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AVALIAÇÃO DO GRAU DE CONVERSÃO E CONTRAÇÃO DE POLIMERIZAÇÃO DE DIFERENTES COMPÓSITOS *BULK-FILL*

FORTALEZA
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Trabalho de Conclusão de Curso (TCC) apresentado ao Curso de Odontologia da Universidade Federal do Ceará, como requisito parcial para obtenção do Título de Bacharel em Odontologia.

Orientador: Prof. Dr. Vicente de Paulo Aragão Saboia

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A Deus

A meus pais e minha irmã

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“A colheita é comum, mas o capinar é sozinho…”
Guimarães Rosa

RESUMO

As resinas compostas são materiais estéticos e de boas propriedades físicas e mecânicas. Porém, devido a sua composição química estes compósitos contraem durante a polimerização, podendo gerar *stress* e, em alguns casos, levar a falhas na interface adesiva. A inserção desses materiais deve ser feita de forma incremental, o que demanda mais tempo, maior risco de contaminação, além de falhas entre as camadas. Buscando minimizar esses problemas foram desenvolvidos os compósitos *bulk-fill, q*ue podem ser inseridos em camadas de até 4mm de espessura sem comprometer seu grau de conversão e suas propriedades mecânicas. O objetivo do estudo foi avaliar as resinas *bulk-fill* SonicFill (Kerr), Filtek BulkFill (3M ESPE) e a resina convencional Z350XT (3M ESPE) aplicada em camadas e em incremento único, avaliando o grau de conversão através de espectroscopia Micro-Raman, microdureza Knoop, tensão de contração de polimerização usando teste mecânico com extensômetro e contração volumétrica através micro-tomografia computadorizada (µCT). Não houve diferença nos valores de grau de conversão dos materiais exceto para a resina convencional fotopolimerizada a uma profundidade de 4mm que apresentou menor taxa de conversão (p= 0,002). No teste de microdureza, Filtek BulkFilll (3M ESPE) e Z350XT (3M ESPE) que foi inserida de forma incremental apresentaram melhores resultados do que a Z350XT (3M ESPE) inserida em um único incremento de 4mm, enquanto SonicFill (Kerr) não apresentou diferença estatística entre as resinas inseridas de forma *bulk* ou incremental (p =0,003). Não houve diferença estatística na tensão de contração de polimerização (p= 0,104) e na retração volumétrica, (p= 0,258) entre as resinas compostas testadas. Dessa forma, pode-se observar que as resinas compostas *bulk-fill* e convencional apresentaram propriedades mecânicas semelhantes. Entretanto, as resinas *bulk-fill* se destacam por oferecer um modo simplificado de inserção, diminuindo, assim, o tempo clínico.

Palavras-chave: Resinas compostas *bulk-fill*. Grau de conversão. Contração de polimerização. Microtomography.

ABSTRACT

Composite resins are aesthetic materials with good physical and mechanical properties. However, due to their chemical composition, these composites contract during polymerization, which can generate stress and, in some cases, lead to failures in the adhesive interface. The insertion of these materials must be done in an incremental way, which demands more time, greater risk of contamination, in addition to failures between the layers. In order to minimize these problems, bulk-fill composites were developed, which can be inserted in layers up to 4mm thick without compromising their degree of conversion and mechanical properties. The objective of the study was to evaluate the bulk-fill resins SonicFill (Kerr), Filtek BulkFill (3M ESPE) and the conventional resin Z350XT (3M ESPE) applied in layers and in a single increment, evaluating the degree of conversion through Micro-Raman spectroscopy, Knoop microhardness, polymerization shrinkage stress using mechanical test with an extensometer and volumetric contraction using computed micro-tomography (μCT). There was no difference in the values of degree of conversion of the materials, except for the conventional light-cured resin at a distance of 4mm, which presented a lower conversion rate ($p < 0.05$). In the microhardness test, Filtek BulkFilll (3M ESPE) and Z350XT (3M ESPE) showed better results than the Z350XT (3M ESPE) inserted in a single 4mm increment, while SonicFill (Kerr) did not show statistical difference between the bulk resin and the conventional resin applied incrementally ($p < 0.05$). There was no statistical difference in the polymerization shrinkage tension and volumetric shrinkage ($p < 0.05$) between the tested composite resins. Thus, it can be seen that the bulk-fill and conventional composite resins showed similar mechanical properties. However, bulk-fill resins stand out for offering a simplified way of insertion, thus reducing the clinical time.

Keywords: Bulkfill composite resins. Degree of conversion. Contraction of polymerization. Microtomography.

SUMÁRIO

1. INTRODUÇÃO

As resinas compostas são materiais estéticos que mimetizam as estruturas dentárias, além de apresentarem capacidade de adesão aos tecidos dentais duros e boas propriedades mecânicas e físicas (JACKSON, 2016). Esses materiais são constituídos por matriz orgânica, partículas inorgânicas, agente de união e sistema iniciador/catalizador da polimerização (RODRIGUES JUNIOR, 2015).

A matriz orgânica é constituída pelos monômeros que possuem a função de alterar a consistência do material, que implicará na quantidade de incorporação de carga na matriz resinosa bem como em alterações no grau de conversão (REIS; LOGUERCIO, 2007). O monômero mais comum que compõe a matriz orgânica é o BisGMA (bisfenol glicidil metacrilato), o qual tem viscosidade e peso molecular altos. Assim, monômeros de baixo peso molecular e viscosidade como o TEGDMA (dimetacrilato de trietilenoglicol) e o EGDMA (dimetacrilato de etilenoglicol) foram adicionados a fim de melhorar as propriedades de manipulação do material (FERRACANE, 2011).

As partículas inorgânicas, quartzo, a sílica coloidal e partículas de vidro tem a função de melhorar a resistência à abrasão, aumentar a resistência mecânica, reduzir o estresse e a contração de polimerização. As resinas compostas podem ser classificadas quanto a essas partículas como: macroparticuladas, microparticuladas, hibridas e nanoparticuladas. A estética e as propriedades mecânicas desses materiais estão diretamente relacionadas à sua composição (REIS; LOGUERCIO, 2007).

O sistema iniciador/catalisador é representado pela canforoquinona e uma amina alifática, tendo a função de absorver a luz e passar para uma fase excitatória (REIS; LOGUERCIO, 2007). Desta forma, após a excitação a canforoquinona transfere um elétron para a amina alifática, resultando na formação de radicais livres que irá clivar as duplas ligações, levando a uma conversão das moléculas de monômero metacrilato em polímeros, estabelecendo uma ligação covalente entre

elas, gerando assim, uma aproximação dessas moléculas (SUNBUL; SILIKAS; WATTS, 2016).

Essa aproximação das moléculas promove uma contração no material, gerando tensões na interface entre a resina composta e a estrutura dentaria. Essa tensão pode resultar em deflexão de cúspides, fendas marginais e microinfiltração nas restaurações (DEMIREL, *et.al*., 2020). Alguns fatores podem influenciar essa tensão, como o tamanho e a natureza dos monômeros, tamanho da cavidade a ser restaurada, forma de inserção da resina e modulação da intensidade da luz (CHO, *et.al.*, 2002).

O design da cavidade a ser restaurada pode ser um fator importante para o aumento do estresse de polimerização (CANEPPELE; BRESCIANI, 2016). A relação entre a área de superfície aderida e a área de superfície livre de uma restauração, chamada de fator de configuração cavitário (fator-C), influencia no escoamento do material e isso pode gerar maior tensão de polimerização nas superfícies aderidas. Assim, quanto menor o fator C, ou seja, quanto menos paredes aderidas, menor será sua tensão de contração e, consequentemente, menor a probabilidade de danos na interface adesiva (MONDELLI, 2005).

O modo de inserção do material na cavidade também pode influenciar a tensão de contração (TARDEM, *et.al.*, 2019). A técnica incremental é realizada através da inserção de porções com no máximo 2mm de espessura, os quais são fotopolimerizados individualmente (CHSANDRASEKHAR, *et.al.,* 2017). Assim, obtém-se uma restauração polimerizada adequadamente, minimizando-se as tensões de contração residuais e obtendo o máximo das propriedades mecânicas dos compósitos (BICALHO, *et.al.*, 2014). Entretanto, algumas falhas podem ocorrer com essa técnica como a possibilidade de vazios entre as camadas, risco de contaminação, dificuldade na colocação das camadas em pequenas cavidades além do aumento considerável do tempo necessário para a realização do procedimento clínico (ABBAS, *et.al*., 2003).

As resinas *bulk-fill* foram desenvolvidas como materiais restauradores que podem ser inseridos em incrementos de até 4 mm de espessura sem comprometer seu grau de conversão e propriedades mecânicas (BOARO, *et.al.*, 2019). Essas resinas apresentam vantagens em comparação com os compósitos convencionais (LINS, *et.al*., 2019), sendo elas: maior profundidade de polimerização, baixas tensões de contração de polimerização, baixa contração volumétrica (TORRES, 2017), redução do número de camadas (CAMPOS, *et.al.,* 2014), redução da deflexão cúspides (VINAGRE, *et.al.*, 2016; ROSATTO, *et.al.,* 2015) e menor tempo clínico em restauração de dentes posteriores (BELINASSO; SOARES; ROCHA, 2019).

Várias modificações foram realizadas na composição desses materiais a fim de atingir bons resultados clínicos com essa nova forma de inserção. Assim, em alguns materiais houve redução na quantidade de Bis-GMA ou substituição deste por monômeros com menor peso molecular como o UDMA (uretano dimetacrilato), gerando, assim, um monômero com menor viscosidade (RIZZANTE, *et.al.*, 2019). Foi realizada diminuição na quantidade de partículas e no aumento do tamanho destas, auxiliando na menor dispersão de luz, levando a penetração de luz em profundidade (BUCUTA; ILIE, 2014). As partículas de carga são constituídas por alumínio, zircônia, silício ou bário, sendo capazes de absorver tensões e incorporações de partículas de vidro para aumentar a resistência e diminuir a redução volumétrica (FRONZA, 2017). Além disso, houve adição de um outro tipo de fotoiniciador, como o Ivocerin (bezoil germânio), possibilitando a formação de radicais livres mais eficientes que a canforoquinona, levando a uma rápida polimerização e a uma alta taxa de conversão monomérica (MOSZNER, *et.al.,* 2008).

As resinas *bulk-fill* assim como as resinas convencionais podem ser classificadas de acordo com sua consistência, podendo ter alta ou baixa viscosidade (*flow*). As resinas *bulk* de alta viscosidade podem ser inseridas de forma única em toda extensão da cavidade (HIRATA, *et.al*., 2015), além de serem facilmente esculpíveis (CERDA-RIZO, *et.al.,* 2019). Os materiais de baixa viscosidade são mais indicados como

bases/revestimentos da cavidade, pois possuem menor dureza e menor modulo de elasticidade, principalmente, por conta da sua baixa concentração de cargas inorgânicas, sendo necessário seu recobrimento com uma resina de maior resistência ao desgaste (ILIE; STARK, 2014).

CERDA-RIZO et. al. (2019) observaram que as resinas *bulk-fill* de alta viscosidade apresentaram melhores resultados de dureza tanto em sua superfície quanto em profundidade, polimerização adequada e menor tensão de polimerização quando comparadas com resinas *bulk-fill* de baixa viscosidade. Colak et. al. (2017) realizaram um ensaio clínico randomizado com um acompanhamento de 12 meses utilizando resinas *bulk-fill* de alta viscosidade em restaurações classe II e mostraram bom comportamento clínico.

As resinas SonicFill (Keer) e a Filtek BulkFill (3M ESPE) apresentam diferenças em sua composição química. A resina SonicFill (Kerr) é um compósito nano-híbrido que apresenta um alto conteúdo de carga inorgânica de 83,5% em peso, a qual é ativada através de energia sônica para diminuir a viscosidade e facilitar sua adaptação às paredes cavitárias. A sua matriz orgânica é composta por BisGMA, assim como os diferentes diluentes como TEGDMA (trietileno glicol dimetacrilato) e EBPDMA (RODRIGUES JUNIOR, 2015). Akalin et. al. (2016) mostraram que a SonicFill (Kerr) apresentou o maior grau de microdureza e isso se deve a sua grande quantidade de partículas inorgânicas. Um estudo clínico randomizado com acompanhamento de 24 meses em cavidades classe II restauradas com resina composta SonicFill (Kerr) mostrou que o comportamento clínico deste material não foi afetado pelo tipo de cavidade (MO, MOD e OD) e a estabilidade do material foi considerada aceitável. Entretanto, houve descoloração marginal, que foi atribuída a técnica adesiva (AKALIN, *et.al.,* 2018)

A Filtek BulkFill (3M ESPE) é um material micro-híbrido com alta viscosidade e com conteúdo de carga inorgânica de 76,5% em peso. Sua matriz contém AFM (monômero para alívio dinâmico de tensões de contração de polimerização), além de possuir monômeros exclusivos como um aromático de alto peso molecular como o dimetacrilato AUDMA, UDMA e 1, 12-dodecano-DMA (SHIBASAKI, *et.al.,* 2017).

Balkaia e col. (2019) realizaram um estudo clínico randomizado e mostraram que após 12 meses, os compósitos Filtek BulkFill (3M ESPE) mantiveram sua forma anatômica, pontos de contato e adaptação marginal, só possuindo diferença em sua coloração e translucidez, mas dentro de um padrão aceitável.

Pela importância clínica do grau de conversão diversos estudos *in vitro* têm sido realizados para avaliar a profundidade de polimerização dos compósitos *bulk-fill* (GORACCI, *et.al.*, 2014; PAR, *et.al.,* 2015). As técnicas para determinar esse parâmetro podem ser descritas em dois grupos: teste de microdureza, como técnica indireta ou o grau de conversão (DC), usando espectroscopia (micro-) Raman ou infravermelho com transformada de Fourier (FTIR), como técnica direta (VAN ENDE, *et.al.,* 2017).

As técnicas de microtomografia (μCT) têm permitido a análise do comportamento do material e a visualização de suas modificações tridimensionais no interior de uma determinada configuração cavitária (HIRATA, *et.al*., 2015). Esse método, que tem a vantagem de ser não destrutivo (FRONZA, *et.al.*, 2015), pode ser usado para quantificar espaços e poros no interior do material restaurador bem como fendas na interface dente/resina (MELEO, *et.al*., 2012).

Como se tratam de materiais ainda relativamente recentes existe a necessidade de mais estudos para comprovar suas propriedades e indicações. Dessa forma, o presente estudo tem como objetivo avaliar a contração volumétrica através da técnica de μCT, a tensão de contração de polimerização e o grau de conversão de resinas *bulk- fill* quando comparadas a uma resina convencional. A hipótese nula do presente estudo é que não há diferença entre as resinas convencionais e resinas compostas *bulk-fill* nos parâmetros avaliados.

2. PROPOSIÇÃO

2.1. Objetivo geral: Avaliar propriedades químico-mecânicas de duas resinas composta *bulk-fill* comparando-as com uma resina composta convencional.

2.2. Objetivo específico:

- Avaliar *in vitro* o grau de conversão das resinas *bulk-fill* Sonic Fill (Kerr), Filtek BulkFill (3M ESPE) e a resina convencional Z350XT (3M ESPE) aplicada em camadas e em incremento único através de espectroscopia Micro-Raman;
- Avaliar a microdureza Knoop das resinas *bulk-fill* Sonic Fill (Kerr), Filtek BulkFill (3M ESPE) e a resina convencional Z350XT (3M ESPE) aplicada em camadas e em incremento único.
- Avaliar a tensão de contração de polimerização, usando teste mecânico com extensômetro das resinas *bulk-fill* Sonic Fill (Kerr), Filtek BulkFill (3M ESPE) e a resina convencional Z350XT (3M ESPE);
- Avaliar a contração volumétrica das resinas *bulk-fill* Sonic Fill (Kerr), Filtek BulkFill (3M ESPE) e a resina convencional Z350XT (3M ESPE) por meio de micro-tomografia computadorizada (µCT).

3. CAPÍTULO

Esta monografia está baseada no Artigo 7, item II das Normas que regularizam o Trabalho de Conclusão do Curso de Odontologia da Faculdade de Farmácia, Odontologia e Enfermagem da Universidade Federal do Ceará. Desta forma, baseado no Regimento Interno do Programa de Pós-Graduação em Odontologia da Universidade Federal do Ceará que regulamenta o formato alternativo para dissertações de Mestrado e teses de Doutorado, e permite a inserção de artigos científicos de autoria ou coautoria do candidato, esse trabalho é composto por um artigo científico que será submetido ao periódico *Odontology*, conforme descrito abaixo:

EVALUATION OF DEGREE OF CONVERSION AND POLYMERIZATION SHRINKAGE OF DIFFERENT BULK-FILL COMPOSITES

RODRIGUES, NS; CUNHA, DA; TOMAZ, BL; RODRIGUES, E; SOUZA, LC; LUNA-CRUZ, SM; MENDONÇA, J; SABOIA, VPA.

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EVALUATION OF DEGREE OF CONVERSION AND POLYMERIZATION SHRINKAGE OF DIFFERENT BULK-FILL COMPOSITES

ABSTRACT

The aim of the study was to assess degree of conversion (DC) the microhardness, the polymerization shrinkage stress (PSS) and the volumetric contraction of two bulk-fill composites (Sonic Fill – KERR (SF) and Filtek BulkFill – 3M ESPE (FB)) and one conventional resin (Z350XT - 3M ESPE) inserted incrementally (Z350I) or in bulk (Z350B). Micro-Raman spectroscopy, Knoop microhardness, PSS test and Micro-computed tomography technique (μCT) 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 was inserted in a single increment ($p = 0.002$). FB and Z350I showed higher microhardness values than Z350B ($p = 0,003$), while SF did not present statistical difference. No statistical differences were found among the composites regarding to PSS ($p = 0,104$) and volumetric shrinkage ($p = 0,258$). Therefore, we can conclude that bulk-fill and conventional composites 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; Microtomography

INTRODUCTION

The direct light-cured resin composites are considered good material choice for esthetic restorations in posterior teeth [1]. However, clinical failures could be related to the limitations of the mechanical properties of the composite resins [2, 3], such as the inherent volumetric shrinkage caused by the polymerization [4] and the development of polymerization stress [5]. The stress may develop marginal gaps at the tooth/restoration interface, which can result in adhesive defects, such as postoperative sensitivity and restoration fracture [6]. The longevity of restorations could be also associate with operative technique, cavity configuration, quantity and quality of the tooth structure, and a patient's occlusion, parafunctional habits such as bruxism failures in restoration over time [7].

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 [8] . 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 [9]. Over time, different technologies have been developed to simplify the restorative method. In this scenario, the 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 [10, 11] associated to low polymerization shrinkage [12]. In addition, these composites require shorter restorative time compared to conventional resins [13].

Several strategies are used by different manufacturers to increase the depth of cure and promote a lower polymerization shrinkage [11]. Among these strategies are a greater translucency, an incorporation of more reactive photoinitiators, modulators monomers that can achieve low polymerization shrinkage [3] and changes in filler size, shape and coating that can influence the light transmittance through a composite [14]. The

composition and filler loading are the most important parameters to affect the polymerization efficiency of these materials [15], because of that, the resins composition depends on each manufacturer.

The Sonic Fill (Kerr) and Filtek BulkFill (3M ESPE) resin composites have organic matrix and fillers very different each other [14]. The Sonic Fill (Kerr) is a single-step 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) [16]. The low viscosity could be achieve using a diluted triethylene glycol dimethacrylate monomer that allows using the resin in an ultrasound [17]. The application may promote effect in the polymerization by increasing free radicals' mobility directly and indirectly [18]. In contrast, the Filtek BulkFill (3M ESPE) is a high-viscosity resin composite and also a single-step restorative material with 76.5% of inorganic filler/weight. This material contains in its matrix organic 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 [17].

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 [18-20] and they can be described in two groups. First, depth of cure can be measured by micro-hardness 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 [14]. Another way to analyze the depth of cure *in vitro* is computed microtomography (µCT). This has been presented in the literature [21, 22] as a safe and non-destructible method that can analyze the behavior of the material in 3D without deteriorating or destroying the specimen [3], which can be used to quantify spaces and pores in the restorative material and the tooth / resin interface [23].

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 therefore designed to evaluate the volumetric contraction using the μCT technique, the polymerization shrinkage stress (PSS), the DC and microhardness of bulk resins when compared to a conventional resin. The null hypothesis was tested that no differences could be observed between the composites resins.

MATERIALS AND METHODS

Three commercial resin-based composites were tested: one conventional material (Z350XT – 3M ESPE) and two bulk fill composites (Sonic Fill (SF) – Kerr and Filltek BulkFill (FB) – 3M ESPE). 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 800 mW/cm^2. 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 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. 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 ^{o}C and 60 ± 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 – 1650 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. The test to assess the surface hardness was performed using the Knoop 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 seconds, 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 μm 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 1μm alumina paste (Alumina 3, ATM, Altenkirchen, Germany). The sandblasted surfaces were covered by a layer of methyl methacrylate (JET Acrilico Auto Polimerizante, Artigos Odontologicos Classico, Sao Paulo, Brazil) and two thin layers of adhesive (Scotchbond Multi- purpose Plus, bottle 3, 3M ESPE).

The bulk-fill composites (n = 5) were light-cured using a light cuing 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 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 computed mcro-tomography (μ-CT). 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 μ-CT for initial scanning. Photopolymerization was performed and the assembly was taken to the μ-CT 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 CTan v.1.12 software

was used to evaluate the variation in volume of the restorations. 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 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).

RESULTS

The average and the standard deviations of the DC are shown in Table 2. There was no significant difference between bulk-fill composites (FB and SF) and conventional resin (Z350XT), when this one was inserted incrementally ($p = 0.002$). These values were statistically higher than those presented by Z350XT when applied in a 4 mm single increment.

FB and Z350XT (inserted incrementally) showed higher microhardness values than Z350XT applied in a single increment at a depth of 4 mm. SF did not present statistical difference compared to other groups as shown in Table 3. No statistical differences were found among the composites regarding to PSS ($p = 0,104$) and volumetric shrinkage ($p = 0,258$). Figure 1 presents illustrative images superposition of three-dimensional pre and post-photoactivation µCT reconstructions.

DISCUSSION

The present study evaluated the volumetric shrinkage through the µCT technique, the PSS and the DC of bulk-fill resins when compared to a conventional resin. The hypothesis of the study was accepted, since

there was no difference between bulk-fill and conventional composites, since this one is inserted incrementally.

SF, FB and Z350XT (applied incrementally) presented acceptable values of DC at the bottom of the specimens as previously reported [3, 24], unlike the Z350XT 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 [3, 25]. 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 [8]. Conventional composites applied in a unique 4 mm increment decreases the passage of light due to the opacity of the material and the load content [26]. Besides, the increase of the surface area between fillers and resin may jeopardize this issue [27]. Therefore, when conventional composites are used, the increasing of the increment thickness results in lower DC, as shown in the present study (Table 2).

Bulk-fill 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 [27, 28]. Furthermore, the polymerization of the resin depends on intrinsic factors, such as the chemical structure and concentration of monomers and photoinitiators [29], and extrinsic factors such as the polymerization conditions [30, 31]. 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 [32]. The use of light-polymerization units delivering an irradiance ≥1000 mW/cm^2 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 [33].

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 composite resins [10]. The microhardness assessment can be considered an indirect way of assessing the DC of resinous materials [34, 35]. In the present work, FB and Z350I showed better results than the Z350B, while the SF showed no statistical difference in relation to the others (Table 3). Gonçalves et al. [36] 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 [37]. On the other side, Garoushi et al. [38] 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 [39]. In the present study, the results of PSS showed no statistical differences among the tested composites (Table 4). We highlight the fact that all composites were tested with the same thickness (1 mm), which it is mandatory according to this standardized test. Different outcomes for this parameter could be expected in clinical practice due to the variation of cavities design and layers thickness of the materials.

Given the advances in μCT, the faster acquisition of high-resolution 3D images, regardless of the shape of the object and its position, can be used for measurements of polymerization contraction with precise results [2]. In the present work, no statistical differences in volumetric shrinkage were found among the groups (Table 5). Using μCT analysis, previous studies showed lower volumetric shrinkage for bulk-fill compared to conventional composites in 2.5 mm deep cavities [40, 41]. In our study, cavities with 4 mm depth were used even for the conventional composite which may have led to lower DC, implying in less volumetric shrinkage [42] compared to bulk-fill composites.

The present study has the limitation of not translating exactly what occurs in clinical practice. Therefore, it is important that clinical trials with

long-term follow-up are performed to better understand the behavior of these composites when subjected to the oral environment. We can conclude that bulk-fill and conventional composites present similar properties. Thus, shorter time required for the use associated with simplified operative technique are considered clinical advantages of bulk-fill composites.

Table 1. Composition of the Materials Used in the Study

Resin Composite	Abbreviation used in text	Type of Material	Composition	Mode of Application
Sonic Fill (KERR)	SF	Sonic Activated Bulk-fill Resin Composite	Bis-GMA, TEGDMA, Bis-EMA, UDMA, silicon dioxide fillers (83.5%)	Insert up to 4 mm thick using an ultrasonic device.
Filtek BulkFill – FB (3M ESPE, 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, USA)	Z350 I	Nanofiller Resin Composite	Bis-GMA, UDMA, TEGDMA, PEGDMA e Bis-EMA, HEMA, silica, zirconia filler and Aggregated zirconia/silica nanoclusters	Insert incrementally in 2 mm increments.
	Z350 B			Insert up to 4 mm thick.

Abbreviations: Bis-GMA: bisphenol-A diglycidyl ether dimethacrylate; Bis-EMA: ethoxylated bisphenol-A dimethacrylate; TEGDMA: triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate.

* Not recommended by the manufacturer.

Table 2. Comparison of Degree of Conversion (%) in the bottom areas (4 mm)

RESIN COMPOSITES	MEAN (SD)
SonicFill	78,282 (±4,6) [A]
Filtek Bulkfill	75,424 (±3,2) [A]
Z350 Incremental	76,228 (±6,1) [A]
Z350 Bulk	60,980 (±9,8) [B]

Resin composites that did not present statistically significant differences are listed above with the same letter (p<0,05).

Table 3. Comparison of Microhardness in the bottom areas (4mm)

RESIN COMPOSITES	MEAN (SD)
SonicFill	60,094 (±5,7) [AB]
Filtek Bulk	62,778 (±8,2) [A]
Z350 Incremental	68,164 (±6,9) [A]
Z350 Bulk	48,138 (±7,7) [B]

Resin composites that did not present statistically significant differences are listed above with the same letter (p<0,05).

Table 4. Comparison of Polymerization Shrinkage Stress (PSS) values obtained for the different composites assessed.

MATERIAIS	MÉDIAS (DEVIO PADRÃO)

SonicFill	3,64 (±0,54)[A]
Filtek Bulk	3,14 (±0,57)[A]
Z350 XT	3,82 (±0,22)[A]

Resin composites that did not present statistically significant differences are listed above with the same letter (p<0,05).

Table 5. Comparison of Volume Shrinkage values obtained for the different composites assessed by Microtomography.

MATERIAIS	MÉDIAS (DESVIO PADRÃO)
SonicFill	2,14 (±0,54)[A]
Filtek BulkFill	1,63 (±0,54)[A]
Z350 XT	2,01 (±0,53)[A]

Resin composites that did not present statistically significant differences are listed above with the same letter (p<0,05).

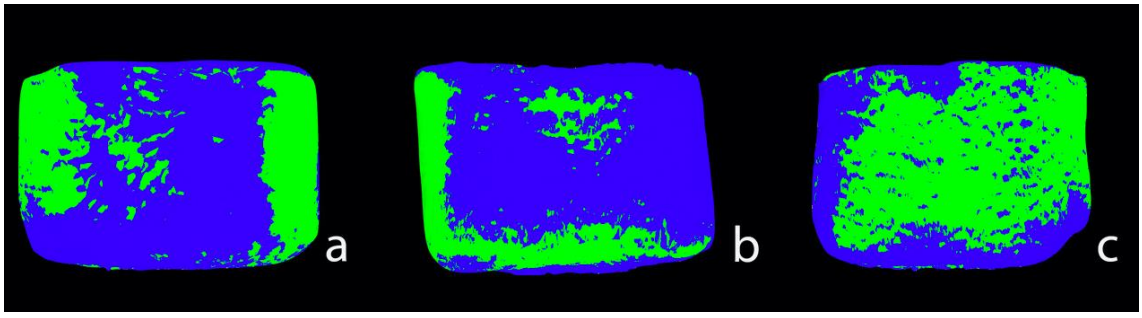

Figure 1. Illustrative superposition of µCT reconstructed images pre (green) and post-photoactivated (blue) of groups: Sonic Fill (a), Filtek Bulk (b) and Z350XT (c).

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4. CONCLUSÃO GERAL

O presente estudo tem a limitação de não traduzir exatamente o que ocorre na prática clínica. Portanto, é importante que ensaios clínicos com acompanhamento em longo prazo sejam realizados para melhor compreender o comportamento desses compósitos quando submetidos ao meio bucal.

Pode-se concluir que os compósitos *bulk-fill* e convencionais avaliados nesse estudo apresentam propriedades físicas e mecânicas similares. Porém, os compósitos *bulk-fill* tem a vantagem de exigirem técnica operatória mais simplificada o que resulta em menor tempo clínico.

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