure and promote a lower polymerization shrinkage (6). Among these strategies are a greater translucency, an incorporation of more reactive photoinitiators, modulators monomers that can achieve low polymerization shrinkage (8) and changes in filler size, shape and coating that can influence the light transmittance through a composite (9). The

composition and filler loading are the most important parameters to affect the polymerization efficiency of these materials (9), so the resins composition depends on each manufacturer.

The SonicFill and Filtek BulkFill (3M ESPE, St Paul, MN, USA) resin composites have organic matrix and fillers very different each other (9). The SonicFill (Kerr Corporation, Orange, CA, USA) is a bulk-fill restorative material that has a sonic-activated system and also combines the advantages of a flowable and universal resin composite with a high inorganic filler load (83.5% of filler/weight). The low viscosity could be achieved using a diluted triethylene glycol dimethacrylate monomer (TEGDMA) that allows using the resin in an ultrasound. The application may promote effect in the polymerization by increasing free radicals' mobility directly and indirectly (10). In contrast, the Filtek BulkFill (3M ESPE, St Paul, MN, USA) is a high-viscosity resin composite and also a bulk-fill composite with 76.5% of inorganic filler/weight. This material contains in its organic matrix a 1,12-dodecane dimethacrylate (DDDMA) which has a hydrophobic backbone and increased molecular mobility. In addition, DDDMA provides flexibility, fast cure, and improved surface characteristics to the matrix (11).

The depth of cure is the main parameter of bulk-fill compounds to be evaluated *in vitro*. There are many techniques to determine the depth of cure (10) and they can be described in two groups. First, depth of cure can be measured by microhardness value, using ISO 4049 standard prescribes. Second, it can be measured directly based on the degree of conversion (DC), using (micro-)Raman or Fourier transform infrared (FTIR) spectroscopy (9). Another way to analyze the depth of cure *in vitro* is micro-computed tomography (μ CT). This has been presented in the literature (12) as a safe and non-destructible method that can analyze the behavior of the material in 3D without deteriorating or destroying the specimen (8), which can be used to quantify spaces and pores in the restorative material and the tooth/resin interface (12).

Thus, it is necessary to evaluate and understand the physical and mechanical properties of these resin composites, since is a desirable material in clinical daily practice. Therefore, the aim of the current study was designed to evaluate the DC, Knoop microhardness, polymerization shrinkage stress (PSS) and volumetric contraction using the μ CT technique of two bulk resins when compared to a conventional nanofiller resin composite. The null hypothesis was tested that no significant differences could be observed among the resin composites.

2 MATERIALS AND METHODS

Three commercial resin composites were tested: one conventional nanofiller material (Z350XT – 3M ESPE, St Paul, MN, USA) and two bulk-fill composites (SonicFill (SF) – Kerr Corporation, Orange, CA, USA and Filtek BulkFill (FB) – 3M ESPE, St Paul, MN, USA). All materials were tested with same color (A2). A polywave light unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) was used for all light-curing procedures with irradiance of 1200 mW/cm². The polymerization was performed for 40 s with the emitting end of the light source as close to the top surface of the composite as possible, against a polyester film to minimize the effects of the oxygen inhibition.

Degree of Conversion (DC)

The resin composites were placed in plastic molds with 5 mm diameter and 4 mm depth. After photopolymerization, the samples were stored dry for 24 h and covered by an aluminum foil at room temperature to guarantee that the polymerization process was complete prior to analysis. The materials were inserted as described in Table 1.

Table 1. Composition of the materials used in the study.

Resin Composite	Abbreviation used in text	Type of Material	Composition	Mode of Application
SonicFill (Kerr Corporation, Orange, CA, USA) (Kerr, USA)	SF	Sonic Activated Bulk-fill Resin Composite	Bis-GMA, Bis-EMA, TEGDMA, UDMA, silicon dioxide fillers (83.5%)	Insert up to 4 mm thick using an ultrasonic device.
Filtek BulkFill – FB (3M ESPE, St Paul, MN, USA, USA)	FB	Bulk-fill Resin Composite	Bis-GMA, AUDMA, UDMA, DDDMA, silica, zirconia filler, ytterbium fluoride	Insert up to 4 mm thick.
Filtek Z350XT (3M ESPE, St Paul, MN, USA, St. Paul, MN, USA)	Z350XT-I	Nanofiller Resin Composite	Bis-GMA, UDMA, TEGDMA, PEGDMA e Bis-EMA, HEMA, silica, zirconia filler and	Insert incrementally in 2 mm increments.
	Z350XT-B		Aggregated zirconia/silica nanoclusters	Insert up to 4 mm thick *.

Abbreviations: Bis-GMA: bisphenol-A diglycidyl ether dimethacrylate; Bis-EMA: ethoxylated bisphenol-A dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate; AUDMA: Aromatic urethane dimethacrylate; DDDMA: 1,12-dodecane dimethacrylate; PEGDMA: poly (ethylene glycol) dimethacrylate.

* Not recommended by the manufacturer.

The DC of each material was evaluated using Xplora micro-Raman spectroscopy (Horiba, Paris, France). Five specimens for each group were analyzed at a standardized

room temperature of 23 ± 1 °C and $60 \pm 1\%$ relative humidity. The %DC was measured on the bottom side of the specimens and it was calculated based on the intensity of the C=C stretching vibrations (peak height) at 1635 cm^{-1} and using the symmetric ring stretching at 1608 cm^{-1} from the polymerized and non-polymerized specimens. A Xplora micro-Raman coupled software registered the Raman spectra data in the range of $1590\text{--}1650\text{ cm}^{-1}$ using the 532 nm laser emission wavelength, with 3 s acquisition time and 3 accumulations. A small amount of uncured resin composite from each material was also obtained and its spectrum was used as unpolymerized reference.

Microhardness Test

The resin-composite specimens ($n = 4$) were made as described for DC test. Then, the specimens were removed from the molds and carefully abraded with 600 and 1200 grit abrasive paper for 20 s and cleaned between each polish procedure. The test to assess the surface hardness was performed using the microhardness testing machine (Micromet 2100 series, Buehler, Lake Bluff, IL, USA). The specimens were placed on the platform of the machine and the measurements were performed with a Knoop type diamond penetrator under a load of 50 gf for 15 s, totaling 5 random equidistant measurements on the bottom of each tested specimen, covering different surface areas. For each specimen, a mean hardness value was calculated.

Polymerization shrinkage stress (PSS) measurements

Poly (methyl methacrylate) rods, 5 mm in diameter and 13 or 28 mm in length, had one of their flat surfaces sandblasted with $250\text{ }\mu\text{m}$ alumina. On the shorter rod, to ensure the highest possible light transmission during photopolymerization, the opposite surface was polished with silicone carbide sandpaper (600, 1200, and 2000 grit) and felt disks with $1\text{ }\mu\text{m}$ alumina paste (Alumina 3, ATM, Altenkirchen, Germany). The sandblasted surfaces were covered by a layer of methyl methacrylate (JET Acrílico Auto Polimerizante, Artigos Odontológicos Clássico, São Paulo, Brazil) and two thin layers of adhesive (Scotchbond Multi- purpose Plus, bottle 3, 3M ESPE, St Paul, MN, USA, St Paul, MN, USA).

The bulk-fill composites ($n = 5$) were light-cured using a light curing unit with irradiance of 1200 W/cm^2 for 40 s. The rods were attached to the opposing clamps of a universal testing machine (Instron 5565, Canton, MA, USA) with the treated surfaces facing each other with a 1-mm gap. The composite tested was inserted into the gap and shaped into a cylinder in order to follow the perimeter of the rods. An extensometer (0.1

μm resolution) attached to the rods (Instron 2630-101, Bucks, UK) provided the feedback to the testing machine to keep the height constant.

Therefore, the force registered by the load cell was necessary to counteract the polymerization shrinkage to maintain the specimen's initial height. A hollow stainless-steel fixture with a lateral slot attached the short rod to the testing machine, allowing the tip of the light guide to be positioned in contact with the polished surface of the rod. Force development was monitored for 10 min from the beginning of the photoactivation and the nominal stress was calculated by dividing the maximum force value by the cross-section of the rod. Five specimens were tested for each resin composite.

Micro-computed Tomography (μCT)

Twenty-four standardized cylindrical cavities ($n = 8$) measuring 4 mm width and 4 mm depth were made in acrylic resin blocks. These acrylic resin blocks were selected because of its radio-translucency, so it did not interfere with the X-ray micro-computed tomography (μCT). The cavities were ultrasonically cleaned in distilled water to remove cutting debris. The resin of each group was inserted with a single increment into the cavity then a protective device was placed to prevent exposure to light. Prior to photoactivation, the assembly was taken to the μCT for initial scanning. Photopolymerization was performed and the assembly was taken to the μCT for final scanning.

The scans were performed with SkyScan 1174 device (Bruker- microCT, Kontich, Belgium) using 50 kV and 800 μA and image acquisition at every 0.7° , filed in TIFF format with a resolution of 14.1 μm and saved on a hard disk. After image reconstruction (NRecon v1.6.9; Bruker-microCT), 3D models were created. The values of the restoration volumes in the pre- and post-polymerization were calculated by means of CTAn v.1.12 software, which allowed for the determination of the volume variation for the different groups. The values were obtained in mm^3 and were later transformed into a percentage to compare pre- and post-polymerization.

Statistical analysis

Data was submitted to normality test (Kolmogorov-Smirnov) and analysis of variance with one factor (One way-ANOVA) followed by Tukey's post hoc test. Significance level was set at 5%. The program used to perform the analyses was IBM SPSS Statistics Version 20.0 (Armonk, NY, USA).

3 RESULTS

The average and the standard deviations of the DC are shown in Table 2.

Table 2. Comparison of Degree of Conversion (%DC) and Knoop microhardness (KHN) in the bottom areas (4 mm).

Resin composites	%DC	KHN
	Mean (SD)	Mean (SD)
Sonicfill	78,282 (4,6) ^a	60.094 (5.7) ^{ab}
Filtek bulkfill	75,424 (3,2) ^a	62.778 (8.2) ^a
Z350xt-i	76,228 (6,1) ^a	68.164 (6.9) ^a
Z350xt-b	60,980 (9,8) ^b	48.138 (7.7) ^b

Mean values of %DC and KHN with the same superscript capital letters in column do not show statistically significant differences ($p < 0.05$)

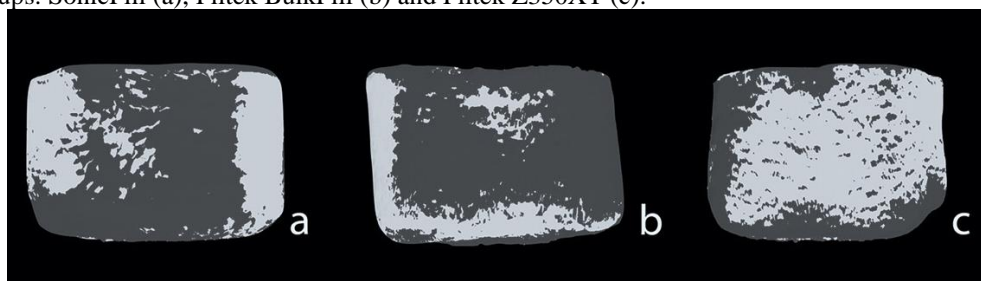
There was no significant difference between bulk-fill composites (FB and SF) and conventional nanofiller resin composite (Z350XT-I), when this one was incrementally inserted ($p > 0.05$). These values were statistically higher than those presented by Z350XT-B, applied in a 4 mm single increment ($p = 0,002$). FB and Z350XT-I showed higher microhardness values than Z350XT-B, applied in a single increment at a depth of 4 mm. SF did not shown statistical differences compared to other groups (Table 2). No statistical differences were found among the resin composites regarding to PSS ($p = 0.104$) and volumetric shrinkage ($p = 0.258$) (Table 3). Figure 1 presents illustrative images superposition of three-dimensional pre- and post-photoactivation μ CT reconstructions.

Table 3. Comparison of Polymerization Shrinkage Stress (PSS) and Volume Shrinkage (%VS) values obtained for the different resin composites.

Resin composites	PSS (mpa)	%vs
	Mean (SD)	Mean (SD)
Sonicfill	3.64 (0.54) ^a	2.14 (0.54) ^a
Filtek bulk	3.14 (0.57) ^a	1.63 (0.54) ^a
Z350xt	3.82 (0.22) ^a	2.01 (0.53) ^a

Mean values of PSS and %VS with the same superscript capital letters in column do not show statistically significant differences ($p < 0.05$).

Figure 1. Illustrative superposition of μ CT reconstructed images pre- (green) and post-photoactivated (blue) of groups: SonicFill (a), Filtek BulkFill (b) and Filtek Z350XT (c).



4 DISCUSSION

The present study evaluated the DC, Knoop microhardness, PSS and the volumetric contraction through the μ CT technique of two bulk-fill resins when compared

to a conventional nanofiller resin composite. The null hypothesis of the study was accepted, since there was no difference between bulk-fill and conventional composites, since this one is inserted following the clinical indication.

SF, FB and Z350XT-I presented acceptable values of DC at the bottom of the specimens as previously reported (8), unlike the Z350XT-B inserted in a single increment. The light, responsible for the activation of the photoinitiator, is attenuated by the absorption and dispersion of the compound, and, therefore, the depth of cure depends on the kinetics of the polymerization reaction and the ability of the material to transmit light (8). Then, it is recommended that conventional resin composites are applied incrementally with 2-mm thickness increments in order to achieve proper DC and mechanical properties, thus reducing the polymerization shrinkage stress (4). Conventional resin composites applied in a unique 4-mm increment decreases the passage of light due to the opacity of the material and the load content (8). Besides, the increase of the surface area between fillers and resin may jeopardize this issue (13). Therefore, when conventional resin composites are used, the increasing of the increment thickness results in lower DC, as shown in the present study (Table 2).

Bulk-fill resin composites were designed to remarkably reduce shrinkage stress. They present different characteristics that enable a higher DC, such as: higher translucency, higher content of photoinitiators, additional alternative photoinitiator and modulating monomers of the photopolymerization reaction (13). Furthermore, the polymerization of the resin depends on intrinsic factors, such as the chemical structure and concentration of monomers and photoinitiators (13), and extrinsic factors such as the polymerization conditions (14). For bulk-fill composites, the photopolymerization process is even more sensitive. The use of curing lights with tips size correspondent to the cavity design and a homogeneous light beam profile are recommended for a proper cure (15). The use of light-curing units delivering an irradiance ≥ 1000 mW/cm² associated to a 20 seconds of exposure time, as used in the present study, seems to be crucial for an acceptable bulk-fill composites polymerization (16). The surface microhardness of the materials tested in this study was evaluated using the Knoop hardness test, which can be considered as an accurate method to estimate the depth of cure of resin composites (17). The microhardness assessment can be considered an indirect way of assessing the DC of resinous materials (18). In the present work, FB and Z350XT-I showed better results than the Z350XT-B, while the SF showed no statistical difference in relation to the others (Table 2). Gonçalves et al. (19) showed that SF applied

in 4-mm depth presented lower light transmittance associated to a lower DC, which could be compensated by a higher amount of radiant exposure (20). On the other side, Garoushi et al. (21) showed that SF presented low values of light irradiance even with the highest DC.

In general, a linear relationship between polymerization shrinkage and the associated polymerization shrinkage stress is reported (22). Different outcomes for this parameter could be expected in clinical practice due to the variation of cavities design and layers thickness of the materials (22). In the present study, the results of PSS showed no statistical differences among the tested composites (Table 3). We highlight the fact that all composites were tested with the same thickness (1 mm), which it is mandatory according to this standardized test.

Given the advances in μ CT, the faster acquisition of high-resolution 3D images, regardless of the shape of the object and its position, it can be used for measurements of polymerization contraction with precise results (23). In the present work, no statistical differences in volumetric shrinkage were found among the groups (Table 3). Using μ CT analysis, previous studies showed lower volumetric shrinkage for bulk-fill compared to conventional resin composites in 2.5 mm deep cavities (24). In this study, cavities with 4 mm depth were used even for the conventional nanofiller resin composite which may have led to lower DC, implying in less volumetric shrinkage (25) compared to bulk-fill composites.

The present study has the limitation of not translating exactly what occurs in clinical practice. In fact, it shows the importance of using an appropriate technique for each material, not neglecting the steps necessary to obtain good clinical results. Shorter time required for the use associated with simplified operative technique are considered clinical advantages of bulk-fill composites. However, it is important that clinical trials with long-term follow-ups are performed to better understand the behavior of these composites when subjected to the oral environment. Thus, based on the employed methodology and results obtained, we failed to reject the null hypothesis previously stipulated since the resin composites were used according to their clinical indication and it can be concluded that bulk-fill and conventional resin composites tested present similar properties.

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