

# Low- and high-viscosity bulk-fill resin composites: a comparison of microhardness, microtensile bond strength, and fracture strength in restored molars

Fabiana M. G. França<sup>1</sup>, Jonathan G. B. Tenuti<sup>1</sup>, Isabela P. Broglio<sup>1</sup>, Lara E. J. Paiva<sup>1</sup>, Roberta T. Basting<sup>1</sup>, Cecília P. Turssi<sup>1</sup>, Flávia L. B. do Amaral<sup>1</sup>, André F. Reis<sup>2</sup>, Waldemir F. Vieira-Junior<sup>1</sup>

1. Faculdade São Leopoldo Mandic, Instituto de Pesquisas São Leopoldo Mandic, Departamento de Odontologia Restauradora, Campinas, Brasil.

2. University of Florida, Health Faculty Dental Practice, Operative Division, Gainesville, USA.

## ABSTRACT

The aim of this study was to compare low- and high-viscosity bulk-fill composites for Knoop microhardness (KHN), microtensile bond strength (MTBS) to dentin in occlusal cavities, and fracture strength (FS) in molars with mesial-occlusal-distal restoration. Disk-shaped samples with different thicknesses (2 or 4 mm) of low-viscosity (SDR Flow, Dentsply) and high-viscosity bulk-fill composites (Filtek BulkFill, 3M ESPE; and Tetric-N Ceram Bulk Fill, Ivoclar Vivadent) were prepared for top and bottom KHN analysis (n=10). MTBS to dentin and fracture pattern was evaluated in human molars with occlusal cavities restored with (n=10): conventional nanocomposite (Z350XT, 3M ESPE), low-viscosity (Filtek Bulk-fill Flow, 3M ESPE) or high-viscosity bulk-fill composites (Filtek BulkFill). The FS and fracture pattern of human molar with mesial-occlusal-distal restorations submitted or not to thermomechanical cycling were investigated (n=10) using: intact tooth (control), and restoration based on conventional microhybrid composite (Z250, 3M ESPE), low-viscosity (SDR

Flow) or high-viscosity bulk-fill composites (Filtek BulkFill). The data were submitted to split-plot ANOVA (KHN), one-way ANOVA (MTBS), two-way ANOVA (FS) followed by Tukey's test ( $\alpha=0.05$ ). For KHN, there was no significant difference for the resin composites between the top and bottom. For MTBS, no significant differences among the materials were detected; however, the low-viscosity composite presented lower frequency of adhesive failures. For FS, there was no significant difference between composites and intact tooth regardless of thermomechanical cycling. Low- and high-viscosity bulk-fill composites have comparable microhardness and microtensile bond strength when used in occlusal restorations. Likewise, the bulk-fill composites present similar fracture strength in molars with mesio-occlusal-distal restorations.

Received: April 2021; Accepted: June 2021.

**Keywords:** composite resins - dental restoration failure - hardness - viscosity.

## Resinas compostas bulk-fill de baixa e alta viscosidade: uma comparação de microdureza, resistência de união à microtração e carga à fratura de molares restaurados

### RESUMO

O objetivo deste estudo foi comparar resinas compostas bulk-fill de baixa e alta viscosidade quanto à microdureza Knoop (KHN), resistência de união a microtração (MTBS) em cavidades oclusais e carga à fratura (FS) em molares com restauração mesio-oclusal-distal. Amostras em forma de disco com diferentes espessuras (2 ou 4 mm) de resinas bulk-fill de baixa viscosidade (SDR Flow, Dentsply) e alta viscosidade (Filtek BulkFill, 3M ESPE; e Tetric-N Ceram Bulk Fill, Ivoclar Vivadent) foram obtidas para análise de KHN no topo e na base (n = 10). A MTBS em dentina e o padrão de fratura foram avaliados em molares humanos com cavidades oclusais restauradas com (n = 10): resina composta nanoparticulada convencional (Z350XT, 3M ESPE), resinas bulk-fill de baixa viscosidade (Filtek Bulk-fill Flow, 3M ESPE) ou alta viscosidade (Filtek BulkFill). Foram investigados a FS e o padrão de fratura de molares humanos em restaurações mesial-oclusal-distais sub

metidas ou não à ciclagem termomecânica (n = 10), sendo: dente íntegro (controle), e restaurações baseadas em resina composta microhíbrida convencional (Z250, 3M ESPE); resinas bulk-fill de baixa viscosidade (SDR Flow) ou alta viscosidade (Filtek BulkFill). Os dados foram submetidos a split-plot ANOVA (KHN), one-way ANOVA (MTBS), two-way ANOVA (FS) seguidos do teste de Tukey ( $\alpha = 0,05$ ). Para KHN, não houve diferença significativa entre o topo e a base para as resinas compostas. Para MTBS, não foram detectadas diferenças significativas entre os materiais; entretanto, a resina bulk-fill de baixa viscosidade apresentou menor frequência de falhas adesivas. Para FS, não houve diferença significativa entre os materiais e o dente íntegro, independentemente da ciclagem termomecânica. As resinas bulk-fill de baixa e alta viscosidade têm microdureza e resistência à microtração comparáveis quando usados em restaurações oclusais. Da mesma forma, as

*resinas bulk-fill apresentam resistência à fratura semelhante em molares com restaurações mesio-oclusal-distais.*

**Palavras-chave:** *resinas compostas - falha de restauração dentária - dureza - viscosidade.*

## INTRODUCTION

Resin-based materials are widely used for anterior and posterior tooth restoration. Nevertheless, although their properties make them suitable for clinical use, these materials shrink during polymerization<sup>1</sup>, which is a disadvantage associated with clinical performance. The polymerization shrinkage of resin composites occurs due to the conversion of monomers into a polymer structure<sup>2</sup> accompanied by shrinkage stress, considered a multifactorial phenomenon determined by different factors (e.g., volumetric shrinkage, viscoelastic behavior, kinetics of reaction)<sup>3</sup>. Moreover, chemical and mechanical stresses in the oral environment can have consequences due to material characteristics such as defects in restoration/tooth interface, debonding, enamel micro-cracking, postoperative sensitivity, and cusp deflection<sup>4-7</sup>.

The type of resin composite and clinical technique of application can affect the restoration properties<sup>4-5</sup>. The insertion of several small increments involves more clinical variables and increases the mean time of the procedure, beyond the difficulty of filling small cavities<sup>8</sup>, besides being dependent on the skill and expertise of clinicians. Thus, bulk-fill composites have emerged on the market to enable restorations in layers of up to 4 or 5 mm, according to manufacturers. These materials are commercially available as low- or high-viscosity composites<sup>9, 10</sup>. Low-viscosity bulk-fill composites (flow/flowable) are usually inserted in cavities/tooth preparations with tips and may require a layer of conventional resin composite on top of the restorations<sup>11</sup>. The high-viscosity bulk-fill composites (conventional/paste/sculptable) have photoinitiators with adequate activation in response to the light-curing units<sup>12</sup>, which increase due to the resin's translucency and enable the passage of light more easily. The advantages of these materials include the simplification of the restorative procedure, time saving, low shrinkage stress depending on the technology used by the manufacturer, and adequate radiopacity<sup>10, 12</sup>. However, the bulk application method of tooth restoration can be associated with debonding and greater shrinkage vectors<sup>13</sup>.

The properties and behavior of these materials

need to be fully investigated. A previous study<sup>14</sup> has suggested that a gradual decrease in the microhardness values from the top to the bottom is composite-dependent, and that an increase in thickness could have a negative effect on the microhardness of conventional resin composites and does not interfere with bulk-fill resin composites. Microhardness could even indirectly indicate the degree of conversion of the polymer network<sup>15</sup> or depth of cure, and studies with different variables and purposes are necessary. A gradual reduction in microhardness values can indicate impairment in the degree conversion and consequently affect the longevity of restorations, which must have a suitable conversion to the base of the increment<sup>16</sup>. Considering that high-viscosity bulk-fill composites need longer curing times than low-viscosity bulk-fill composites for optimal properties<sup>17</sup>, it is necessary to evaluate the influence of viscosity of bulk-fill composites on the microhardness and mechanical behavior of restored teeth.

In addition to physical properties, it is also relevant to consider performance during the cyclic efforts of mastication, extensive cavities, or other physicochemical challenges that can occur in the oral environment. The influence of viscosity on bulk-fill resins used for restoration under mechanical cycling and fracture strength of posterior teeth should be investigated, especially considering Class II mesio-oclusal-distal cavities with loss of marginal ridges, which are important strengthening structures for tooth resistance<sup>18</sup>. Furthermore, the bond strength to dentin needs to be evaluated in Class I occlusal restoration, whereas deep cavities can present high stress levels according to the technique or material applied<sup>19</sup>.

Thus, the objective was to evaluate the influence of high- or low-viscosity bulk-fill composites on microhardness, bond strength to dentin in deep occlusal restorations, and fracture strength in molars with mesio-oclusal-distal restorations submitted or not to thermomechanical cycling. The null hypotheses tested were: 1) Increment thickness or viscosity of bulk-fill resin composites does not interfere with microhardness values; 2)

Bond strength to dentin of Class I restorations is not affected by the resin composite used; and 3) Restoration with bulk-fill composites of different viscosities, submitted or not to thermomechanical cycling, does not affect the fracture strength of the restored tooth.

## MATERIALS AND METHODS

### Experiment 1

*Resin composite samples and Knoop microhardness analysis.*

Disk-shaped samples ( $\varnothing$  6 mm) were made from a low-viscosity bulk-fill composite (SDR Flow, Dentsply, Milford, DE, USA) and high-viscosity bulk-fill composites (Filtek Bulk Fill, 3M ESPE, Saint Paul, MN, USA; and Tetric-N Ceram Bulk Fill, Ivoclar Vivadent, Schaan, Liechtenstein). Table 1 provides

the specifications of the materials used. Samples 2 and 4 mm thick were made to evaluate the role of increment thickness on microhardness. For the microhardness test, disk-shaped samples ( $n = 10$ ) were prepared by placing the single incremental material (2 or 4 mm) in a bipartite Teflon matrix. A polyester matrix strip associated with a glass plate was superimposed under pressure of 500 g / 15 s prior to photoactivation to standardize the samples. The resin composites were photopolymerized for 40 s by means of a LED curing light unit (1200 mW/cm<sup>2</sup>; BluePhase, Ivoclar Vivadent, Schaan, Liechtenstein). The bipartite Teflon matrices were standardized at 2 mm, and the 4 mm thick samples were obtained by positioning the two aligned matrices at 2 mm.

The surface Knoop microhardness analysis (KHN)

**Table 1. Description of materials and tests used in the present study.\***

| Material  | Manufacturer                            | Composition  | Analyses/Tests   |
|---|---|--|--|
| SureFil SDR Flow (low-viscosity bulk-fill resin composite)                        | Dentsply Caulk, Milford, DE, USA        | Modified UDMA, dimethacrylate and diluents, barium boron fluoro- aluminosilicate, colorants, strontium aluminosilicate glass, silicon dioxide - amorphous, titanium dioxide  | Top and bottom microhardness<br><br>Fracture strength (mesio-occlusal-distal restoration)  |
| Filtek Bulk-fill Flow (low-viscosity bulk-fill resin composite)                   | 3M ESPE, Saint Paul, MN, USA            | Bis-GMA, Bis-EMA, UDMA, TEGDMA, Ethyl 4-(dimethylamino)benzoate, substituted dimethacrylate, silane treated ceramic, ytterbium fluoride  | Microtensile bond strength (occlusal restoration)  |
| Filtek Bulk fill Posterior Restorative (high-viscosity bulk-fill resin composite) | 3M ESPE, Saint Paul, MN, USA            | Aromatic urethane dimethacrylate, ytterbium dimethacrylate (YbF <sub>3</sub> ), UDMA, 1,12-Dodecane dimethacrylate (DDDMA), water, modified methacrylate monomer, ethyl 4-dimethyl aminobenzoate (EDMAB), benzotriazol, silane treated ceramic, silane treated silica, silane treated zirconia | Top and bottom microhardness<br><br>Microtensile bond strength (occlusal restoration)<br><br>Fracture strength (mesio-occlusal-distal restoration) |
| Tetric N-Ceram Bulk fill (high-viscosity bulk-fill resin composite)               | Ivoclar Vivadent, Schaan, Liechtenstein | Bis-GMA, UDMA, Bis-EMA, dimethacrylates, isofiller (cured dimethacrylates, glass filler and ytterbium fluoride), spherical mixed oxide   | Top and bottom microhardness   |
| Filtek Z350 XT; (conventional nanocomposite)                                      | 3M ESPE, Saint Paul, MN, USA            | Bis-EMA, UDMA, TEGDMA, Bis-GMA, polyethylene glycol dimethacrylate, silane treated zirconia, silane treated ceramic, silane treated silica   | Microtensile bond strength (occlusal restoration)  |
| Filtek Z250 (conventional microhybrid resin composite)                            | 3M ESPE, Saint Paul, MN, USA            | Bis-GMA, Bis-EMA, UDMA, TEGDMA, ceramic treated silane, silica treated silane  | Fracture strength (mesio-occlusal-distal restoration)  |

\*Information provided by the manufacturers. Abbreviations: Bis-GMA, bisphenol-A diglycidyl ether dimethacrylate; Bis-EMA, bisphenol-A hexaethoxylated dimethacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

was performed using a Knoop indenter with a 50-g load for 15 s in a digital microhardness tester (Pantec HVS-1000, Digimess, São Paulo, SP, Brazil). Three indentations were made on the top and bottom of disk-shaped samples, keeping a 100  $\mu\text{m}$  distance between indentations.

## Experiment 2

### *Microtensile bond strength test in occlusal restorations.*

After approval by the Local Ethics Committee in Human Research (# 87973218.3.000.5374), 30 recently extracted human third molars were used. Interproximal radiographs were performed to verify the distance between the central sulcus and the pulp chamber of each tooth, selecting teeth with a minimum distance of 5 mm. Occlusal cavities were made using a cavity preparation machine (Elquip, São Carlos, SP, Brazil) and cylindrical diamond tips positioned parallel to the long axis of the tooth. Cavity dimensions were 4 x 5 x 4 mm, checked after each cavity preparation using a digital caliper. Then the teeth were randomly divided into the following restorative treatments (n = 10):

- (I) Restoration using a conventional nanocomposite (Filtek Z350 XT; 3M ESPE, Saint Paul, MN, USA) and incremental filling technique as a control. The insertion was performed using small increments (2 mm) and light-curing each portion for 10 s.
- (II) Restoration using a low-viscosity bulk-fill composite (Filtek Bulk-fill Flow, 3M ESPE, Saint Paul, MN, USA). The insertion was performed applying a 4-mm layer and light-curing for 20 s.
- (III) Restoration using a high-viscosity bulk-fill composite (Filtek Bulk Fill, 3M ESPE). The insertion was performed using a 4-mm layer and light-curing for 20 s.

A LED curing light (BluePhase, Ivoclar Vivadent AG, Schaan, Liechtenstein) was used for the photoactivation of the resin composite and adhesive system. All teeth were restored using a universal adhesive (Adper Single Bond Universal, 3M ESPE, Saint Paul, MN, USA) applied as a self-etching adhesive system.

After the restorative procedures, the teeth were stored for 7 days at 37 °C. After this time, the teeth were individually fixed on an acrylic plate that was attached to a precision cutting machine (Isomet 1000,

Buehler, Lake Bluff, IL, USA) and high-concentration diamond disc (Buehler) was used to serially section the samples, providing stick-shaped specimens composed of resin composite bonded to dentin. Each tooth resulted in approximately 4 sticks of 1 mm<sup>2</sup>.

Tensile testing was performed in a universal testing machine (EMIC, São José dos Pinhais, PR, Brazil). The sticks were individually attached to the grips of a microtensile device. The test was conducted at a crosshead speed of 0.5 mm/min until debonding or fracture of the stick, and the corresponding force values were obtained in newtons (N). The stick debonding tensions were calculated in megapascals (MPa) after measuring the bonding area with a digital caliper. The interface of the fractured sticks was examined under a stereoscopic microscope (30x magnification) to classify the fracture pattern. The fractures were classified such as: (a) adhesive; (b) cohesive in resin; (c) cohesive in dentin; or (d) mixed.

## Experiment 3

### *Fracture strength in molars with mesio-occlusal-distal restorations.*

Human third molars with MOD cavities were evaluated according to the following treatments/techniques: intact tooth (control, no treatment or preparation); restoration with conventional microhybrid composite (Z250, 3M ESPE, Saint Paul, MN, USA); restoration with low-viscosity bulk-fill composite (SureFil SDR Flow, Dentsply); or restoration with high-viscosity bulk-fill composite (Filtek Bulk Fill, 3M ESPE). These teeth were submitted or not to thermomechanical cycling and were assessed, with n = 10, for fracture strength by axial compressive loading (ACL) and qualitative evaluation of fracture pattern.

Eighty human third molars without caries, stains, or cracks (Local Ethics Committee in Human Research approval - #60999616.4.0000.5374) were used in this assessment. The teeth were scraped with periodontal curettes (Duflex - SS White / Dental Articles Ltd., Rio de Janeiro, RJ, Brazil) and stored in 0.1% thymol solution. All teeth were submitted to dental X-ray to check the distance between the central sulcus and the pulp chamber, and teeth in which they were less than 5 mm apart were excluded. Other variables measured were total tooth, crown, and root length, in order to ensure that tooth sizes were distributed evenly among the groups. After the

measurements, the teeth were classified according to size as small, medium and large, and randomly assigned to groups so that all groups consisted of similar amounts of each size.

To simulate the periodontal ligament<sup>20</sup>, the dental roots were immersed in wax (# 7 Lysanda Produtos Odontológicos Ltd., São Paulo, SP, Brazil), obtaining a layer 0.2 mm thick. A polyvinyl chloride ring (PVC, Tigre S.A., Joinville, SC, Brazil) 25 mm in diameter and 25 mm high was placed around the root that was embedded with a polystyrene resin. After this, a soft polyether impression material (Impregum, 3M ESPE, Seefeld, Germany) was manipulated according to the manufacturer's instructions and applied to the root. MOD cavities were prepared using a cavity preparation machine (Elquip, São Carlos, SP, Brazil) associated with copious air-water spray. The preparations were performed with 1/3 of the intercuspal width, within a 5 mm-deep occlusal box, and without a proximal box using a diamond bur (#3145 KG Sorensen Ind. e Com. Ltd, Cotia, SP, Brazil).

All restored groups received an application of 35% phosphoric acid (3M ESPE) for 15 s for dentin and 30 s for enamel; flush with water for 15 s; removal of the excess water with a light air jet for 2 s; application of the adhesive system (Adper Single Bond 2, 3M ESPE, Saint Paul, MN, USA) according to the manufacturer's recommendation; and photoactivation for 20 s by the LED curing light (BluePhase, Ivoclar Vivadent AG, Schaan, Liechtenstein). The groups were divided according to the following description:

- (I) Control: Intact tooth without preparation or restoration.
- (II) Conventional microhybrid composite (Z250): The composite was incrementally inserted in three oblique layers that were photoactivated individually.
- (III) Low-viscosity bulk-fill composite (SDR): The material was inserted in a single layer of 4 mm and photoactivated, followed by the layer insertion (1 mm) of microhybrid resin (Filtek Z250, 3M ESPE) and photoactivated.
- (IV) High-viscosity bulk-fill composite (Filtek Bulk Fill): The resin was inserted in a single layer (5 mm) and photoactivated for 60 s: 20 s from the occlusal surface, 20 s from the buccal surface, and 20 s from the lingual surface.

The resin-based materials of all restored groups were photoactivated by the LED curing light unit mentioned above. Twenty teeth were subjected to each treatment, and half of each group was submitted to a thermomechanical cycling test, establishing  $n=10$  per group.

Thermomechanical cycles were simulated to induce material fatigue (Elquip, São Carlos, SP, Brazil). The teeth received loading in the axial direction and were cycled 100,000 times with 50 N load and 2 Hz frequency. During the test, the teeth were stored at a relative humidity and submerged cyclically between 5 °C and 55 °C (1 min). The compressive loading test was performed in a Universal Testing Machine (EMIC DL 2000, São José dos Pinhais, PR, Brazil) with axial loading of compression, at 0.5 mm/min (crosshead speed). The values obtained were expressed in newtons (N). After the fracture strength test, the teeth were evaluated for fracture pattern and classified as: (a) coronary fracture up to the middle third; (b) coronary fracture up to the cervical; (c) root fracture up to the cervical; and (d) severe root fracture in the middle and apical third.

### Statistical analysis

The statistical models used followed the experimental design of each experiment. All analyses were performed at the SAS (SAS Institute Inc., Cary, NC, USA, Release 9.2, 2010) considering the significance level of 5%. After the exploratory analysis, the KHN data were submitted to split-plot analysis of variance (ANOVA) and Tukey's test for multiple comparisons. The split-plot ANOVA was used because the experiment was performed considering two factors (bulk-fill composite and thickness), and the KHN values of the top and bottom were considered as a subplot. This analysis considered main factors, double and triple interactions.

The results obtained for occlusal restorations were evaluated by one-way ANOVA to determine whether the bond strength values were influenced by the resin composite used. A G-test was performed to assess the fracture pattern for microtensile bond strength test. For MOD restorations, the values of fracture strength after logarithmic transformation were analyzed by two-way ANOVA. The two-way ANOVA was used in order to consider the two factors (tooth restoration x thermomechanical cycling) and interactions. The fracture pattern was assessed by Fisher's exact test.

The calculation of sample size was performed using GPower software. The sample size ( $n=10$ ) was provided considering the power setting of 0.80, significance level of 0.05, and following parameters for the detectable minimum effect sizes: 0.51 (large) for KHN; 0.52 (large) for fracture pattern; and 0.38 (medium to large) for fracture strength.

## RESULTS

The KHN results (Table 2) had the following  $p$ -values:  $p(\text{composite}) = 0.0004$ ;  $p(\text{thickness}) = 0.04$ ;  $p(\text{composite vs. thickness}) = 0.01$ ;  $p(\text{top/bottom}) = 0.51$ ;  $p(\text{composite vs. top/bottom}) = 0.16$ ;  $p(\text{thickness vs. top/bottom}) = 0.38$ ;  $p(\text{composite vs. thickness x top/bottom}) = 0.11$ . There was no significant difference between the top and bottom for KHN values ( $p = 0.51$ ). At the top, the high-viscosity bulk-fill composite (Tetric-N) showed significantly higher KHN values for an increment thickness of 4 mm in comparison to 2 mm ( $p = 0.01$ ). At the bottom and increment thickness of 4 mm, the other high-viscosity bulk-fill composite (Filtek BF) presented a significantly lower surface microhardness values than the other composites ( $p$

$= 0.01$ ).

Concerning the results of microtensile bond strength test for occlusal restorations (Table 3), there was no significant difference for MPa values among all groups, regardless of the restorative material used ( $p = 0.15$ ). Nevertheless, there was a statistically significant difference among groups regarding fracture pattern ( $p = 0.04$ ). Adhesive-type fracture patterns were more prevalent in high-viscosity bulk-fill (46.7%) and nanocomposite (47.4%) than in low-viscosity bulk-fill composite (20%). The dentin cohesive-type fracture pattern was more frequent in the tooth restored with low-viscosity bulk-fill composite (50%).

The results of fracture load (Table 4) presented the following  $p$ -values:  $p(\text{treatment}) = 0.88$ ;  $p(\text{cycling}) = 0.81$ ; and  $p(\text{treatment vs. cycling}) = 0.34$ . There was no significant difference between treatments or thermomechanical cycling (with and without) for fracture strength. Fisher's exact test showed that the distribution of the fracture pattern varied according to the treatment ( $p = 0.007$ ), and these results are presented in Table 5. All fractures were coronary, with the majority being coronary until the middle

**Table 2. Surface microhardness values (Mean  $\pm$  SD) in the top and bottom of materials in relation to increment thickness ( $n = 10$ ).\***

| Bulk-fill resin composite |                            | Thickness                        |                                  | Result of split-plot ANOVA   |
|---------------------------|----------------------------|----------------------------------|----------------------------------|--|
|                           |                            | 2 mm                             | 4 mm                             |  |
| Top                       | Low viscosity (SDR)        | 127.50 $\pm$ 24.13 <sup>Aa</sup> | 118.88 $\pm$ 35.66 <sup>Aa</sup> | Main effect:<br>Composite, $p = 0.0004$<br>Thickness, $p = 0.04$<br>Top/bottom, $p = 0.51$<br>Interaction effect:<br>Composite x Thickness, $p = 0.01$<br>Composite x Top/bottom, $p = 0.16$<br>Thickness x Top/bottom, $p = 0.38$<br>Composite x Thickness x Top/bottom, $p = 0.11$ |
|                           | High viscosity (Filtek BF) | 103.23 $\pm$ 21.65 <sup>Aa</sup> | 109.69 $\pm$ 28.84 <sup>Aa</sup> |  |
|                           | High viscosity (Tetric-N)  | 98.99 $\pm$ 8.52 <sup>Ba</sup>   | 141.72 $\pm$ 30.86 <sup>Aa</sup> |  |
| Bottom                    | Low viscosity (SDR)        | 130.52 $\pm$ 22.22 <sup>Aa</sup> | 134.38 $\pm$ 26.37 <sup>Aa</sup> |  |
|                           | High viscosity (Filtek BF) | 100.40 $\pm$ 17.04 <sup>Aa</sup> | 99.16 $\pm$ 12.20 <sup>Ab</sup>  |  |
|                           | High viscosity (Tetric-N)  | 114.72 $\pm$ 30.86 <sup>Aa</sup> | 133.76 $\pm$ 21.65 <sup>Aa</sup> |  |

\*Values followed by different letters (uppercase in horizontal and lowercase in vertical) differ from each other. Abbreviations: SDR, SureFil SDR Flow; Filtek BF, Filtek Bulk Fill Posterior Restorative; Tetric-N, Tetric N-Ceram Bulk Fill.

**Table 3. Results (Mean  $\pm$  SD) of microtensile bond strength test (MPa) and relative frequency (%) of fracture pattern according to the restorative materials used for occlusal restorations ( $n = 10$ ).\***

| Resin composite                             | MPa                            | Fracture pattern (%)         |       |                    |                   |
|---|--------------------------------|------------------------------|-------|--------------------|-------------------|
|   |                                | Adhesive                     | Mixed | Cohesive in dentin | Cohesive in resin |
| Conventional nanocomposite (Filtek Z350 XT) | 28.62 $\pm$ 18.38 <sup>a</sup> | 47.4                         | 36.8  | 10.5               | 5.3               |
| Low-viscosity bulk-fill (Filtek BF Flow)    | 31.88 $\pm$ 14.53 <sup>a</sup> | 20                           | 15    | 50                 | 15                |
| High-viscosity bulk-fill (Filtek BF)        | 41.40 $\pm$ 11.91 <sup>a</sup> | 46.7                         | 13.3  | 20                 | 20                |
| Results of statistical tests                | MPa, $p = 0.15$                | Fracture pattern, $p = 0.04$ |       |                    |                   |

\*For MPa results, mean  $\pm$  SD followed by the same letters did not differ from each other. Abbreviations: Filtek BF, Filtek Bulk Fill.

**Table 4. Mean  $\pm$  SD of fracture load (N) according to treatment and thermomechanical cycling (n = 10).\***

|  | Thermomechanical cycling         |                                   | Result of two-way ANOVA  |
|--|----------------------------------|-----------------------------------|--|
|  | without                          | with                              |  |
| Intact tooth (control)                         | 2188.4 $\pm$ 445.8 <sup>Aa</sup> | 2005.5 $\pm$ 688.5 <sup>Aa</sup>  | Main effect:<br>Treatment, $p = 0.88$<br>Cycling, $p = 0.81$<br><br>Interaction effect:<br>Treatment x Cycling, $p = 0.34$ |
| Conventional microhybrid composite (Z250)      | 2310.8 $\pm$ 717.2 <sup>Aa</sup> | 2165.1 $\pm$ 775.9 <sup>Aa</sup>  |  |
| Low-viscosity bulk-fill composite (SDR)        | 2025.7 $\pm$ 713.7 <sup>Aa</sup> | 2069.7 $\pm$ 673.3 <sup>Aa</sup>  |  |
| High-viscosity bulk-fill composite (Filtek BF) | 1906.2 $\pm$ 589.2 <sup>Aa</sup> | 2507.5 $\pm$ 1002.9 <sup>Aa</sup> |  |

\*Values followed by the same letters (uppercase in horizontal and lowercase in vertical) did not differ from each other ( $p > 0.05$ ). Abbreviations: SDR, SureFil SDR Flow; Filtek BF, Filtek Bulk Fill Posterior Restorative.

**Table 5. Number of fractured samples and frequency distribution (%) of fracture pattern according to cycling and restorative materials used for mesio-occlusal-distal restorations (n = 10).\***

| Cycling                    | Groups                     | Fracture pattern                |                      |                                  |                      |
|----------------------------|----------------------------|---------------------------------|----------------------|----------------------------------|----------------------|
|                            |                            | Coronary up to the middle third | Coronary to cervical | Root fracture up to the cervical | Severe root fracture |
| without                    | Intact tooth (control)     | 6 (60%)                         | 4 (40%)              | 0 (0%)                           | 0 (0%)               |
|                            | Z250 (conventional)        | 7 (70%)                         | 0 (0%)               | 3 (30%)                          | 0 (0%)               |
|                            | SDR (low viscosity)        | 2 (20%)                         | 4 (40%)              | 4 (40%)                          | 0 (0%)               |
|                            | Filtek BF (high viscosity) | 9 (90%)                         | 1 (10%)              | 0 (0%)                           | 0 (0%)               |
| with                       | Intact tooth (control)     | 7 (70%)                         | 3 (30%)              | 0 (0%)                           | 0 (0%)               |
|                            | Z250 (conventional)        | 7 (70%)                         | 3 (30%)              | 0 (0%)                           | 0 (0%)               |
|                            | SDR (low viscosity)        | 4 (40%)                         | 2 (20%)              | 3 (30%)                          | 1 (10%)              |
|                            | Filtek BF (high viscosity) | 6 (60%)                         | 2 (20%)              | 0 (0%)                           | 2 (20%)              |
| <b>Fisher's exact test</b> |                            | $p = 0.007$                     |                      |                                  |                      |

\*Abbreviations: Z250, conventional microhybrid composite; SDR, SureFil SDR Flow, low-viscosity bulk-fill composite; Filtek BF, Filtek Bulk Fill Posterior Restorative, high-viscosity bulk-fill composite.

third for the following groups: intact tooth (with and without cycling); conventional microhybrid composite (Z250) with cycling; and high-viscosity bulk-fill composite (Filtek BF) without cycling. For the conventional microhybrid composite (Z250) without cycling, 70% of the teeth presented a coronary fracture until the middle third, and the remainder had a root fracture up to the cervical. In the treatments with low-viscosity bulk-fill composite (SDR) or high-viscosity bulk-fill composite (Filtek BF) with cycling, the root fractures were observed in the middle and apical thirds.

## DISCUSSION

The present study showed there was no significant difference between the top and bottom in the surface microhardness of bulk-fill composites with different viscosities, so the results fail to reject the first

null hypothesis. Bulk-fill composites have higher translucency than conventional resin composites<sup>12</sup>, and the translucency of resins depends on the factors of increment thickness, dispersion/absorption coefficients of material, pigments, and opacifiers<sup>21-23</sup>. Moreover, increase in the cure depth of a bulk-fill resin can relate not only to higher translucency compared to a conventional resin but also to modified monomers, incorporation of stress relievers, or photoinitiator systems included in its composition<sup>21</sup>, especially because the decrease in polymerization shrinkage is manufacturer-dependent and may be associated with different attenuation mechanisms<sup>7</sup>. The viscosity of material is influenced by monomer and filler content associating with the reaction kinetics and final polymerization. Modifications of the monomer and filler components make bulk-fill resins more translucent/transparent by adding

so-called polymerization modulators or initiation boosters<sup>14, 24</sup>. Considering the low-viscosity bulk-fill composites studied, SDR (Dentsply) presented favorable and constant results regarding top or bottom microhardness and fracture strength of restored molars. SDR presents a uniform degree of conversion at a depth of 1-4 mm and a low proportion of internal gaps in dental restoration<sup>25</sup>. According to the manufacturer, an adequate degree of conversion and reduction of shrinkage stress is related to chemically modified polymer formation, which is flexible with the homogeneous network. Furthermore, SDR is composed of a modulator chemically incorporated to UDMA that could interact synergistically with camphorquinone, culminating in adequate polymerization<sup>21</sup>.

Considering the KHN results, Tetric-N was the high-viscosity bulk-fill composite that presented a significantly higher top KHN value at 4 mm than at the increment thickness of 2 mm. Tetric-N has a photoinitiator described as a polymerization booster (Ivocerin®) which, associated with the camphorquinone/amine initiator system, can polymerize the material in depth<sup>21</sup>. Ivocerin® is a dibenzoyl germanium derivative system with the highest absorption of wavelengths around 370 to 460 nm<sup>26</sup>. At the bottom, with increment thickness of 4 mm, the other high-viscosity bulk-fill composite studied (Filtek Bulk Fill) showed significantly lower surface microhardness compared to the other bulk-fill composites. This can be explained by the absence of TEGDMA in the composition of this material, which has approximately half the molecular weight of the other monomers<sup>27</sup>. The microhardness variable has a high correlation with the filler content of material, and the lower microhardness values at the bottom surface of the Filtek Bulk Fill could occur due to light attenuation<sup>28</sup>. Nevertheless, no differences were found between increments of 2 mm or 4 mm, indicating an adequate depth of cure.

Consistently with the KHN results, the bond strength results (MPa) showed that there were no differences between the conventional nanocomposite, high- and low-viscosity bulk-fill composites, so the results fail to reject the second null hypothesis. In posterior teeth cavities, bond strength is expected to be equal to or lower than the dental cohesive strength, because in case of failures after the adhesion process, it is preferable that these failures should occur in the material, protecting the remaining dental

structure. Nevertheless, sufficient bond strength is necessary in order to resist the mechanical and chemical challenges in the oral environment. In the present study, the bulk-fill composites did not differ from conventional nanocomposite for MPa. This result is relevant because the conventional nanocomposite has been extensively studied and its performance is considered satisfactory<sup>29</sup>. Adhesive-type failures were more usual in the high-viscosity composites (bulk-fill or conventional) that are nonflowable and sculptable, while cohesive-type failures occurred more frequently in low-viscosity bulk-fill composite. Flowable resin composites generally have lower filler loading and are more fluid<sup>30</sup>, promoting adequate adaptation in the pulpal floor and decreasing internal irregularities of the preparation. Other factors could contribute to the fracture pattern results, such as the elastic modulus of high-viscosity composites, which is higher than in the low-viscosity composites<sup>31</sup> and consequently promotes lower capacity for flow and adaptation on the deeper walls. Furthermore, bond strength was measured in the pulpal floor in deep occlusal cavities. This area is challenging for adhesive procedures due to the humidity, permeability, and characteristics of the intertubular dentin<sup>32</sup>, which may accentuate premature loss of adhesion.

Considering the fracture strength in molars with MOD restorations, the results fail to reject the third null hypothesis because no difference was observed in the fracture strength values of cavities restored with a conventional resin composite or bulk-fill composites of different viscosities, including the comparison with the intact tooth. This result is compatible with a previous study<sup>33</sup> in which teeth treated endodontically with conventional composite resins or bulk-fill composites were tested for fracture strength, and there was also no difference between these materials, even when compared to the intact teeth. A high elastic modulus can inhibit the ability to deform, generating greater stress in the dental structures<sup>34</sup>. The adequate elastic modulus of bulk-fill resins to substitute dentin or enamel<sup>20</sup> allows the material to deform and absorb the stress generated during the thermomechanical cycles, similarly to the microhybrid resin used as a control. However, further studies should evaluate the behavior of teeth fully restored with low-viscosity composites, since in the present study, a conventional composite surface layer was used, considering the high occlusal



load to which MOD cavities are submitted.

In a clinical situation, the size, type, and location of the cavity should guide the choice of material; therefore, the present study investigated the behavior of these materials in cavities/restorations of different configurations. The mechanical properties of the different materials vary considerably. Thus, the low-viscosity composites seem appropriate for liner, deep cavities and restorations after endodontic treatments, since the low viscosity facilitates adaptation in less accessible spaces<sup>30</sup>. On the other hand, high-viscosity composites are materials with more filler content and could be used in cavities considering their resistance to fracture or wear<sup>11</sup>. The fracture strength and the bond strength presented by both viscosities of bulk-fill composites are similar. The fracture strength of molars restored with these composites is equivalent to that of the tooth structure and the bond strength is comparable to that of a conventional composite. However, these resin composites require care during the insertion step, especially in the deep walls, in order to reduce adhesive or adaptation failures. Under controlled situations, as employed in the present study, the

behavior of the different conventional or bulk-fill resin composites, regardless of viscosity, was similar, in agreement with previous investigations<sup>35-37</sup> that reported clinical performance of bulk-fill resin composites similar to that of conventional resin composites. Further *in vitro* studies and clinical trials of bulk-fill composites remain necessary to continue their validation.

In general, according to the results of experiments, relevant findings were: the viscosity of bulk-fill resin composites included in this study did not influence the microhardness of top and bottom, regardless of increment thickness (2 or 4 mm); the dentin bond strength of bulk-fill resin composites, regardless of viscosities inserted as a single increment, was similar to conventional nanocomposite incrementally inserted in deep occlusal cavities, although adhesive failures were less frequent in low-viscosity bulk-fill composites compared to other materials. Moreover, the fracture strength of molars with MOD cavities restored with bulk-fill composites, regardless of viscosity, was similar to intact tooth and conventional microhybrid resin restorations, even after thermomechanical cycling.

#### DECLARATION OF CONFLICTING INTERESTS

The authors declare no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

#### FUNDING

None.

#### CORRESPONDENCE

Dr. Fabiana Mantovani Gomes França

Faculdade São Leopoldo Mandic, Instituto de Pesquisas São Leopoldo Mandic

Rua José Rocha Junqueira, 13 – Swift, Campinas – SP, 13045-755

Brasil

biagomes@yahoo.com

#### REFERENCES

1. Ferracane JL. Resin composite-state of the art. *Dent Mater* 2011; 27: 29-38.
2. Al Sunbul H, Silikas N, Watts DC. Polymerization shrinkage kinetics and shrinkage-stress in dental resin-composites. *Dent Mater* 2016;32:998-1006.
3. Braga RR, Ballester RY, Ferracane JL. Factors involved in the development of polymerization shrinkage stress in resin-composites: a systematic review. *Dent Mater* 2005;21:962-970.
4. Bicalho AA, Pereira RD, Zanatta RF, Franco SD et al. Incremental filling technique and composite material. Part I: cuspal deformation, bond strength, and physical properties. *Oper Dent* 2014;39:E71-E82.
5. Bicalho AA, Valdivia AD, Barreto BC, Tantbirojn D et al. Incremental filling technique and composite material. Part II: shrinkage and shrinkage stresses. *Oper Dent* 2014;39:E83-E92.
6. Tantbirojn D, Pfeifer CS, Amini AN, Versluis A. Simple optical method for measuring free shrinkage. *Dent Mater* 2015;31:1271-1278.
7. Soares CJ, Faria-e-Silva AL, Rodrigues MP, Vilela ABF et al. Polymerization shrinkage stress of composite resins and resin cements - What do we need to know? *Braz Oral Res* 2017;31:e62.
8. Kumagai RY, Zeidan LC, Rodrigues JA, Reis AF et al. Bond Strength of a Flowable Bulk-fill Resin Composite in Class II MOD Cavities. *J Adhes Dent* 2015;17:427-432.
9. Kim EH, Jung KH, Son SA, Hur B et al. Effect of resin thickness on the microhardness and optical properties of bulk-fill resin composites. *Restor Dent Endod* 2015;40:128-135.
10. Fugolin APP, Pfeifer CS. New Resins for Dental Composites. *J Dent Res* 2017; 96: 1085-1091.
11. Van Ende A, de Munck J, Van Landuyt KL, Poitevin A et

- al. Bulk-filling of high C-factor posterior cavities: Effect on adhesion to cavity-bottom dentin. *Dent Mater* 2013;29:269-277.
12. Zorzin J, Maier E, Harre S, Fey T et al. Bulk-fill resin composites: polymerization properties and extended light curing. *Dent Mater* 2015;31:293-301.
  13. Kaisarly D, El Gezawi M, Keßler A, Rösch P et al. Shrinkage vectors in flowable bulk-fill and conventional composites: bulk versus incremental application. *Clin Oral Investig* 2021;25:1127-1139.
  14. Flury S, Peutzfeldt A, Lussi A. Influence of increment thickness on microhardness and dentin bond strength of bulk fill resin composites. *Dent Mater* 2014;30:1104-1112.
  15. Yan YL, Kim YK, Kim KH, Kwon TY. Changes in Degree of Conversion and Microhardness of Dental Resin Cements. *Oper Dent* 2010;35:203-210.
  16. Flury S, Hayoz S, Peutzfeldt A, Hüsler J et al. Depth of cure of resin composites: is the ISO 4049 method suitable for bulk fill materials. *Dent Mater* 2012;28:521-528.
  17. Miletic V, Pongprueksa P, De Munck J, Brooks NR et al. Curing characteristics of flowable and sculptable bulk-fill composites. *Clin Oral Investig* 2017;21:1201-1212.
  18. Shahrabaf S, Mirzakouchaki B, Oskoui SS, Kahnamoui MA. The effect of marginal ridge thickness on the fracture resistance of endodontically-treated, composite restored maxillary premolars. *Oper Dent* 2007;32:285-290.
  19. Ausiello PP, Ciarabella S, Lanzotti A, Ventre M et al. Mechanical behavior of Class I cavities restored by different material combinations under loading and polymerization shrinkage stress. A 3D-FEA study. *Am J Dent* 2019;32:55-60.
  20. Rosatto CM, Bicalho AA, Veríssimo C, Bragança GF et al. Mechanical properties, shrinkage stress, cuspal strain and fracture resistance of molars restored with bulk fill composites and incremental filling technique. *J Dent* 2015;43:1519-1528.
  21. Kim RJ, Kim YJ, Choi NS, Lee IB. Polymerization shrinkage, modulus, and shrinkage stress related to tooth-restoration interfacial debonding in bulk-fill composites. *J Dent* 2015;43:430-439.
  22. Haas K, Azhar G, Wood DJ, Moharamzadeh K et al. The effects of different opacifiers on the translucency of experimental dental composite resins. *Dent Mater* 2017;33:e310-e316.
  23. Finan L, Palin WM, Moskwa N, McGinley EL et al. The influence of irradiation potential on the degree of conversion and mechanical properties of two bulk-fill flowable RBC base materials. *Dent Mater* 2013;29:906-912.
  24. Ilie N, Bucuta S, Draenert M. Bulk-fill resin-based composites: an in vitro assessment of their mechanical performance. *Oper Dent* 2013;38:618-625.
  25. Fronza BM, Rueggeberg FA, Braga RR, Mogilevych B et al. Monomer conversion, microhardness, internal marginal adaptation, and shrinkage stress of bulk-fill resin composites. *Dent Mater* 2015;31:1542-1551.
  26. Moszner N, Fischer U, Ganster B, Liska R et al. Benzoyl germanium derivatives as novel visible light photoinitiators for dental materials. *Dent Mater* 2008;24:901-907.
  27. Gajewski VE, Pfeifer CS, Fróes-salgado NR, Boaro LC et al. Monomers used in resin composites: degree of conversion, mechanical properties and water sorption/solubility. *Braz Dent J* 2012;23:508-514.
  28. Son SA, Park JK, Seo DG, Ko CC et al. How light attenuation and filler content affect the microhardness and polymerization shrinkage and translucency of bulk-fill composites? *Clin Oral Investig* 2017;21:559-565.
  29. de Andrade AK, Duarte RM, Medeiros e Silva FD, Batista AU et al. Resin composite Class I restorations: a 54-month randomized clinical trial. *Oper Dent* 2014;39:588-594.
  30. Baroudi K, Rodrigues JC. Flowable Resin Composites: A Systematic Review and Clinical Considerations. *J Clin Diagn Res* 2015;9:ZE18-ZE24.
  31. Rizzante FAP, Mondelli RFL, Furuse AY, Borges AFS et al. Shrinkage stress and elastic modulus assessment of bulk-fill composites. *J Appl Oral Sci* 2019;27:e20180132.
  32. Pashley DH, Carvalho RM. Dentine permeability and dentine adhesion. *J Dent* 1997;25:355-372.
  33. Isufi A, Plotino G, Grande NM, Ioppolo P et al. Fracture resistance of endodontically treated teeth restored with a bulk fill flowable material and a resin composite. *Ann Stomatol (Roma)* 2016;7:4-10.
  34. Lohbauer U, Frankenberger R, Krämer N, Petschelt A. Strength and fatigue performance versus filler fraction of different types of direct dental restoratives. *J Biomed Mater Res B Appl Biomater* 2006;76:114-120.
  35. Kruly PC, Giannini M, Pascotto RC, Tokubo LM et al. Meta-analysis of the clinical behavior of posterior direct resin restorations: Low polymerization shrinkage resin in comparison to methacrylate composite resin. *PLoS One* 2018;13:e0191942.
  36. Veloso SRM, Lemos CAA, de Moraes SLD, do Egito Vasconcelos BC et al. Clinical performance of bulk-fill and conventional resin composite restorations in posterior teeth: a systematic review and meta-analysis. *Clin Oral Investig* 2019;23:221-233.
  37. Balkaya H, Arslan S. A two-year clinical comparison of three different restorative materials in class II cavities. *Oper Dent* 2020;45:E32-E42.