



DOUTORADO EM ODONTOLOGIA  
ÁREA DE CONCENTRAÇÃO EM DENTÍSTICA

**ROBERTO CÉSAR DO AMARAL**

**AVALIAÇÃO DAS PROPRIEDADES MECÂNICAS E DA  
RESISTÊNCIA DE UNIÃO À DENTINA DE RESINAS  
COMPOSTAS BULK-FILL COM DIFERENTES  
VISCOSIDADES**

Guarulhos  
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RESISTÊNCIA DE UNIÃO À DENTINA DE RESINAS  
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VISCOSIDADES**

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Orientador: Prof. Dr. André F. Reis

Co-orientador: Prof. Dr. José Augusto Rodrigues

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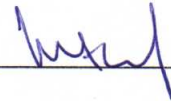
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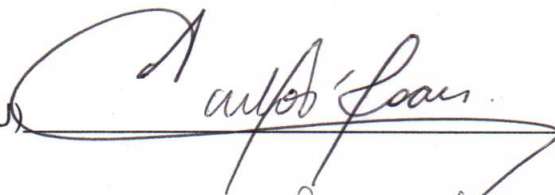
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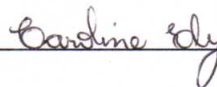
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“Os que se encantam com a prática sem a ciência são como os timoneiros que entram no navio sem timão nem bússola, nunca tendo a certeza do seu destino”

(Leonardo da Vinci)

## RESUMO

Os objetivos no presente estudo foram: (1) avaliar a resistência de união (RU) à dentina de resinas composta *bulk-fill* de diferentes viscosidades, na parede gengival de cavidades Classe II MOD após a ciclagem térmica e mecânica; (2) avaliar a resistência coesiva (RC) e à flexão (RF) em diferentes espessuras; (3) verificar o grau de conversão (GC) de uma resina composta *bulk-fill* de alta viscosidade. Três resinas compostas foram avaliadas: Tetric EvoCeram (TEC), convencional como controle, Tetric EvoCeram Bulk Fill (TECB), compósito de alta viscosidade e Tetric EvoFlow Bulk Fill (TEFB), compósito de baixa viscosidade. Para a resistência de união o teste de microtração foi realizado. As resinas de cada grupo foram inseridas em cavidades MOD com 4 e 6 mm de profundidades nas caixas proximais. A (TEC) foi inserida com a técnica incremental e (TECB) e (TEFB) em incremento único. Os dentes restaurados foram termo ciclados a 350,000 ciclos. Após, os dentes foram seccionados para se obter palitos da parede gengival das caixas proximais. Estes foram testados sob tração a uma velocidade de 1 mm/min. Para o teste de (RC) e (RF) foram confeccionadas 10 amostras em forma de ampulheta e 10 em forma de barras em uma matriz com 6 mm de espessura. As resinas de cada grupo foram inseridas e fotoativadas por 20s. Após removidas do molde, as amostras foram seccionadas em 2 partes de 2 mm cada para obtenção da porção superficial (2 mm), meio (4 mm) e base (6 mm). As amostras em forma de ampulhetas foram testadas sob tração para se obter a RC e as barras foram testadas pelo teste de RF. Adicionalmente o GC da resina TECB foi obtido nas profundidades de 2, 4 e 6 mm com FTIR. Não foram observadas diferenças na RU entre 4 e 6 mm de profundidade para todas as resinas testadas. As resinas (TECB) e (TEFB) apresentaram maiores valores de RU que (TEC). Entre (TECB) e (TEFB) não houve diferença significativa. Os maiores valores de RC e RF obtidos foram para TEC em 2 mm diminuindo significativamente em 4 mm e em 6 mm. A RC e RF das resinas TECB e TEFB foram similares em 2 e 4 mm e significativamente inferior em 6 mm. O GC da TECB foi maior para a espessura de 2 mm e significativamente maior em comparação a 4 e 6 mm. Pelos resultados obtidos, as resinas compostas *bulk-fill* demonstraram ser uma excelente opção para amplas restaurações diretas em dentes posteriores diante do comportamento apresentado frente aos ensaios mecânicos empregados em nosso estudo.

**Palavras-Chave:** microtração, resina de incremento único, tensão de polimerização, resistência flexural, grau de conversão.

## ABSTRACT

The aims of this study were to: (1) evaluate microtensile bond strength ( $\mu$ TBS) of flowable and nonflowable bulk-fill composites to dentin from gingival walls of Class II MOD cavities after thermo-mechanical load cycling; (2) evaluate the ultimate tensile strength (UTS) and flexural strength (FS) of two bulk-fill composites as function of increment thickness; (3) determine the degree of conversion (DC) of a high viscosity bulk-fill composite. Three composite resins were used: a conventional composite (TEC - Tetric EvoCeram), a high-viscosity bulk-fill composite (TECB - Tetric EvoCeram Bulk Fill) and a low-viscosity bulk-fill composite (TEFB -Tetric EvoCeram Bulk Fill Flow). Composites were applied in MOD cavities with proximal boxes of 4 and 6 mm depth. Conventional composite was placed incrementally and bulk-fill composites were placed in bulk. Restored teeth were subjected to 350.000 thermo-mechanical cycles. Afterwards, teeth were sectioned into beams with a cross-sectional bonded area of approximately 1 mm<sup>2</sup>. Bonded beams obtained from the gingival walls of the proximal boxes were tested in tension at a crosshead speed of 1 mm/min. For UTS and FS tests, 6-mm thick specimens were prepared for each composite. Molds were bulk-filled with composite and light-cured for 20 s. Samples were sectioned horizontally to obtain 3 hourglass-shaped specimens for UTS and 3 rectangular bars-shaped specimens for FS (2, 4 and 6 mm). For UTS, specimens were tested in tension. For FS, the three-point bending test was performed until failure occurred. Additionally, DC was obtained for TECB at 2, 4 and 6 mm. No significant differences in  $\mu$ TBS values were observed between 4 and 6 mm-depth proximal boxes for all composites tested ( $p>0.05$ ). However, bulk-fill composites presented significantly higher  $\mu$ TBS values than the incrementally placed conventional composite ( $p<0.05$ ).  $\mu$ TBS values for the flowable and nonflowable bulk-fill composites were not significantly different. The DC for TECB presented the highest values at 2 mm, which were significantly higher than 4 mm ( $p<0.05$ ), and decreased significantly for 6 mm specimens ( $p<0.05$ ). Both low- and high-viscosity composites presented higher flexural strength than the conventional composite when cured in 4-mm thick bulk increments. However, FS of the low-viscosity bulk-fill composite was significantly lower than the conventional composite at 2 mm. Bond strength to the gingival walls of Class II cavities did not differ between 4 and 6 mm-deep proximal boxes, however bulk-fill composites performed significantly better than the incrementally placed conventional composite. According to the results of the present investigation, both bulk-fill composites proved to be a reliable alternative for restoration of posterior teeth.

**Keywords:** microtensile bond strength, bulk-fill composites, polymerization stress, flexural strength, degree of conversion.

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## 1. INTRODUÇÃO

A indicação do uso de resinas compostas diretas em dentes posteriores tem sido impulsionada nas últimas décadas em virtude da melhoria da composição química destes materiais, contribuindo para o melhor desempenho em relação às propriedades físicas e mecânicas, resultando em restaurações com maior durabilidade clínica (Demarco et al., 2012).

Ao longo dos últimos anos estudos clínicos demonstram um bom desempenho de restaurações diretas de resinas compostas em dentes posteriores, com 10 a 22 anos de longevidade e baixas taxas de falhas anuais (Da Rosa Rodolpho et al., 2006; Van Dijken, 2010; Da Rosa Rodolpho et al., 2011; Demarco et al., 2012). Contudo o uso de compósitos convencionais em dentes posteriores ainda ocasiona alguns efeitos indesejáveis, tais como: sensibilidade pós-operatória, manchamento nas margens da restauração, trincas na estrutura dental e cáries secundárias (Kleverlaan; Feilzer, 2005; Hofmann; Hunecke, 2006; Park et al., 2008; Burgess; Cakir, 2010; Heintze; Rouson, 2012) em decorrência da contração volumétrica inerente a este tipo de material durante o processo de polimerização.

As resinas compostas tradicionais são materiais de uso rotineiro nos procedimentos restauradores diretos (Ilie; Hickel, 2011), embora estejam sujeitos à sensibilidade da técnica. Necessitam ser inseridas de maneira incremental em espessuras de no máximo 2,0 mm e fotopolimerizadas entre cada camada. Isto para que ocorra suficiente penetração da luz para o processo de conversão dos monômeros em polímeros além de contribuir na redução da tensão resultante da contração volumétrica, típica do mecanismo de polimerização (Van Ende et al., 2013; Lazarchik et al., 2007).

Sendo a contração de polimerização o principal efeito deletério originado no mecanismo de conversão, destaca-se o fato de que as tensões geradas nas paredes da cavidades acarretam uma deformação na estrutura

dental, representado por uma movimentação das cúspides, gerando falhas na interface dente e restauração (Moorthy et al., 2012). O comprometimento da integridade da interface proporciona a formação de micro-fendas tornando a restauração mais suscetível ao desenvolvimento de micro-infiltrações e lesões de cáries secundárias (Park et al., 2008; Hofmann; Hunecke, 2006; Kleverlaan; Feilzer, 2005) principalmente em amplas cavidades de Classe II com ausência de esmalte nas margens (Poggio et al., 2013; Pecie et al., 2013).

A inserção da resina composta de maneira incremental e em camadas oblíquas permite uma melhor distribuição das tensões geradas nas paredes da cavidade em comparação a inserção em camadas horizontais (Soares et al., 2013) e torna-se a alternativa clínica mais aceita para restaurações com compósitos convencionais.

Mesmo sendo uma das maneiras de minimizar as tensões geradas no processo de polimerização, a técnica incremental oblíqua apresenta algumas desvantagens, tais quais, a possibilidade da incorporação de bolhas, falhas na união e/ou contaminação entre incrementos, dificuldade de inserção em cavidades extremamente conservadoras e o aumento do tempo de trabalho necessário para realizar o procedimento restaurador em função da fotopolimerização de cada camada (Abbas et al., 2003; Sarrett, 2005; El-Safty et al., 2012). Procedimentos restauradores diretos com menos sensibilidade da técnica e que possam ser executados mais rapidamente constituem uma necessidade apresentada pelos clínicos sem que a restauração apresente os efeitos indesejáveis proporcionados pela contração de polimerização (Jang et al., 2015).

Nos últimos anos os fabricantes vêm introduzindo no mercado diferentes técnicas e materiais para melhorar o desempenho clínico dos compósitos e aprimorar a qualidade da interface adesiva formada com a estrutura dentária. Estas modificações resultam em menor contração volumétrica do material após a polimerização e incluem a utilização de novas resinas com monômeros alternativos ao metacrilato, como o Silorano (Hofmann; Hunecke, 2006; Braga et al., 2005), inovações nas partículas de

carga com tamanho nanométrico, modificações nos monômeros tradicionais da matriz e o uso de novas moléculas fotoiniciadores (Moszner et al., 2008; Cramer et al., 2011; Fujita et al., 2011).

Recentemente um dos principais focos de estudo são as resinas compostas *bulk-fill*. Estes materiais apresentam uma modificação na composição química permitindo baixa contração de polimerização, além da inserção e fotoativação em incremento único de 4,0 – 5,0 mm (Czasch; Ilie, 2013; Tiba et al., 2013; Ilie et al., 2013). Suas grandes vantagens são a facilidade técnica na aplicação e a diminuição do tempo clínico para confeccionar à restauração (Park et al., 2008; Burgess; Cakir, 2010; Ilie et al., 2013).

A primeira resina composta *bulk-fill* introduzida no mercado nacional foi a Surefil SDR *flow* (Dentsply). Trata-se de um compósito de baixa viscosidade com alta capacidade de escoamento e uma menor contração de polimerização em comparação as resinas *flow* tradicionais e os compósitos à base de Silorano (Burgess; Cakir, 2010; Ilie; Hickel, 2011; Moorthy et al., 2012).

Atualmente encontram-se disponíveis no mercado resinas *bulk-fill* de diferentes marcas comerciais e com diferentes viscosidades. As resinas de baixa viscosidade são chamadas de “*flowable*” e escoam com mais facilidade nas paredes das cavidades. Estas possuem menos partículas de carga e normalmente necessitam de uma resina convencional de cobertura para finalizar a restauração.

Já as resinas *bulk-fill* regular não necessitam de uma resina de cobertura e são esculpíveis. Estas características reológicas influenciam as propriedades mecânicas destes materiais e sua avaliação permite uma comparação com as resinas compostas convencionais para posterior aplicabilidade clínica. Um recente estudo avaliando diferentes propriedades mecânicas demonstrou que as resinas *bulk-fill*, independente da viscosidade, demonstraram desempenho superior a resina convencional testada. Já na comparação direta entre os tipos de *bulk-fill*, diante da viscosidade, as



resinas “*flowable*” possuem propriedades mecânicas inferiores às resinas *bulk-fill* de alta viscosidade (Rosatto et al., 2015).

O comportamento físico mecânico das resinas compostas é otimizado quando o módulo de elasticidade do material apresenta-se semelhante aos tecidos dentais para que não ocorra uma transferência de tensão exacerbada durante a mastigação (Leprince et al., 2013). Uma eficaz polimerização dos compósitos significa uma alta conversão monomérica e isto depende de alguns fatores, tais como: fonte de luz utilizada, molécula fotoiniciadora do material, propriedades ópticas, capacidade de transmissão da luz em profundidade e a viscosidade do material (Ferracane, 2013; Leprince et al., 2014; Price et al., 2015).

Em restaurações amplas e profundas, tais quais cavidades classe II MOD restauradas com resinas *bulk-fill*, ocorre a necessidade de uma grande quantidade de material. Desta forma, a luz necessita de intensidade suficiente para atingir todo o volume do material para que uma polimerização eficiente ocorra. Nestas situações, as propriedades mecânicas do material podem estar comprometidas e conseqüentemente pode ocorrer a redução da longevidade das restaurações (Price; Felix, 2009; Rueggeberg, 2011).

A caracterização mecânica das resinas compostas *bulk-fill* é essencial para o entendimento do seu comportamento durante o ato mastigatório. As avaliações laboratoriais das propriedades e dos fatores específicos envolvidos no desempenho do material, ajudam a prever resultados clínicos com estes compósitos com alta e baixa viscosidade (Fronza et al., 2015; Rosatto et al., 2015).

Para que uma condição mais próxima do ambiente bucal possa ser simulada em laboratório, os métodos mais válidos para correlacionar os resultados encontrados em testes *in vitro* com dados *in vivo* são: avaliar a durabilidade da interface adesiva entre compósitos e estrutura dental pelo envelhecimento artificial, com o armazenamento em água em diferentes períodos, ou submetendo as restaurações a ciclagem térmica e carga oclusal (De Munck et al., 2005). A ciclagem térmica e mecânica permite uma

condição muito similar ao comportamento de uma restauração em boca ao longo do tempo.

Em situações clínicas, é muito comum a restauração com resinas compostas de amplas cavidades de Classe II, muitas vezes sendo indicativo de substituição de uma antiga restauração em amálgama. Entretanto o local que torna a restauração de Classe II mais susceptível à falhas é sem dúvida à margem gengival da caixa proximal, principalmente em cavidades com ausência de esmalte nesta margem.

Portanto nos resta elucidar se as resinas compostas *bulk-fill* com diferentes viscosidades possuem o mesmo comportamento que as resinas compostas convencionais inseridas de maneira incremental diante de testes mecânicos em cavidades de Classe II.

## 2. PROPOSIÇÃO

O principal objetivo deste trabalho foi estudar o comportamento *in vitro* de resinas compostas *bulk-fill* em diferentes viscosidades. Assim, os objetivos específicos foram:

- Avaliar após a ciclagem térmica e mecânica, a resistência de união à dentina de resinas compostas *bulk-fill* na parede gengival de cavidades padronizadas de Classe II.
- Avaliar o comportamento mecânico das resinas compostas *bulk-fill* com diferentes viscosidades e em diferentes profundidades através da resistência coesiva, resistência flexural e o grau de conversão da resina de alta viscosidade.

### **3. DESENVOLVIMENTO**

#### **3.1. Capítulo 1**

##### **Bond strength of flowable and nonflowable bulk-fill resin composites in class II MOD cavities after thermo-mechanical challenge**

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**Abstract**

This study evaluated the microtensile bond strength ( $\mu$ TBS) of flowable and nonflowable bulk-fill composites to dentin from gingival walls of Class II MOD cavities after thermo-mechanical load cycling. Preparations were made in 48 human molars with the distal and mesial proximal boxes measuring 4 and 6 mm deep, respectively. Six experimental groups (n=16) were obtained in a factorial design including "composite" in three levels and "depth" in two levels. A conventional composite (Tetric EvoCeram, placed incrementally), a high-viscosity (nonflowable) bulk-fill resin composite (Tetric EvoCeram Bulk Fill Regular) and a low-viscosity (flowable) bulk-fill composite (Tetric EvoFlow Bulk Fill) were applied in MOD cavities with proximal boxes of 4 and 6 mm depth with gingival margins placement in dentin. Restored teeth were subjected to 350.000 thermo-mechanical cycles. Afterwards, teeth were sectioned into beams with a cross-sectional bonded area of approximately 1 mm<sup>2</sup>. Bonded beams obtained from the gingival walls of the proximal boxes were tested in tension at a crosshead speed of 1 mm/min. Data were submitted to 2-way ANOVA and Tukey test. No significant differences in  $\mu$ TBS values were observed between 4 and 6 mm-depth proximal boxes for all composites tested ( $p>0.05$ ). However, bulk-fill composites presented significantly higher  $\mu$ TBS values than the incrementally placed conventional composite ( $p<0.05$ ).  $\mu$ TBS values for the flowable and nonflowable bulk-fill composites were not significantly different. Bond strength to the gingival walls of Class II cavities did not differ between 4 and 6 mm-deep proximal boxes, however bulk-fill composites performed significantly better than the incrementally placed conventional composite.

**Keywords:** microtensile bond strength, bulk-fill composites; polymerization stress.

## INTRODUCTION

The clinical success of composite resin restorations in posterior teeth depends on the quality of the bond between composite and tooth structure and an excellent adaptation of material to the cavity walls (Peumans et al., 2005). Methacrylate-based composites are routinely used in direct restorative procedures. Technically composites are sensitive materials because they need to be inserted in increments of up to 2 mm (Sakaguchi et al., 1999; Pilo et al., 1999) to allow sufficient light penetration for polymerization, resulting in enhanced physical and mechanical properties (Versluis et al., 1996).

The main deleterious effect in this conversion process is polymerization shrinkage. Shrinkage manifests itself as stress at the bonded cavity walls, which may develop interfacial defects, enamel fractures, cuspal movements, and micro-cracks (Carvalho et al., 1996; Han et al., 2005; Park et al., 2008). The control of contraction stress of dental composites is essential to ensure margin integrity and restoration longevity (Drummond, 2008). The incremental technique minimizes the deleterious of polymerization shrinkage, resulting in a lower C-factor during polymerization of each layer (Lazarchik et al., 2007; Park et al., 2008; Van Ende et al., 2013).

However, the incremental technique presents some disadvantages such as the possibility of incorporating voids, or contamination between composite layers, resulting in possible bond failures between increments. In addition, the time required to place and polymerize each layer is longer than

in the bulk-filling technique (Abbas et al., 2003; Sarrett 2005; El-Safty et al., 2012; Jang et al., 2015; Rosatto et al., 2015).

Recently, a new category of composites with modified chemical compositions to reduced polymerization shrinkage has been marketed for bulk application in direct posterior composite restorations. The bulk-fill composites can be inserted in a single increment of 4 to 5 mm in thickness and cured in one single step without the adverse effects on polymerization shrinkage stresses and degree of conversion (Czasch; Ilie, 2013; Tiba et al., 2013; Ilie et al., 2013). This allows a significant time reduction of the procedure (El-Damanhoury; Platt, 2014; Moorthy et al., 2012).

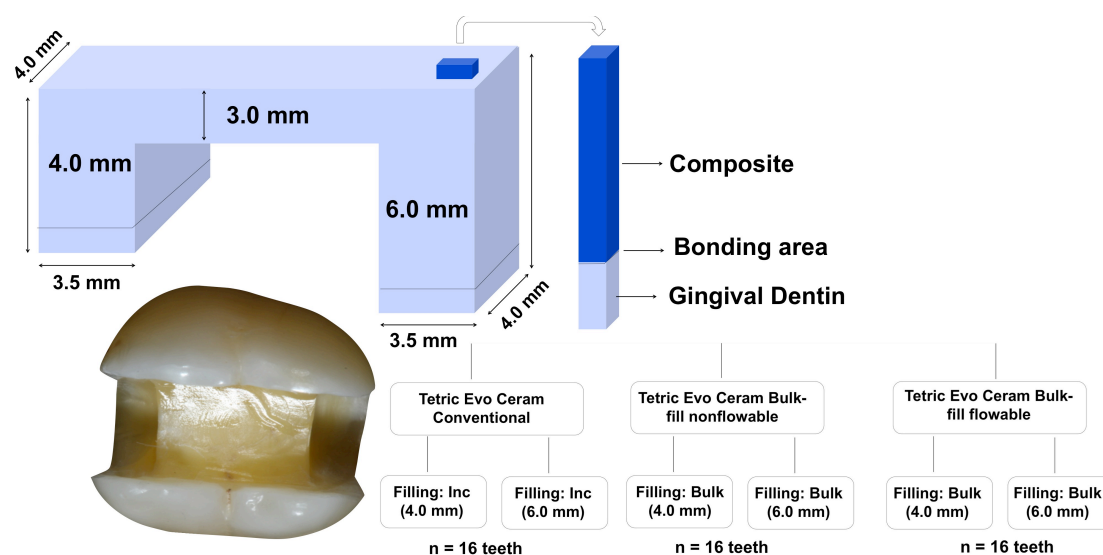
Recent studies have evaluated the performance of bulk-fill composites in clinically relevant cavities such as large Class II MOD preparations (de Campos et al., 2014; Al-Harbi et al., 2015; Kumagai et al., 2015). Several studies have shown similar or better performance of restorations made with bulk fill when compared to those made with traditional composite resins placed incrementally (Garcia et al., 2014; de Campos et al., 2014; Rengo et al., 2015; Al-Harbi et al., 2015; Kumagai et al., 2015). Bulk-fill composites are commercially available in different viscosities, low-viscosity (flowable) and high-viscosity (nonflowable), however, currently there is no information on whether they would perform similarly if subjected to thermo-mechanical challenge.

The null hypotheses tested were: (1) there is no difference in  $\mu$ TBS to dentin in Class II MOD cavities restored with a conventional resin composite, a nonflowable and a flowable bulk-fill composite; and (2) the proximal box depth does not affect bond strength to the gingival walls of MOD cavities.

## MATERIALS AND METHODS

### *Experimental design*

This study was approved by the Guarulhos University's Ethic Committee. Forty-eight molars received standardized Class II MOD preparations with 4 or 6 mm of depth in the distal and mesial proximal boxes, respectively. Two experimental and one control group with 16 teeth each were obtained by a factorial design. The factors under study were "composite" in 3 levels: bulk-fill nonflowable, bulk-fill flowable and conventional nanohybrid composite and "cavity depth" in 2 levels: 4 and 6 mm (Figure 1).



**Figure 1.** Flow chart of the study obtained by a factorial design.

### *Cavity Preparation*

Sound human molars extracted for therapeutic reasons were stored in 1% thymol solution and debrided of residual plaque and calculus. The roots were removed with a diamond saw (IsoMet 1000; Buehler Ltd, Lake Bluff, IL, USA) under water cooling, then the pulp chambers were cleaned and filled with AdheSE Universal (Ivoclar Vivadent, Schaan, Liechtenstein) self-etch



adhesive system and Variolink Veneer (Ivoclar Vivadent, Schaan, Liechtenstein).

Standardized Class II MOD preparations were performed using a diamond bur (# 3101 KG Sorensen, Barueri, São Paulo, Brazil) in a high-speed handpiece with air-water spray. The cavity dimensions were: 4 mm width bucco-lingually, 3 mm in depth in occlusal box, and 4 mm or 6 mm depth randomized in proximal boxes with gingival margins placement in dentin

After preparation the cavities were checked for defects using a stereomicroscope at 50X magnification (Fischer Scientific Model SCW, Thailand). Teeth that present cavities with pulpal exposure or enamel margins were excluded and replaced. Teeth were randomly divided into 3 groups according to the material to be tested: Group 1 (G1) – Control, conventional nanohybrid composite resin (Tetric EvoCeram; Ivoclar Vivadent); Group 2 (G2) – Bulk-fill high-viscosity (nonflowable) composite resin (Tetric EvoCeram Bulk Fill; Ivoclar Vivadent) and Group 3 (G3) – Bulk-fill low-viscosity (flowable) composite resin (Tetric EvoFlow Bulk Fill; Ivoclar Vivadent, Schaan, Liechtenstein). Materials composition and batch numbers are reported in Table 1.

### *Restorative procedure*

A metal matrix band was placed around the tooth using a Tofflemire retainer. All cavities were restored with the same adhesive system Adhese Universal (Ivoclar Vivadent, Schaan, Liechtenstein) used as a two-step etch-and-rinse system applied according to the manufacturer's recommendations. All cavity preparations were etched with 37% phosphoric acid (Ivoclar

Vivadent, Schaan, Liechtenstein) for 15 s, washed for 15 s and gently dried with compressed air. The adhesive was dispensed onto a disposable brush and scrubbed into the cavity for 20 s. The solvent was evaporated with an oil- and moisture-free compressed air for at least 5 seconds. Then the adhesive was photo-activated for 10 s using an LED light-curing unit Bluephase G2 (Ivoclar, Vivadent, Schaan, Liechtenstein) with an average power output of  $1.550 \text{ mW/cm}^2$  periodically monitored with a radiometer (Demetron, Kerr, Orange, CA, USA).

The cavities were randomly filled with composites according to groups G1 (TeInc): Tetric EvoCeram shade A2 (Ivoclar, Vivadent, Schaan, Liechtenstein) in 2 or 3 layers, or in bulk increment G2 (TeBul): Tetric EvoCeram Bulk Fill shade IVA (Ivoclar, Vivadent, Schaan, Liechtenstein), and G3 (TeBFI: Tetric EvoFlow Bulk Fill shade IVA (Ivoclar, Vivadent, Schaan, Liechtenstein).

For (TeInc), the incremental technique was used, where both 4-mm and 6-mm boxes were filled in increments of approximately 2-mm thickness consecutively layered starting from the proximal boxes. The 4-mm box received 2 increments and 6-mm box 3 increments, and the occlusal box one increment. Each layer was cured for 20 s using an LED light-curing unit Bluephase G2 (Ivoclar, Vivadent, Schaan, Liechtenstein) whose tip was placed in contact with the coronal edge of the matrix band. With the bulk-fill technique, (TeBul) and (TeBFI), composites were filled the entire cavities MOD and cured for 20 s such as described above. According to manufacturer's recommendation, Tetric EvoCeram Bulk Fill (nonflowable) and Tetric EvoFlow Bulk Fill (flowable) do not need conventional composite

coverage for the occlusal layer. After storage in distilled water at 37°C for 24 h, restored teeth were prepared for the thermo-mechanical challenge.

**Table 1.** Composition, manufacturer and batch number for materials used in the present investigation

<b>Material, manufacturer and batch number</b>	<b>Composite Type, Shade and photoinitiator</b>	<b>Composition</b>
Total Etch®  (Ivoclar, Vivadent), U33824		37% phosphoric acid, silica
Adhese Universal®  (Ivoclar, Vivadent), T33767		Adhese Universal contains methacrylates, ethanol, water, highly dispersed silicon dioxide, initiators and stabilizers.
Tetric EvoCeram®  (Ivoclar, Vivadent), U26271	Nanohybrid composite  Camphorquinone  A2	Dimethacrylates (17–18% weight). The fillers contain barium glass, ytterbium trifluoride, mixed oxide and prepolymer (82–83% weight). Additional contents: additives, catalysts, stabilizers and pigments (<1.0% weight). The total content of inorganic fillers is 75–76% weight or 53–55% volume. The particle size of the inorganic fillers is between 40 nm and 3,000 nm with a mean particle size of 550 nm.
Tetric EvoCeram Bulk Fill®  (Ivoclar, Vivadent), U24443	Bulk-fill high-viscosity  IVA  Ivocerin®	Bis-GMA, UDMA Ba-Al-Si-glass, prepolymer filler (monomer, glass filler and ytterbium fluoride), spherical mixed oxide. Filler 79–81 wt.% (including 17% prepolymers) / 60–61 vol.%
Tetric EvoFlow Bulk Fill®  (Ivoclar, Vivadent), TM0056	Bulk-fill low-viscosity  IVA  Ivocerin®	Dimethacrylates (28 wt.%). The fillers included barium glass, ytterbium trifluoride and copolymers (71 wt.%). Additives, initiators, stabilizers and pigments are additional ingredients (<1.0 wt.%). The total content of inorganic fillers is 68.2 wt.% / 46.4 vol.%. The particle size of the inorganic fillers range between 0.1 µm and 30 µm with a mean particle size of 5 µm,

BisGMA: bisphenol A dimethacrylate, UDMA: urethane dimethacrylate, Ba-Al-Si: barium-alumino-silicate glasses

### *Chewing and thermocycling challenge*

All teeth were subjected simultaneously to a mechanical and thermal cycling challenge in a chewing simulator CS-3 (SD Mechatronik, Germany). The machine parameters was adjusted for 350.000 cycles, with load of 9 Kg cycling at 1.2 HZ for the mechanical stress; the temperature was adjusted for cycles between  $5 \pm 2^{\circ}\text{C}$  and  $55 \pm 2^{\circ}\text{C}$  with a dwell time of 30s.

### *Microtensile Bond Strength Test*

The proximal boxes of teeth were serially sectioned in the buccal/lingual and mesial/distal directions in order to obtain at least 3 beams with a cross-sectional bonded area of approximately  $1 \text{ mm}^2$  at the gingival wall (IsoMet 1000; Buehler Ltd, Lake Bluff, IL, USA). Beams were tested in tension in a Universal Testing Machine (OM-100 Luzerna, Santa Catarina, Brazil) at a crosshead speed of 1 mm/min until fracture. The cross-sectional area of each specimen was measured with a digital caliper (Mitutoyo, Tokyo, Japan) and the microtensile bond strength was expressed in MPa.

Failure mode was recorded using a light microscope at 50X magnification (Fischer Scientific Model SCW, Thailand). Failure mode was classified into 1 of 3 types: AD (adhesive failure between hybrid layer and dentin), CC (cohesive failure in composite resin) and M (mixed failures). The area percentage of each type of failure in each specimen was recorded (Kumagai et al., 2015).

### *Statistical analysis*

Data were submitted to a 2-way ANOVA considering the factors

“composite” and “depth” followed by a post hoc Tukey’s test at a pre-set alpha of 5%, using statistical software (IBM SPSS version 20.0.0, IBM; Armonk, NY, USA). Pre-test failures were not included in the statistical analysis.

## RESULTS

The mean  $\mu$ TBS values are presented in Table 2. Two-way ANOVA showed significant differences for the factor “composite” ( $p < 0.05$ ). But failed to identify significant difference for the factor “depth” ( $p = 0.0720$ ) and for the interaction between factors ( $p > 0.05$ ).

**Table 2-** Means bond strength values in MPa and standard deviation (number of beams/pre-test failure) for the different composite resins applied.

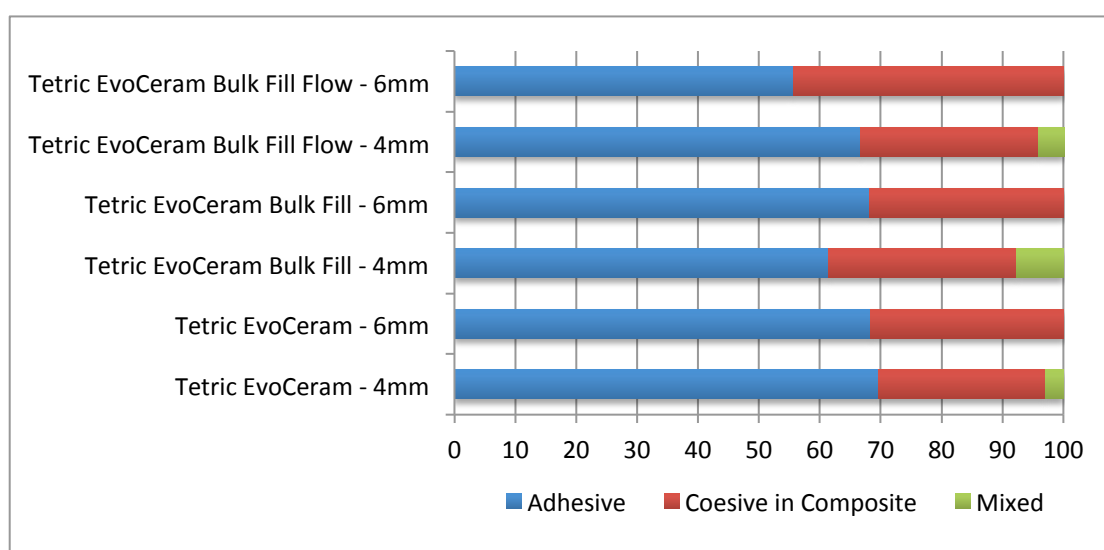
	<b>4 mm</b>	<b>6 mm</b>
<b>Tetric EvoCeram</b>	45.4±14.3 Ba (32/3)	39.7±15.6 Ba (32/13)
<b>Tetric EvoCeram Bulk Fill</b>	50.5±17.5 Aa (32/8)	54.4±14.2 Aa (32/11)
<b>Tetric EvoFlow Bulk Fill</b>	49.5±18.4 Aa (28/8)	55.2±18.6 Aa (28/14)

Means followed by different superscript letters (upper case: column; lower case: row) are significantly different according to Tukey’s test at the 95% confidence level.

There was no significant difference between the 4 and 6 mm deep proximal boxes, independent of the composite used ( $p > 0.05$ ). The bulk-fill composites (flowable and nonflowable) presented significantly higher bond strength values than Control Group, independent of depth. No significant

difference was observed between flowable and nonflowable Bulk Fill composites.

Descriptive data of failure mode analysis (Fig. 2) showed a higher percentage of adhesive failures between composite and dentin for all groups. However, a high percentage of cohesive failures in composite resin were also observed, especially for the flowable Bulk Fill composite placed in a 6 mm increment.



**Figure 2.** Percentage of failure modes in the groups tested.

## DISCUSSION

The incremental layering technique, used as control in the present investigation, has been recognized as a standard procedure in direct posterior composite restorations to reduce polymerization shrinkage stress and achieve an adequate degree of conversion (Park et al., 2008). However, recent investigations have demonstrated similar or improved performance of restorations made with bulk fill composites (Rosatto et al., 2015; Kumagai et al., 2015; Barreto et al., 2015; Van Ende et al., 2013; Van Dijken; Pallesen, 2015; Van Dijken; Pallesen, 2014). Both bulk fill composites tested in the

present investigation produced higher bond strength to the gingival walls of large Class II MOD cavities when compared to the conventional composite placed incrementally. This information leads us to reject the first null hypothesis.

The second null hypothesis was accepted, because no significant differences were observed in bond strength values to the gingival walls of 4 and 6 mm proximal boxes. Cavity depth did not influence bond strength values of any composite independent of the filling technique. Modifications in monomer composition, filler particle distribution and photoinitiator system allow efficient polymerization in thicker layers, with decreased shrinkage stress. Tetric EvoCeram Bulk Fill and Tetric EvoFlow Bulk Fill present Ivocerin as photoinitiator. Ivocerin is a new germanium-based light-initiator with a higher photocuring activity than camphoroquinone that allows the bulk-fill composite to cure faster and deeper than other composite materials without having to increase translucency or reduce working time (Moszner et al., 2008; Fujita et al., 2011; Cramer et al., 2011; Rosatto et al., 2015). However, when the low viscosity bulk fill composite was used in a 6 mm bulk increment, extrapolating manufacturer's recommendation, occurrence of increased number of cohesive failures in composite resin was observed (Fig. 2), probably due to a lower degree of conversion in that region. In addition, an increased number of pre-test failures were also observed. Thus, even though no significant difference in bond strength was observed between 4 and 6 mm-deep cavities, reduced mechanical properties should be expected if a 6 mm increment is used.

The use of bulk-fill composites in deep, wide dental cavities is faster

and easier than the traditional incremental restoration. In addition, the use of low-viscosity bulk fill composites also saves time and improves material handling and adaptation (Benetti et al., 2015; Czash; Ilie, 2013; Ilie; Hickel, 2011). In the present investigation we compared bulk fill materials from the same manufacturer, but with different viscosities. In order to produce a low-viscosity material, filler content was reduced from ~80% in Tetric EvoCeram Bulk Fill, to ~68% in EvoFlow Bulk Fill (Table 1). With lower filler content, different mechanical properties and shrinkage stress development would be expected for EvoFlow Bulk Fill (Rosatto et al., 2015). It is currently accepted that composites with a high modulus of elasticity produce higher shrinkage stresses than do composites with a low modulus of elasticity (Soares et al., 2013; Ferracane, 2005).

The thermo-mechanical load cycling regimen was used in the present investigation to simulate an in vivo environment and challenge the restorations (Gale; Darvell, 1999; Li et al., 2002; Bedran de Castro et al., 2004). Interestingly, the flowable and nonflowable bulk fill composites did not differ between each other, and produced significantly higher bond strength values to the gingival walls of large Class II MOD cavities than did the conventional composite placed incrementally. These results probably occurred due to lower polymerization stress development at the adhesive interface. Tetric EvoCeram Bulk Fill and Tetric EvoFlow Bulk Fill include a new, additional filler technology that is flexible and relieves shrinkage strain during polymerization. This shrinkage stress reliever has been described as a special patented filler functionalized by silanes, which features a lower modulus of elasticity generating an attenuation of forces during polymerization



(Jang et al., 2015). Presence of pre-polymerized fillers included might contribute to decreased volumetric shrinkage (Ilie et al., 2013; Jang et al., 2015). Previous literature has reported favorable results for Tetric EvoCeram Bulk Fill (Benetti et al., 2015; Jang et al., 2015; Kim et al., 2015).

Regardless of the viscosity, the bulk-fill composites used in the present investigation provided higher bond strength values than the conventional composite placed incrementally, proving to be a reliable alternative for restoration of posterior teeth.

## **CONCLUSION**

Compared to a conventional nanohybrid resin composite placed incrementally, the bulk-fill composites tested in two viscosities, nonflowable and flowable resulted in higher bond strength at gingival walls of large Class II MOD cavities after a chewing and thermocycling challenge. Regardless of type of composite resin used, bond strength to the gingival walls of Class II cavities did not differ between 4 and 6 mm-deep proximal boxes.

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## 3.2. Capítulo 2

### MECHANICAL PROPERTIES OF FLOWABLE AND NONFLOWABLE

### BULK-FILL COMPOSITES AS FUNCTION OF INCREMENT THICKNESS

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**Abstract**

This study evaluated the ultimate tensile strength (UTS) and flexural strength (FS) of two bulk-fill composites as function of increment thickness. Three composite resins were used: a conventional composite (TEC - Tetric EvoCeram), a high-viscosity bulk-fill composite (TECB - Tetric EvoCeram Bulk Fill) and a low-viscosity bulk-fill composite (TEFB -Tetric EvoCeram Bulk Fill Flow). 6-mm thick specimens were prepared for each composite. Molds were bulk-filled with composite and light-cured for 20 s. Samples were sectioned horizontally to obtain 3 hourglass-shaped specimens for UTS and 3 rectangular bars-shaped specimens for FS (2, 4 and 6 mm). For UTS, specimens were tested in tension at a crosshead speed of 1.0 mm/min. For FS, the three-point bending test was performed until failure occurred. Significantly lower UTS and FS were observed for the conventional composite when thickness increased to 4 mm. For the bulk-fill composites UTS and FS decreased significantly only at 6 mm. At 4 mm the bulk-fill composites presented significantly higher FS than the conventional composite. Degree of conversion was also determined for (TECB). UTS and FS data were submitted to 2-way ANOVA and Tukey test. The DC for TECB presented highest values at 2 mm, which were significantly higher than 4 mm ( $p < 0.05$ ), and decreased significantly for 6 mm specimens ( $p < 0.05$ ). Both low- and high-viscosity composites presented higher flexural strength than the conventional composite when cured in 4-mm thick bulk increments. However, FS of the low-viscosity bulk-fill composite was significantly lower than the conventional composite at 2 mm.



**Keywords:** bulk-fill composites, flexural strength; polymerization stress, degree of conversion.

## INTRODUCTION

The restorative procedure with conventional methacrylate resin-based composites requires multiple layers due to limited depth of cure (Shortall et al., 2008; Leprince et al., 2012) and to reduce the consequences of shrinkage stress (Versluis et al., 1996). Polymerization shrinkage stress of conventional composites can produce changes in marginal integrity, marginal leakage, enamel fractures, cuspal deflection, secondary caries and post-operative sensitivity (Han et al., 2005; Park et al., 2008; Ilie; Hickel, 2011; Van Ende et al., 2013; Czasch; Ilie, 2013).

The control of shrinkage stresses in resin-based composite restorations is essential to ensure marginal integrity and longevity (Drummond, 2008). This need led researchers and industry to seek constant development of materials with low-stress behavior and bulk application for direct resin composite restorative procedures.

These bulk-fill composites are designed to be placed in increments from 4 to 5 mm and cured in one step (Czasch and Ilie, 2013; Tiba et al., 2013; Ilie et al., 2013). As advantages, they provide a faster option than the conventional layering technique (Park et al., 2008; Jang et al., 2015). Recently, bulk-fill composites with different viscosities were introduced in the market. However, it's not known if mechanical properties vary between the high-viscosity and low-viscosity bulk-fill materials. Some bulk-fill composites are flowable due to lower amount of filler particles and need occlusal

coverage with a conventional composite, while others present higher viscosity due to increased amount of filler particles in its composition and can be sculptable. Therefore, as a result of variations in these materials composition, different mechanical properties can be expected.

The long term clinical success of resin composite restorations depends on the efficiency of the polymerization process, in order to enhance mechanical properties and biocompatibility (Bicalho et al., 2014; Leprince et al., 2012). After secondary caries, one of the clinical failures most commonly seen in posterior teeth with composite restorations is the occurrence of fractures (Roulet, 1988; Demarco et al., 2012). Although there was no significant differences to the technique used (bulk-fill vs. incremental) for the bulk fill resins, a recent clinical trial comparing restorations made with bulk-fill composites with traditional resin-based composites in posterior teeth showed that fracture is the most common failure in a 3-year follow-up (Van Dijken; Pallesen, 2015).

The mechanical characterization of bulk-fill composites is very important to understand the biomechanical behavior during oral function. Laboratorial evaluations of composite properties and the specific factors involved in physical behavior help predict clinical outcomes of direct restorations with bulk-fill composites with high- and low-viscosity. Ultimate tensile strength and flexural strength tests can evaluate the behavior of these materials (Fronza et al., 2015) and correlate with clinical outcomes.

Another clinical indication of the bulk-fill composite is the use in deeper cavities as a dentin-replacement. This behavior can be influenced by the

viscosity of the composite. The flexural strength testing can assess the ability of a material to function as a dentin-replacement in high stress bearing areas (Goracci et al., 2014).

To evaluate the maximal increment thickness of bulk-fill composites, the depth of cure can be performed with some mechanical tests. Researches have used different mechanical tests such as, degree of conversion with spectroscopic techniques, acetona shake test, micro-hardness, elastic modulus, pos-gel shrinkage, cuspal strain, compressive strength, ultimate tensile strength and flexural strength (Abbas et al., 2003; Goracci et al., 2014; Bicalho et al., 2014; Leprince et al., 2014; Rosatto et al., 2015).

Therefore the aim of this study was to assess the ultimate tensile strength (UTS) and flexural strength (FS) of two bulk-fill composites (low and high-viscosity), in different thickness compared with a conventional nanohybrid resin composite. A further objective of the investigation was to assess the degree of conversion (DC) of Tetric EvoCeram Bulk Fill in different thicknesses. The null hypotheses to be tested were: (1) there is no difference in the ultimate tensile strength (UTS) and flexural strength (FS) of the different composites; and (2) there is no difference in the mechanical properties of the composite resins with increasing depth.

## **MATERIALS AND METHODS**

### *Study design*

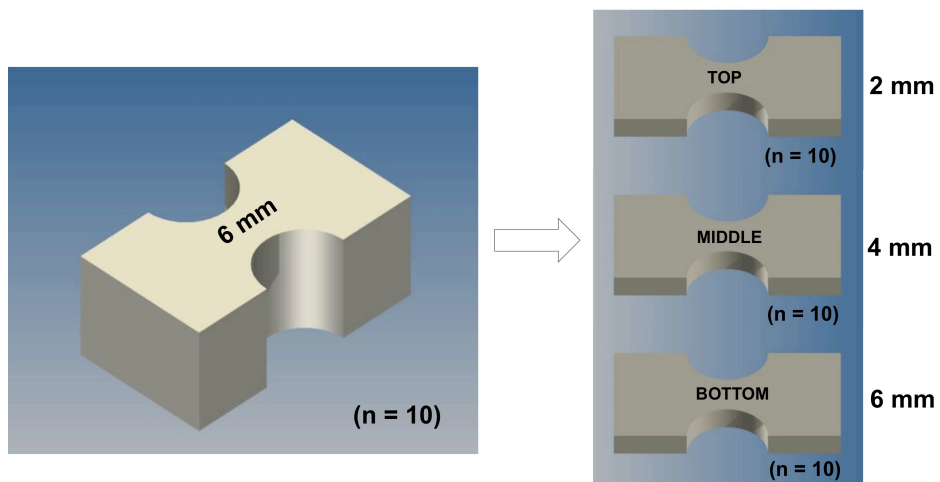
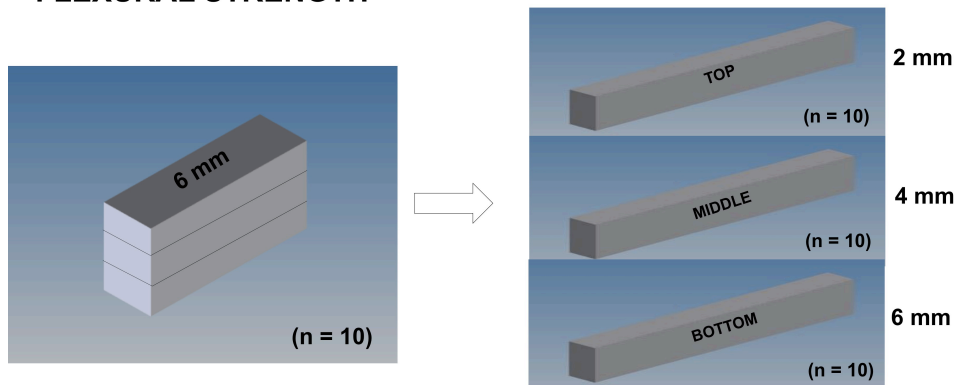
This study used three different resin composites in order to investigate the influence of its composition type (conventional, high-viscosity bulk-fill and low-viscosity bulk-fill) on the mechanical properties. Materials were allocated into 3 groups:

TEC (control group): the conventional nanohybrid composite Tetric EvoCeram (Ivoclar Vivadent, Schaan, Liechtenstein);

TECB: the high-viscosity bulk-fill composite Tetric EvoCeram Bulk Fill (Ivoclar Vivadent);

TEFB: the low-viscosity bulk-fill composite Tetric EvoFlow Bulk Fill Flow (Ivoclar Vivadent).

Specimens used for the ultimate tensile strength and for the flexural strength tests were prepared in silicone molds of 6 mm thickness (n=10). Subsequently, the specimens obtained were sectioned with a diamond saw under water cooling for assessment of these properties at depths of 2, 4 and 6 mm. The degree of polymerization was evaluated after 10 s of photoactivation (Figure 1).

**ULTIMATE TENSILE STRENGTH****FLEXURAL STRENGTH**

**Figure 1.** Flow chart of the study, schematic preparation of specimens

**Table1.** Description of tested materials.

Materials and manufacturer	Abbreviation	Composite Type	Composition	Shade and Batch
Tetric EvoCeram® Ivoclar Vivadent (Schaan, Liechtenstein)	TEC	Nanohybrid composite	Dimethacrylates (17–18 wt.%), barium glass, ytterbium trifluoride, mixed oxide and prepolymer (82–83 wt.%). Additives, catalysts, stabilizers and pigments (<1.0 wt.%). Inorganic fillers (75-76 wt.%) or (53-55 vol%).	A2 U26271
Tetric EvoCeram Bulk Fill® Ivoclar Vivadent (Schaan, Liechtenstein)	TECB	Bulk-fill high-viscosity	Bis-GMA, UDMA Ba-Al-Si-glass, prepolymer filler (monomer, glass filler and ytterbium fluoride), spherical mixed oxide. Filler 79–81 wt.% (including 17% prepolymers) / 60–61 vol.%.	IVA U24443
Tetric EvoFlow Bulk Fill® Ivoclar Vivadent (Schaan, Liechtenstein)	TEFB	Bulk-fill low-viscosity	Dimethacrylates (28 wt.%), barium glass, ytterbium trifluoride, and copolymers (71 wt.%). Additives, initiators, stabilizers and pigments are additional ingredients (<1.0 wt.%). Inorganic fillers (68.2 wt.%) or (46.4 vol%).	IVA TM005 6

BisGMA: bisphenol A dimethacrylate, UDMA: urethane dimethacrylate, Ba-Al-Si: barium-alumino-silicate glasses

### *Ultimate Tensile Strength (UTS) test*

A polyether hourglass shape mold (10 mm in length x 6 mm thick) with a transverse-sectional area of 1.5 mm<sup>2</sup> was used for the specimens preparation. The mold was filled with composites according to groups and a polyester strip was placed on the matrix in order to prevent the oxygen inhibited layer formation. The photo-curing was performed only in the top surface for 20 s using a polywave LED light-curing unit (BluePhase G2, Ivoclar Vivadent, Schaan, Liechtenstein). Photoactivation was performed using the high-power irradiation mode with an average power output of 1550 mW/cm<sup>2</sup> measured by a radiometer (Demetron, Kerr, Orange, CA, USA). After photoactivation, the samples were carefully removed from the mold and stored dry and in the dark for 24 h at 37 ° C before analysis.

After the storage time, the samples with 6 mm were sectioned (Isomet 1000; Buehler Ltd, Lake Bluff, IL, USA) in order to obtain 3 hourglass shape samples with 2 mm each. The samples were identified in top, middle and bottom portions.

The cross-sectional area of each sample was measured with a digital caliper (Mitutoyo, Tokyo, Japan). The samples were adapted in a metal jig testing machine and tensioned in a Universal Testing Machine OM-100 (Odeme, Luzerna, Santa Catarina, Brazil) at a crosshead speed of 1.0 mm/min until rupture. The UTS results were expressed in MPa.

### *Flexural Strength (FS)*

A polyether mold (10 mm length X 2 mm width X 6 mm thickness) was used to prepare the samples. After insertion and light-activation of the composites, samples were carefully removed from the mold and stored dry for 24 h at 37° C in the dark before analysis. After this time samples were sectioned (Isomet 1000; Buehler Ltd, Lake Bluff, IL, USA) in two parts in order to obtain 3 rectangular bars with 2 mm thickness each. The rectangular bars were identified in top, middle and bottom portions. All samples were then submitted to three-point bending test in a OM-100 universal testing machine (Odeme, Luzerna, Santa Catarina, Brazil) at a crosshead speed of 1.0 mm/min until failure occurred. The flexural strength (FS) results were obtained in MPa according to the following equation:

$$\sigma = 3Fl / 2bh^2$$

Where:  $\sigma$  = Flexural Strength,  $F$  is the maximum load (N),  $l$  is the distance between the supports (mm),  $b$  is the sample width (mm) and  $h$  is the sample height (mm).

### *Degree of conversion (DC)*

Degree of conversion analysis of the TECB composite resin was evaluated in 3 different situations (n=5) according to the depth: 2 mm, 4 mm and 6 mm.

A disc-shaped Teflon mold was placed on the horizontal diamond attenuated total reflectance unit (ATR) (Golden Gate, Specac, Woodstock, USA) attached to a Fourier Transform Infrared spectrometer (Tensor 27,



Bruker Optik GmbH, Ettlingen, Germany). As such, the mold allowed the ATR diamond surface to remain exposed. The respective mold (according to the depth evaluated) was used to prepare disk-shaped samples of Tetric EvoCeram Bulk Fill high-viscosity composite ( $n = 5$ ), with the bottom resin surface in direct contact with the diamond surface. The material was placed and polymerized in bulk. During polymerization a polyester strip was positioned between the curing tip and the sample to avoid the oxygen inhibition layer and a 2 mm thick glass slide was positioned directly over and pressed flat to spread the material on the surface. Photo polymerization was performed for 10 s using the same light-curing unit as in the preparation of samples for the UTS and FS measurements. All procedures were performed at 25 °C, and a single trained operator conducted all the steps to minimize the interoperator variability during the specimen preparation.

Infrared (IR) spectra were obtained between 1670  $\text{cm}^{-1}$  and 1570  $\text{cm}^{-1}$  at a resolution of 4  $\text{cm}^{-1}$  (16 scans per spectrum). Monomer conversion was calculated by standard methods using changes in the ratios of the aliphatic-to-aromatic C=C absorption peaks at 1639  $\text{cm}^{-1}$  and 1608  $\text{cm}^{-1}$  in both the uncured and cured states obtained from the infrared spectra according to the following equation, where *abs* is absorbance:

$$\text{DC (\%)} = 1 - \frac{[\text{abs (C=C aliphatic)} / \text{abs (C=C aromatic)}]_{\text{polymer}}}{[\text{abs (C=C aliphatic)} / \text{abs (C=C aromatic)}]_{\text{monomer}}} \times 100$$

### Statistical analysis

UTS and FS data were analyzed by two-way ANOVA and Tukey test. DC data were analyzed by one-way ANOVA and Tukey test. The level of significance was set at 0.05 (SANEST).

## RESULTS

### Ultimate tensile strength (UTS)

Mean ultimate tensile strength (UTS) values obtained for the different composite resins at the different depths are presented in Table 2. Two-way ANOVA showed significant differences for the factors “composite resin” ( $p=0.01140$ ), “depth” ( $p=0.00001$ ) and for the interaction between the two factors ( $p=0.00558$ ).

**Table 2.** Ultimate tensile strength values in MPa (SD) of the composite resins tested in different thicknesses.

	2 mm	4 mm	6 mm
<b>Tetric EvoCeram TEC</b>	35.3 (6.0) Aa	26.6 (6.7) Ab	18.3 (5.4) Ac
<b>Tetric EvoCeram Bulk Fill TECB</b>	27.4 (3.0) Ba	25.3 (3.2) Aa	19.6 (3.2) Ab
<b>Tetric EvoFlow Bulk Fill TEFC</b>	30.3 (3.1) Ba	29.3 (2.6) Aa	22.8 (4.6) Ab

Means followed by different letters (upper case: column; lower case: row) are significantly different by Tukey test at the 95% confidence level.

At 2 mm, TEC composite resin presented the highest UTS values, significantly higher than both bulk-fill composites, which did not differ among them. At 4 and 6 mm, no significant differences were observed among the three composite resins tested ( $p>0.05$ ). However, at 4 mm the TEC presented

UTS values significantly lower than 2 mm, which decreased significantly at 6 mm ( $p < 0.05$ ). On the other hand, both bulk-fill composites presented similar UTS values for 2 and 4 mm groups ( $p > 0.05$ ). However, both bulk-fill composites presented significantly lower UTS values at 6 mm ( $p < 0.05$ ).

### *Flexural Strength*

Mean flexural strength (FS) values obtained for the different composite resins at the different depths are presented in Table 3. Two-way ANOVA showed significant differences for the factors “composite resin” ( $p = 0.01182$ ), “depth” ( $p = 0.00001$ ) and for the interaction between factors ( $p = 0.00006$ ).

**Table 3.** Mean flexural strength values in MPa (SD) of the composite resins tested in different thicknesses.

	<b>2 mm</b>	<b>4 mm</b>	<b>6 mm</b>
<b>Tetric EvoCeram TEC</b>	111.2 (16.2) Aa	55.8 (14.2) Bb	56.2 (19.6) ABb
<b>Tetric EvoCeram Bulk Fill TECB</b>	94.2 (9.5) ABa	86.3 (20.7) Aab	68.9 (16.9) Ab
<b>Tetric EvoFlow Bulk Fill TEFC</b>	82.7 (21.7) Ba	80.1 (12.0) Aa	48.7 (11.9) Bb

Means followed by different letters (upper case: column; lower case: row) are significantly different by Tukey test at the 95% confidence level.

The highest FS values were obtained for the TEC composite resin at 2 mm, which were significantly higher than the TEFC composite ( $p < 0.05$ ). At 2 mm, the TECB composite presented intermediate values, which were not significantly different from the TECB and TEFC composites ( $p > 0.05$ ). On the other hand, at 4 mm, the TEC composite presented significantly lower FS values than those presented by both bulk-fill composites ( $p < 0.05$ ), which did

not differ among them ( $p>0.05$ ). At 6 mm, the TECB resin presented significantly higher FS values than the flowable bulk-fill composite TEFC ( $p<0.05$ ). The TEC composite presented intermediate FS values at 6 mm, which were not significantly different from the bulk-fill materials ( $p>0.05$ ).

The TEC composite presented a significant reduction in FS values at 4 and 6 mm ( $p<0.05$ ), with no difference between 4 and 6 mm FS values ( $p>0.05$ ). Both bulk-fill composites presented similar FS values between 2 and 4 mm groups ( $p>0.05$ ). However, significantly lower FS values were observed for 6 mm groups for the TEFC composite ( $p<0.05$ ).

#### *Degree of conversion*

Mean degree of conversion (DC) values obtained for the TECB composite at the different depths are presented in Table 4. ANOVA showed significant differences among groups ( $p<0.0001$ ). A significant decrease in DC was observed with increasing depth. The highest DC values were obtained at 2 mm, which were significantly higher than 4 mm ( $p<0.05$ ), and decreased significantly for 6 mm specimens ( $p<0.05$ ).

**Table 4.** Degree of conversion in (%) of the high-viscosity bulk-fill composite resin light-cured for 10 seconds in three different thicknesses.

<b>Tetric EvoCeram Bulk Fill (TECB)</b>	<b>Degree of Conversion (%)</b>
<b>2 mm</b>	40.2 (1.3) A
<b>4 mm</b>	37.1 (1.9) B
<b>6 mm</b>	31.4 (1.1) C

Means followed by different letters are significantly different by Tukey test at the 95% confidence level.

## DISCUSSION

Bulk-fill composites have gained increased attention recently (Ilie; Hickel, 2011; Goracci et al., 2014; Fronza et al., 2015; Rosatto et al., 2015). Different brands in different viscosities are currently available in the market. In the current investigation, the mechanical properties of bulk-fill composites offered in different viscosities (high and low) were compared with a conventional incrementally placed composite resin. The first null hypothesis was rejected, because significant differences were observed between the bulk-fill and the conventional nanohybrid composites in different thickness. In addition, significant difference in DC of Tetric EvoCeram Bulk Fill<sup>®</sup> among 2, 4 and 6 mm thickness was observed, which lead us to reject the second null hypothesis.

Since the first report on a bulk-fill material in 2009 (Reis et al., abstract IADR 2009), this new class of materials rapidly gained interest from clinicians, researchers and manufacturers, due to the faster application technique and decreased polymerization shrinkage stresses (Leprince et al., 2013; Czasch; Ilie, 2013; Tiba et al., 2013; Ilie et al., 2013; Benetti et al., 2015). The first bulk-fill material, Surefil SDR Flow<sup>®</sup>, is a low-viscosity material indicated for placement in increments up to 4 mm, as dentin replacement. An additional layer of a conventional composite resin is recommended as a capping layer, to better withstand occlusal loads (Ilie; Hickel, 2011). However, some recently introduced bulk-fill materials do not require a capping layer with a conventional composite resin due to higher filler loading in order to be able to withstand occlusal loads (Goracci et al., 2014), and consequently are high-viscosity materials. In addition, high-viscosity bulk-fill materials handling allow

sculpture of occlusal anatomy, which cannot be performed with low-viscosity materials.

On the other hand, low-viscosity materials present better adaptation and faster insertion in large preparations. Thus, a recent bulk-fill low-viscosity material that does not require a capping layer was introduced in the market (Ivoclar/Vivadent). Tetric EvoFlow Bulk Fill ultimate tensile strength and flexural strength values did not differ from the high-viscosity bulk-fill material when tested in 2 and 4 mm thicknesses. However, at 2 mm, FS and UTS values presented by the TEFB were significantly lower than those presented by the TEC. The high-viscosity bulk-fill material also presented UTS values lower than the conventional composite; however, FS values did not differ from the conventional resin composite. These observations lead us to be cautious with the indication of the flowable bulk-fill composite in high stress bearing occlusion areas.

The nanohybrid composite Tetric EvoCeram is recommended to be applied using the conventional incremental filling technique, not exceeding 2-mm thickness per increment to ensure adequate polymerization and reduce shrinkage stresses (Lazarchik et al., 2007; Van Ende et al., 2013; Finan et al., 2013). When it was cured in a 2-mm increment, the highest UTS and FS values were observed. However, when increment thickness was exceeded to 4 and 6 mm, a significant reduction in mechanical properties was observed. FS and UTS values showed a reduction of approximately 50 and 40%, respectively. At 4 mm, FS values were significantly lower than those obtained by both bulk-fill composites. Conversely, both bulk fill composites presented no reduction in mechanical properties when increment thickness was

increased to 4 mm. This observation is in agreement with previous reports (Goracci et al., 2014; Leprince et al., 2014; Rosatto et al., 2015) and encourages the use of these materials in 4-mm increments.

Modifications in monomer composition, filler particle distribution and photoinitiator system allow efficient polymerization in thicker layers, with decreased shrinkage stress (Bucuta; Ilie, 2014; Garoushi et al., 2013; Goracci et al., 2014). Tetric EvoCeram Bulk Fill and Bulk Fill Flow present Ivocerin as photoinitiator. Ivocerin is a new germanium-based light-initiator with a higher photocuring activity than camphorquinone that allows the bulk-fill composite to cure faster and deeper than other composite materials without having to increase translucency or reduce working time (Moszner et al., 2008; Fujita et al., 2011; Rosatto et al., 2015). However, when both bulk-fill composites were used in a 6-mm increment, extrapolating manufacturer's recommendation, a decrease in UTS and FS values were observed.

Results from the degree of conversion (DC) analysis of the high-viscosity bulk-fill composite demonstrated a significant decrease with the increase in increment thickness from 2 to 4 to 6 mm. When thickness increase to 4 mm, a reduction in DC of 7.7% was observed. However, when thickness increased to 6 mm, a reduction in DC of 21.9% was observed. This reduction in DC for the 4-mm thick specimens was probably not high enough to result in lower mechanical properties (Tarle et al., 2015; Fronza et al., 2015; Alshali et al., 2015). Despite being an important method to characterize the polymeric reaction, a high degree of conversion is not necessarily related to an increase of crosslink density (Tarle et al., 2015). However, the 21.9% reduction in DC resulted in lower FS and UTS for Tetric EvoCeram Bulk Fill. Therefore,

increment thickness of both high- and low-viscosity bulk-fill composites should not exceed the 4-mm thickness recommended by manufacturer.

## **CONCLUSION**

The low-viscosity bulk-fill composite presented significantly lower flexural strength than the conventional composite resin when a 2-mm layer was cured. Thus its indication in stress bearing areas should be made with caution. Both low- and high-viscosity composites presented higher flexural strength than the conventional composite when cured in 4-mm thick bulk increments. The degree of conversion for TECB presented highest values at 2 mm, which were significantly higher than 4 mm and decreased significantly for 6 mm specimens.

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## 4. CONCLUSÃO

As resinas compostas *bulk-fill* de alta e baixa viscosidade, apresentaram maiores valores de resistência de união na parede gengival de amplas cavidades de Classe II após a ciclagem térmica e mecânica em comparação à resina composta nanohíbrida convencional inserida com a técnica incremental.

Contudo as propriedades de resistência à flexão da resina *bulk-fill* de baixa viscosidade na região de 2 mm ficaram abaixo da resina composta convencional, sugerindo a necessidade de cobertura com uma resina composta convencional na região oclusal. A aplicação da resina composta convencional não deve ultrapassar 2 mm de espessura, e das resinas compostas *bulk-fill* não deve ultrapassar 4 mm.

Diante disso, as resinas compostas *bulk-fill* demonstraram ser uma excelente opção para amplas restaurações diretas em dentes posteriores diante do comportamento apresentado frente aos ensaios mecânicos empregados em nosso estudo.

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