UNIVERSIDADE ESTADUAL DE CAMPINAS
FACULDADE DE ODONTOLOGIA DE PIRACICABA

BRUNA SCARCELLO STRINI

AVALIAÇÃO COMPARATIVA DE RESINAS BULK-FILL: MICRODUREZA KNOOP, RESISTÊNCIA À TRAÇÃO DIAMETRAL E GRAU DE CONVERSÃO

COMPARATIVE EVALUATION OF BULK-FILL RESINS: KNOOP MICROHARDNESS, DIAMETRAL TENSILE STRENGTH AND DEGREE OF CONVERSION

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Trabalho de Conclusão de Curso apresentado à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para obtenção do título de Cirurgião Dentista.

Undergraduate final work presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Dental Surgeon.

Orientador: Ms. Joyce Figueiredo de Lima Marques

Coorientador: Prof. Dr. Flávio Henrique Baggio Aguiar

ESTE EXEMPLAR CORRESPONDE À VERSÃO FINAL DO TRABALHO DE CONCLUSÃO DE CURSO APRESENTADO PELA ALUNA BRUNA SCARCELLO STRINI E ORIENTADA PELO PROF. DR. FLÁVIO HENRIQUE BAGGIO AGUIAR.

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RESUMO

Considerando a recente introdução de novas formulações de resinas Bulk-Fill no mercado odontológico, este trabalho teve como objetivo avaliar e comparar a microdureza Knoop (KHN), a resistência à tração diametral (DTS) e o grau de conversão (DC) de três resinas Bulk-Fill e uma convencional. Para isso, 60 amostras (n=15; 8 mm ø x 4 mm de altura) foram confeccionadas usando um molde de silicone por adição. As resinas Filtek Bulk-Fill (FBF), Tetric N-Ceram Bulk-Fill (TNC) e SonicFill 2 (SF2), foram acomodadas em incrementos de 4 mm, e a resina Filtek Z350 (FZ350) em incrementos de 2 mm. Cada incremento foi fotopolimerizado pelo tempo recomendado pelo fabricante de cada resina. O valor de KHN do topo e da base das amostras foram avaliados usando microdurômetro a 10 gf/10 s, a DTS foi testada sob carga compressiva de 1,0 mm/min e o DC foi medido por espectroscopia FTIR-ATR. Os resultados de DTS e DC foram analisados pela ANOVA e pelo teste de Tukey, enquanto os de KHN, foram analisados por Kruskal-Wallis e Wilcoxon (α=0,05). O topo de todas as amostras apresentou maiores valores de KHN do que da base das mesmas. O topo e a base das amostras do grupo FZ350 apresentou maiores valores de KHN do que os grupos TNC e SF2. Para DTS, FBF apresentou os maiores valores, seguido pela FZ350 e SF2. O maior valor de DC foi obtido pela SF2 e o menor pela FBF. As diferenças na composição das resinas Bulk-Fill testadas influenciaram nos diferentes resultados.

Palavras-chave: Resinas compostas. Dureza. Resistência à tração. Espectroscopia de infravermelho com transformada de Fourier.

ABSTRACT

Due the recent introduction of new formulations of Bulk-Fill resins in the dental market, this study aimed to evaluate and compare Knoop microhardness (KHN), diametral tensile strength (DTS) and degree of conversion (DC) of three Bulk-Fill resins and a conventional one. For this, sixty samples (n = 15; 8 mm ø x 4 mm high) were confectioned using a mold. Filtek Bulk-Fill (FBF), Tetric N-Ceram Bulk-Fill (TNC) and SonicFill 2 (SF2) were placed in 4 mm increments, and Filtek Z350 (FZ350) was placed in 2 mm increments. They were light-cured according to manufacturer recommendations. The KHN of top and bottom surfaces were tested using Knoop Hardness tester at 10 gf/10 s. The DTS was tested under compressive load at 1.0 mm/min. The DC was measured by FTIR spectroscopy using attenuated total reflectance. Differences in DTS and DC were analyzed by ANOVA and Tukey post hoc test. For KHN, Kruskal-Wallis and Wilcoxon tests were performed at $\alpha = 0.05$. Top surfaces of all resins had higher KHN than bottom surfaces. At top and bottom surfaces, FZ350 showed higher KHN than TNC and SF2. The highest DTS was obtained by FBF, followed by FZ350 and SF2. The highest DC was obtained by SF2, the lowest one was obtained by FBF. The differences in the composition of the Bulk-Fill resins tested are reflected in the different results.

Key words: Composite resins. Hardness. Diametral tensile strength. Fourier transform infrared spectroscopy.

SUMÁRIO

1 INTRODUÇÃO

Atualmente os materiais restauradores odontológicos mais utilizados são as resinas compostas. Isso se deve principalmente às suas excelentes propriedades estéticas, módulo de elasticidade semelhante ao da dentina, biocompatibilidade e possibilidade de adesão aos dentes, o que resulta em preparações cavitárias menos invasivas (Ribeiro e Pazinatto, 2016).

Contudo, durante o processo de polimerização das resinas compostas, as conexões entre os monômeros são convertidas em ligações covalentes que possuem menor espaço dimensional, sofrendo uma contração volumétrica (Rueggeberg et al., 2017). Essa contração pode comprometer sua integridade marginal e, consequentemente, a longevidade clínica da restauração, tornando-a mais suscetível à microinfiltração e sensibilidade pós-operatória (Marí et al., 2019; Sampaio et al., 2019).

Dessa forma, visando diminuir os efeitos da contração volumétrica, tem sido recomendado colocar resinas compostas convencionais em incrementos de até 2 mm (Karacolak et al., 2018). No entanto, essa técnica bem conhecida é considerada altamente sensível e demorada (Pereira et al., 2018).

Assim, com o objetivo de reduzir os efeitos de contração do material, diminuir o tempo clínico e facilitar o manuseio do material, as resinas compostas Bulk-Fill foram desenvolvidas (Tauböck e Thomas, 2016; Vicenzi e Benetti, 2018; Bahbishi et al, 2020). Segundo os fabricantes, as resinas Bulk-Fill podem ser inseridas na cavidade e fotopolimerizados com até 4mm de material por incremento, sem comprometer suas propriedades físicas (Machado e Rodrigues, 2012).

Essa polimerização com maior quantidade de material é possível devido às modificações feitas nas composições das resinas Bulk-Fill. Dentre as modificações propostas, (1) o uso de materiais fluidos foi aplicado, com menor conteúdo de carga, que possibilitou melhor adaptação do material na cavidade; (2) diferentes tipos de partículas, para melhorar a transmissão de luz em camadas profundas; (3) uso de iniciadores mais eficientes; (4) melhorias no sistema monomérico que promete maior alívio de tensões durante a polimerização (Pfaifer, 2017). Exemplos comerciais de resinas Bulk-Fill que possuem algumas dessas tecnologias são: Filtek One Bulk-Fill (3M-Oral Care) e Tetric N-Ceram (Ivoclar-Vivadent).

Além desses exemplos comerciais existem outros como a SonicFill 2, que por ser bastante recente no mercado, requer mais investigações quanto à sua formulação. Segundo os fabricantes, essa resina "se baseia no premiado desempenho de sua antecessora" (Perfil Técnico SonicFill 2), a SonicFill. No entanto, os fabricantes não deixam claro quais alterações em sua composição foram feitas e, até onde sabemos, existem poucos estudos avaliando a SonicFill 2.

Diante da popularização e do aumento da comercialização das resinas Bulk-Fill se faz necessário avaliar a eficácia dessas alterações de composição nas propriedades mecânicas das restaurações. Para isso, alguns testes in vitro são comumente realizados para simular as forças que afetam o conjunto dente-restauração durante a mastigação, como por exemplo, a análise da microdureza que pode ser definida como a capacidade de um material de resistir à permanente deformação (Oliveira et al., 2015). Essa análise está relacionada indiretamente ao grau de conversão (Oliveira et al., 2015) que mede a conversão dos monômeros resinosos em um polímero resinoso rígido. Quando a reação acontece adequadamente, em média 60% dos monômeros se converte em polímeros, ocorre uma melhoria nas propriedades físicas das resinas, como uma maior resistência à fratura, ao desgaste e à compressão, possibilitando maior longevidade as restaurações resinosas (Machado e Rodrigues, 2012).

Assim como a microdureza, que avalia a resposta dos materiais restauradores frente às forças que incidem sobre o dente e a restauração durante o ato da mastigação in vitro, a análise da resistência à tração diametral também pode ser realizada, que mostra a capacidade da resina composta em suportar cargas de compressão sem fraturar (Marques et al., 2014).

Diante disso, o objetivo deste estudo foi avaliar e comparar o grau de conversão (DC), microdureza (KHN) e resistência à tração diametral (DTS) de 3 resinas Bulk-Fill comerciais (SonicFill 2, Filtek Bulk-Fill, Tetric N- Ceram) e 1 convencional (Filtek Z350). A hipótese nula testada é de que não haveria diferença entre elas.

ARTIGO:

Comparative evaluation of Bulk-Fill resins: Knoop microhardness, diametral tensile strength and degree of conversion

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ABSTRACT

Objective: Bulk-fill resins were developed to reduce time and facilitate the restorative procedure. However, considering their recent introduction on the market and the new formulations, their performance still requires evaluation. This study aimed to evaluate Knoop microhardness (KHN), diametral tensile strength (DTS) and degree of conversion (DC) of three Bulk-Fill resins and a conventional one. **Materials and Methods:** Sixty samples (n=15; 8 mm ø x 4 mm height) were confectioned using a mold. Filtek Bulk-Fill (FBF), Tetric N-Ceram Bulk-Fill (TNC) and SonicFill 2 (SF2) were placed in 4 mm increments, and Filtek Z350 (FZ350) was placed in 2 mm increments. The KHN of top and bottom surfaces were tested using Knoop Hardness tester at 10 gf/10 s. The DTS was tested under compressive load at 1.0 mm/min. The DC was measured by Fourier Transform Infrared (FTIR) spectroscopy. Differences in DTS and DC were analyzed by ANOVA and Tukey *post hoc* test. For KHN, Kruskal-Wallis and Wilcoxon tests were performed at $\alpha = 0.05$. **Results:** Top surfaces of all resins had higher KHN than bottom surfaces. At top and bottom surfaces, FZ350 showed higher KHN than TNC and SF2. The highest DTS was obtained by FBF, followed by FZ350 and SF2. The highest DC was obtained by SF2, the lowest one was obtained by FBF. **Conclusions:** From Bulk-Fill resins, FBF presented the best KHN and DTS results. The SF2 showed the best DC. Further studies are required to ensure whether these differences can negatively influence the behavior of in vivo restorations.

Keywords: Composite Resins, Bulk-Fill; FTIR; Hardness; Tensile Strength.

INTRODUCTION

Composite resins are the most currently used restorative material mainly due to their excellent aesthetic properties and possibility of adhesion to teeth, which results in less invasive cavity preparations [1]. When light-activation is performed, this material suffers a volumetric shrinkage that can compromise the marginal integrity of the restoration and consequently its clinical longevity [2,3]. For this reason, it has been recommended to place conventional composite resins in increments of 2 mm to reduce the adverse effects of the polymerization stress [4]. However, this well-known technique is considered highly sensitive and time-consuming [5]. Thereby, to reduce the conventional composite shrinkage stress, decrease clinical time and facilitate material handling, the Bulk-Fill composite resins were developed [1, 6].

According to manufacturers, they can be light-cured with up to 5mm of material in the cavity, without compromise of their physical properties. This polymerization with a greater amount of material is possible due to the changes made in the compositions of the Bulk-Fill resins. Among the proposed modifications, (1) the use of fluid materials was applied, with lower load content (2) as well as different types of particles, to improve light transmission in deep layers, (3) the use of more efficient initiators, (4) and improvements in the monomer system that promises greater stress relief during polymerization [7]. Commercial examples of Bulk-Fill resins that possess some of these technologies are: Filtek One Bulk-Fill (3M-Oral Care) and Tetric N-Ceram (Ivoclar-Vivadent).

Systematic review and meta-analyzes [8, 9, 10] have shown that Bulk-Fill composite resins have better or similar performance to the conventional resins. However, their introduction on the market is recent, and new formulations have been developed that requires further investigation, such as the SonicFill 2 (Kavo-Kerr). According to the manufactures, this resin "is based on the award-winning performance of its predecessor" (Technical Profile SonicFill 2), the SonicFill. Nevertheless, the manufactures do not make clear what changes in its composition were made, and to the best of our knowledge, there are few studies evaluating the SonicFill 2.

In view of the popularization and increased commercialization of Bulk-Fill resins, it is necessary to evaluate their performance in both clinical and laboratorial studies. Among *in vitro* tests commonly used to simulate the forces that affect the tooth-restoration complex during the chewing, the microhardness test is one of the most used because it can predict whether the restorative material will resist wear [11]. This analysis is indirectly related to the degree of conversion and studies have shown that both tests show results with lower values according to depth, that is, with increments of composite resin in greater thickness [12,13], as occurs in Bulk-Fill resins. The diametral tensile strength test, on the other hand, shows the capacity of the composite resin to withstand compression loads, such as, for example, occlusal loads that generate tensile stresses without fracturing [14].

Thus, the objective of this study was to evaluate and compare the degree of conversion (DC), microhardness (KHN) and diametral tensile strength (DTS) of 3 commercial Bulk-Fill resins (SonicFill 2, Filtek Bulk-Fill, Tetric N-Ceram) and 1 conventional one (Filtek Z350). The null hypothesis tested is that there would be no difference between them.

MATERIALS AND METHODS

For this study, sixty samples (n=15) of composite resin were made. The conventional composite Filtek Z350 (FZ350) was considered as the control group, while Bulk-Fill composites, such as Filtek Bulk-Fill (FBF), Tetric N-Ceram Bulk-Fill (TNC) and SonicFill 2 (SF2), as the experimental ones. The materials tested and their composition are described on Table 1.

Table 1. Materials used, their composition and manufacturers.

COMPOSITE RESINS	MANUFACTURERS	COMPOSITION
Filtek Z350	3M Oral Care, St. Paul, MN, USA (Lot number: 1930300193 / Exp: 10-02-2022)	Organic content: Bis-GMA, UDMA, TEGDMA, PEGDMA and Bis-EMA. Inorganic content: 20 nm non-agglomerated silica nanoparticles, 4 to 11 nm non-agglomerated zirconia nanoparticles, and combined zirconia (4 to 11 nm) and aggregated silica (20 nm) charge. The average size of the clusters varies from 0.6 to 1 µm. 78.5% by weight and 63.3% by volume.
Filtek One Bulk-Fill	3M Oral Care, St. Paul, MN, USA (Lot number: 1829200459 / Exp: 07-01-2021)	Organic content: AFM (dynamic stress-relieving monomer), AUDMA, UDMA and 1, 12-dodecane-DMA. Inorganic content: non-agglomerated/non-aggregated 20 nm silica filler, a non-agglomerated/non-aggregated 4 to 11 nm zirconia filler, an aggregated zirconia/silica cluster filler (comprised of 20nm silica and 4 to 11 nm zirconia particles) and ytterbium trifluoride filler consisting of agglomerate 100 nm particles: 76.5% by weight and 58.5% by volume.

Tetric N-Ceram Bulk-Fill	Ivoclar-Vivadent, Schaan, Lichtenstein (Lot number: YA71111 / Exp: 11-10-2023)	The monomeric matrix is composed of dimethacrylates (20 to 21% by weight). The fillers contain barium glass, ytterbium trifluoride, mixed oxide and copolymers (79-81% by weight). Additives, initiators, stabilizers and pigments are additional ingredients (<1.0% by weight). The total content of inorganic fillers is 76–77% by weight or 53–54% by volume. The particle size of inorganic fillers varies between 40 nm and 3 µm.
SonicFill 2	Kavo-Kerr, Orange, CA, USA (Lot number: 71123323 / Exp: 03-12-2021)	Bis-GMA, TEGDMA, EBPADMA, SiO2, glasses and oxides: 83.5% by weight and 67% by volume.

Samples preparation:

For samples preparation, a silicon mold of 8 mm of internal diameter and 4mm of height was used. The materials were inserted into the mold using two methods, according to the type of composite: for the conventional composite, by increments of 2 mm following the incremental technique, and for Bulk-Fill resin composites, in single increments of 4 mm thick. A handpiece provided by the manufacture was used to insert the SF2, which according to them, sonically activates the material and decreases its viscosity at the insertion time. All the materials were tested with the A1 shade. Each increment was light cured according to the time recommended by the manufacturer for each resin (20 s for FBF, SF2 and FZ350, and 10 s for TNC), using a 3rd generation LED (VALO Ultradent Products Inc., South Jordan, UT, USA) in its standard output mode. The distance of the tip device to the mold surface was standardized to approximately 1 mm with the aid of a holder. After filling the matrix, a polyester strip was placed over the resin with light pressure, and a glass plate was placed over the strip for 10 seconds to obtain a flat and even surface prior the light curing. The power of the device (1.000 ± 9.2 mW/cm^2) was verified during all experiment with a potentiometer. Then, the samples were removed from the mold and dry stored at 37 ± 1 ° C for 24 h in a light shelter.

The top and bottom of all samples were then polished by the same operator using 1200 and 4000-grit silicon carbide (SiC) abrasive papers (CarbiMet Abrasive Discs, Buehler, Lake Bluff, IL). After polishing with each grit, the samples were cleaned with distilled water in an ultrasonic vat (Sonica, SoltecS.il Co., Milano, Italy) for 5 minutes to remove any debris on the surface. The schematic representation of samples preparation is shown on Figure 1.

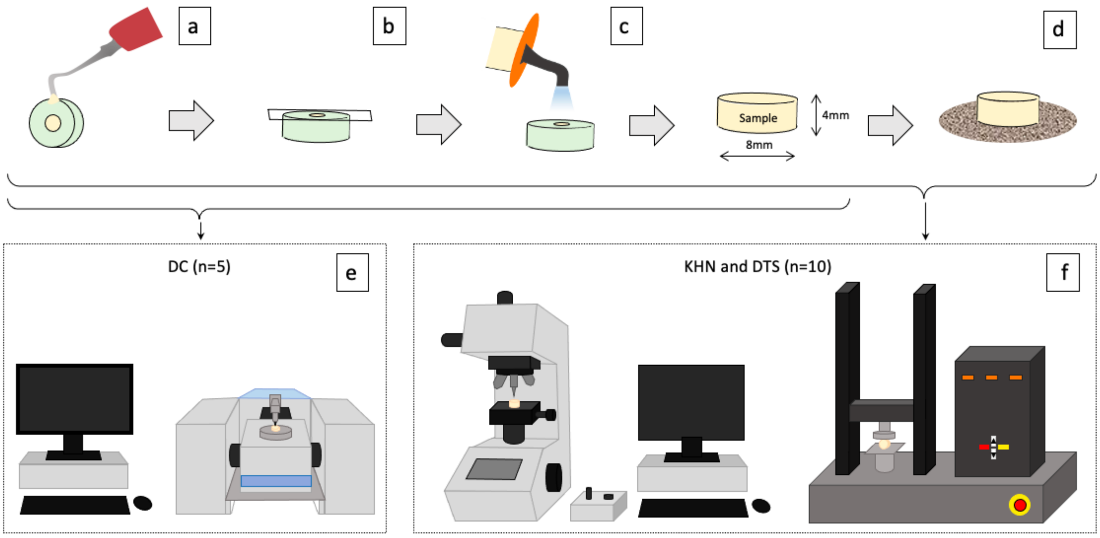

Figure 1. Samples preparation and their respective analysis. **a:** Insertion of the composite in the silicon mold. **b:** Flattening the composite with polyester strip and glass plate for 10 s. **c:** Sample light curing. **d:** Polishing with 1200 and 400-grit silicon carbide (SiC) abrasive papers. **e:** Degree of conversion test. **f:** Knoop microhardness and diametral tensile strength tests.

Surface Microhardness:

The microhardness readings (n=10) were performed on the top and bottom surfaces of each sample using the Future Tech FM-ARS surface microhardness machine (Future-Tech Corp., Tokyo, Japan) with a Knoop diamond indenter under a 10 g for 10 s. Five measurements were made on the surface of each sample by the same operator, one at the sample center and the other four at a distance of ~100 µm from the central location. The average of these five measurements was considered as the final Knoop microhardness (KHN) value.

Diametral Tensile Strength:

For the DTS test, the same samples (n = 10) of microhardness were used. A digital caliper (Mitutoyo, Tokyo, Japan) was used to measure the samples dimensions and ensure that the diameter was twice its height, as recommended by ADA No. 27 for this analysis [15]. After this verification, each sample was positioned on its side between the parallel plates of the Universal Mechanical Testing Machine (Instron, 4411, Canton, MA, England). A continuously load cell of 5.000 N was applied at a crosshead speed of 1 mm/min until fracture. The data obtained were then applied to the formula: DTS = 2F/πdh, where DTS = diametral tensile strength value (MPa); F = applied force to failure (N); d = diameter of the sample (mm); h = height of the sample (mm).

Degree of conversion:

The DC of bottom surfaces (n=5) was obtained by Fourier Transformed Infrared Spectroscopy (FTIR – Spectrum 100 FTIR/ATR; Perkin Elmer, Waltham, MA, USA). All spectra were obtained in of 650 - 4000 cm^{-1} with 32 scans and 4 cm^{-1} resolutions. Baseline correction was performed in region between 1590 to 1660 cm^{-1}. The percentage of unreacted carbon–carbon double bonds (C=C) were determined from the ratio of absorbance intensities of aliphatic vinyl bonds of methacrylate C=C (peak at 1638 cm^{-1}) against the internal standard (aromatic bisphenol C=C, peak at 1608 cm^{-1}) for non-polymerized and polymerized resin composite. The non-polymerized resin composite was used as a reference. The DC was determined by the following formula: DC (%) = $[1 – (R^{polymerized}/R^{non\text{-}polymerized})] \times 100$, where R = ratio by band height at 1638 cm^{-1}/band height at 1608 cm^{-1}.

Statistical analyses:

After exploratory and descriptive analyzes, the data were submitted to statistical tests. The DTS and DC data were submitted to One-way ANOVA and Tukey's test. For KHN data were performed the non-parametric Kruskal-Wallis's test for the comparison between groups, and Wilcoxon, for the comparison between the analyzed surfaces (top and base). All the statistical analyzes were performed in SPSS 25 Software (IBM SPSS Statistics 25 Corp) at the significant level of 0.05.

RESULTS

The statistical analyzes for the DTS and DC revealed that there was a significant difference between the groups, and for the KHN values it was possible to identify that there was a difference between the groups and between the analyzed surfaces (top and bottom).

The results for KHN demonstrated that the values of the top surface of the samples were statistically higher when compared to the values of the bottom surface, for all groups. In addition, FZ350 group showed higher KHN values than TNC and SF2 in both top and bottom surfaces. The FBF group, in turn, was statistically similar to all groups (Table 2).

Table 2. Median (minimum-maximum) of KHN values in different groups and different surfaces (top and bottom).

Groups *	**Surface**	
	Top	**Bottom**
FBF	87.205 (81.73 – 89.92) **ABa**	63.98 (53.75 - 67.49) **ABb**
TNC	82.21 (73.97 - 89.47) **Ba**	61.11 (55.06 - 64.32) **Bb**
SF2	79.685 (71.85 - 87.11) **Ba**	56.455 (52.81 - 68.58) **Bb**
FZ350	99.895 (88.60 - 104.01) **Aa**	91.845 (79.22 - 97.51) **Ab**

Different letters indicate statistical difference. Uppercase letters compare the groups in the same surface (in the column) and lowercase compare the surfaces in the same material (in the row) ($p \leq 0.05$).

***FBF:** Filtek Bulk-Fill; **TNC:** Tetric N-Ceram Bulk-Fill; **SF2:** SonicFill 2; **FZ350:** Filtek Z350.

The results for DTS showed that the FBF group presented higher values of resistance to diametral traction when compared to the other groups. On the other hand, the FZ350 group showed statistically higher values when compared to the SF2 group. The TNC group, on the other hand, presented statistically similar values both to the FZ350 group and to the SF2 group (Table 3).

Table 3. Means (standard deviation) of the DTS in the different groups.

Groups	DTS
FBF	56.736(3.311) **A**
TNC	42.319(5.935) **BC**
SF2	38.275(3.690) **C**
FZ350	46.611(4.630) **B**

Means followed by different letters indicate a statistically significant difference ($p \leq 0.05$).
***FBF:** Filtek Bulk-Fill; **TNC:** Tetric N-Ceram Bulk-Fill; **SF2:** SonicFill 2; **FZ350:** Filtek Z350.

The results for DC, in turn, demonstrated that the SF2 group showed the highest values, being statistically superior to all other groups. In contrast, the FBF group showed the lowest DC, being statistically lower than all other groups. The TNC and FZ350 groups were statistically similar to each other (Table 4).

Table 4. Means (standard deviation) of the DC in the different groups.

Groups	DC
FBF	36.596 (6.61) **C**
TNC	47.501 (3.07) **B**
SF2	63.667 (2.31) **A**
FZ350	53.844 (1.301) **B**

Means followed by different letters indicate a statistically significant difference ($p \leq 0.05$).
***FBF:** Filtek Bulk-Fill; **TNC:** Tetric N-Ceram Bulk-Fill; **SF2:** SonicFill 2; **FZ350:** Filtek Z350.

DISCUSSION

The decision of which composite resin to use during a certain restorative procedure must consider its mechanical properties, in order to obtain an increase in the longevity of the restoration [16, 17]. In this context, the evaluation of a material properties *in vitro* becomes essential because it simulate the ability of this material to resist the stress suffered in the oral environment without fracturing or suffering wear [4, 14]. In this study, the microhardness (top and bottom), the degree of conversion (base), and the diametrical tensile strength of 3 commercial Bulk-Fill resins compared to 1 conventional resin were evaluated. According to the results, the null hypothesis tested that there would be no difference between the properties of composite resins was rejected.

As the results for KHN showed, the values of the top surface of all groups were statistically higher than the values of the bottom surface. Regardless of the composite resin evaluated, the irradiance that reaches the bottom surfaces is affected for both the increase of distance to the guide tip of the light-curing unit and light scattering by the filler particles and resinous matrix [18]. It is well understood that the irradiance is inversely proportional to the distance of the light-curing unit guide tip [19] and that light penetrates the composite layer and is partially absorbed, partially scattered by fillers and other constituents [20, 21], respectively.

The conventional composite FZ350 specifically showed the highest values of KHN both at the top and at the bottom surfaces, when compared to the other composites. Its composition comprises nano-agglomerates of fillers (nanoclusters), which due to its characteristics, allow a better distribution and a larger amount of particles in the resin matrix, improving its mechanical properties [22]. Furthermore, the high hardness, characteristic of the zirconia particles incorporated in the composition of many 3M composites [5; 23], may also have contributed to this outcome. Bahbishi et al (2020) evaluated color stability and microhardness of Bulk-Fill composites, and also found that all of them had lower microhardness values when compared to FZ350 [1]. One should also consider that, unlike Bulk-Fill composites, FZ350 was built-up in two increments of 2 mm. As already mentioned, the penetration of light through an increment during curing is a complex phenomenon dependent on many factors, such as type and size of filler, light source, as well as the difference of refractive index between fillers and monomers [20, 24]. With such number of factors in the path, it is likely that light penetration decreases as the thickness of composite increases [20, 25]. Thus, the microhardness result presented by FZ350 is also possibly due to the fact that its increment was smaller than the increments of the Bulk-Fill composites.

Such features may further justify the DTS values of FZ350, which were higher than SF2 ($p=0,0000$), and the DC ones, which were higher than FBF ($p=0,000$). The smaller increment might have led to improve of DC and consequently increase of mechanical strength (DTS). Yet, possible void incorporation during the filling through incremental technique explains the reason why FZ350 presented lower DTS than FBF [26]. During DTS test the voids may have acted as areas of sample fracture origin; unlike FBF, which despite having a constitution of filler particles largely similar to FZ350, once it was inserted in single increment, presented higher values of DTS.

The FBF is also manufactured by 3M and as previously mentioned, is composed of nanoparticles of fillers (nanoclusters), including zirconia ones, which even in lower amount (58.4 vol%) when compared to FZ350 (63.3 vol%), might have played a role in this composite performance [27]. This composition characteristic, added to the fact that it is a single fill composite, which practically nullifies the chances of incorporating bubbles during the insertion of the material in the matrix cavity, may justify why this resin presented the highest values for DTS, as well as top and bottom KHN values similar to those of the FZ350. Similarly, Pereira et al (2019) reported similar performance of FBF and FZ350 [21]. However, despite what we expected for the results of the DC, this composite showed the lowest values. This finding can be understood by the nature of its organic matrix. Among the modifications proposed by the manufacturer, different monomers were developed and incorporated into its composition, such as the high molecular weight aromatic dimethacrylate (AUDMA), capable of increasing the rigidity of the polymer chain formed [28]. On the one hand, a more rigid chain increases the resistance of the material [29], which may also have contributed to the higher values of DTS. On the other hand, it decreases the number of reactive groups on the organic matrix and hinders their mobility during the polymerization reaction. Thus, one can understand a low DC value for this composite at the bottom of the samples, as also demonstrated by Habib and Waly (2018) [30].

However, not necessarily lower values of DC at the bottom of the samples imply unacceptable clinical performance [31], since it has been shown that more important than the amount of conversion of carbon double bonds into single bonds, is the quality of these bonds, that is, the degree of cross-linking [23]; and this cannot be measured by FTIR, since this is a quantitative analysis. The degree of crosslinking of this composite, in turn, can be compensated by other monomers incorporated in its composition: the additional fragmentation monomer (AFM), which can cleave, relaxing the polymerization stresses that are generated during the formation of the chain, and still react by forming other cross bonds due to the free radical generated by the break [28]; and the UDMA monomer, which has the presence of imino groups (-NH-) in its structure, responsible for the characteristic chain transfer reactions, which provide an alternative path for the polymerization [5].

As observed in the results, the TNC and SF2 composites showed the lowest values of both KHN and DTS tests. The TNC composite has prepolymer particles (isofillers) in its composition, which, due to its resinous nature, presents lower modulus of elasticity. This lower modulus of elasticity causes worsening on stress distribution, which makes the material more susceptible to deformations [31]. This fact could explain the lower KHN and DTS values of TNC [13, 33]. Also, this composite showed DC values below those of SF2 ($p = 0.0000$). According to the manufacturers, besides camphorquinone, an alternative photoinitiator known as Ivocerin was added to this composite to provide better light-curing and consequently better

DC. However, as inferred by the present study and proved by previous ones [13, 33], this additional photoinitiator did not provide higher DC for this composite when compared to others. This may be associated with low penetration in the resinous matrix of the violet light (between 380 and 450nm), capable of activating the Ivocerin, for two different reasons: the first, due to the short wavelength of this light, that restricts the action of the additional photoinitiator only to the most superficial layers of the composite [33]; and second, due to the composite filler content. Although from all composites tested TNC presents the lowest amount of fillers, the size of particle reaches up to 3 µm. As well elucidated by Emami et al (2005), the higher the particle size, the higher the chance of absorption changes, so more likely to occur light scattering [20].

The SF2 composite, on the other hand, is a reformulation of its predecessor SonicFill. According to the manufacturer, the modifications of its composition have improved the composite performance. However, to the best of our knowledge, few in vitro studies [1, 34, 35, 36] and no in vivo studies have been found so far evaluating SF2 properties. Yet, one may note that the composite line-up remained presenting high filler content (81.3 wt%). If, on the one hand, it can be inferred that the high filler content could improve the composite's mechanical properties, on the other hand such feature, added to the composite's low translucency, already pointed out by its predecessor, could also hinder the transmission of light through the material and result in inefficient DC, especially in deeper layers [13, 21, 30]. Conversely, our study showed the lowest KHN and DTS, but the highest DC values for SF2. Indeed, the reason for the SF2 KHN and DTS results may lie in the composite sonic activation feature and monomer content. The use of sonic activation to place the material into the cavity, as recommended by the manufacturer, may in fact result in voids development [1]. This happens because when the composite portion that comes out of the tip, more flowable, reaches the portion that has already been inserted in the cavity, more dense, the difference in consistency between them prevents a perfect adaptation of the increment. Hence, the chance of cracks development and bulk fracture is increased, as already demonstrated in another study [37].

Regarding its monomer content, SF2 contains TEGDMA. Although its exact quantity is not informed by the manufacturer, a previous study highlighted the elution of TEGDMA from SF2 in comparison to other Bulk-Fill composites [36], indicating that the monomer content is not low. TEGDMA have two functional terminal methacrylate groups similar to Bis-GMA. The difference is that there is a linear chain between the groups, decreasing its viscosity [38]. The lesser viscosity may reduce the composite's mechanical properties [38], which could further explain our KHN and DTS findings. Nonetheless, the low viscosity of this monomer favors the movement of reactive species responsible for both initiation and propagation of the polymerization reaction, yielding higher conversion degree values [34]. The temperature generated by sonic activation might also have played a role in the DC values obtained by SF2. As suggested by Garoushi et al (2016), one may speculate that the increase of temperature during the material application through activation by sonic energy can increase the composite's DC in two ways: directly, by increasing the mobility of free radicals during the formation of polymer chain, and indirectly, by reducing the composite's viscosity [13].

As noted, the differences in composition of the Bulk-Fill composites tested influenced in different physical and mechanical behaviors among them. In this in vitro study, properties were evaluated separately. However, the behavior in function of a restoration and its longevity will depend, as far as the material is concerned, on a sum between the characteristics of the resin matrix and that of the filler particles [39]. Thus, the importance of in vitro studies consists of the first level of evidence to assess the quality of new commercially available materials. However, only controlled and randomized clinical studies will confirm its effectiveness and permanence in the market.

CONCLUSIONS

Differences in the composition of the evaluated Bulk-Fill resins resulted in different results for the in vitro analyzes performed. From Bulk-Fill resins, FBF presented the best KHN and DTS results. The SF2 showed the best DC. However, it remains need to know whether these differences can negatively influence the behavior of in vivo restorations. Accordingly, more clinical studies should be carried out in long term.

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3 CONCLUSÃO

As diferenças na composição das resinas Bulk-Fill avaliadas influenciaram nos diferentes resultados para as análises *in vitro* realizadas. No entanto, ainda é necessário saber se essas diferenças podem influenciar negativamente no comportamento das restaurações *in vivo*. Assim, mais estudos clínicos devem ser realizados em longo prazo.

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APÊNDICE – Delineamento experimental e metodologia ilustrada

MATERIAIS UTILIZADOS

• Três resinas do tipo Bulk-fill e uma convencional (Tabela e Figura 1)

• Matriz de silicone (8mm de diâmetro e 4mm de altura)

• Espátula de inserção (Espátula Suprafill SSWhite Duflex–Juiz de Fora, MG)

• Tira de poliéster

• Fonte de luz LED (VALO, Ultradent Products Inc; S. Jordan, UT, USA – 1400mW/cm2)

• Politriz giratória (Modelo APL-4; Arotec, SP, Brasil)

• Discos de lixa de carbeto de silício nas granulações #1200 e #4000 (CARBIMET Paper Discs; Buehler, IL, EUA)

• Cola termoplástica (Brascola, São Bernardo do Campo, SP, Brasil).

• Papel absorvente (Kleenex – Kimberly – Clark, São Paulo, SP, Brasil)

• Cuba ultrassônica (Ultra Clearer USC-1450/ Frequência 25 kHz, UNIQUEBrasil)

Tabela 1. Grupos experimentais (n=15)

Grupo	Nome do compósito e fabricante
FZ350	Filtek Z350 - 3M ESPE, St.Paul, MN, EUA
FBF	Filtek One Bulk-Fill - 3M ESPE, St.Paul, MN, EUA
TNC	Tetric N-Ceram Bulk-Fill - Ivoclar-Vivadent
SF2	SonicFill 2 - Kavo-Kerr

GRUPOS EXPERIMENTAIS

60 amostras de resina composta foram confeccionadas (n=15). A resina composta convencional Filtek Z350 (FZ350), foi considerada como grupo controle, enquanto as resinas do tipo Bulk-Fill, como a Filtek Bulk-Fill (FBF), Tetric N-Ceram Bulk-Fill (TNC) e SonicFill 2 (SF2), como grupos experimentais (Figura 1).

CONFECÇÃO DAS AMOSTRAS

Para a confecção das 60 amostras (n=15) foi utilizada uma matriz de 8mm de diâmetro interno e 4mm de altura, seguindo recomendação da ADA nº 27 que preconiza que o diâmetro deve possuir o dobro da altura da amostra (American Dental Association 1977) para a análise da tração diametral (Figura 2). A cavidade da matriz foi preenchida em incremento único para as amostras em resina do tipo Bulk-Fill e pela técnica incremental para as amostras em resina do tipo Convencional. Após a colocação do material no interior da matriz, foi posicionada sobre a resina uma tira de poliéster com leve pressão, e sobre a tira foi colocado uma placa de vidro por 10 segundos para se obter uma superfície plana e regular (Figura 3). Após a remoção da placa, o material foi fotoativado com auxílio de um anteparo para padronizar a distância de aproximadamente 1mm da ponta do aparelho à superfície da amostra, com LED de 3ª geração (VALO Ultradent Products Inc., South Jordan, UT, EUA) com intensidade de luz de 1000mW/cm^2, sendo a potência do mesmo verificada com auxílio de um potenciômetro (Figura 4). As amostras foram fotopolimerizadas de acordo com o tempo recomendado pelo fabricante para cada resina, sendo 20 segundos para a Filtek Bulk-Fill, para a SonicFill 2 e para a Filtek Z350 e 10 segundos para a Tetric N-Ceram. Para obtenção de amostras com superfície homogênea, o topo e a base de todas as amostras foram polidas pelo mesmo operador utilizando lixas dágua de granulação #1200 e #4000 (Modelo APL-4; Arotec, SP, Brasil) (Figura 5). Após o polimento, as amostras foram limpas com água destilada em cuba ultrassônica (Sonica, SoltecS.i.l. Co., Milano, Itália) por 5 minutos para remover quaisquer detritos que estivessem na superfície. Após o polimento das amostras, as amostras ficaram armazenadas a seco por 24hrs a 37±1° C, em abrigo de luz, antes de iniciar as análises.

Figura 1. Compósitos utilizados

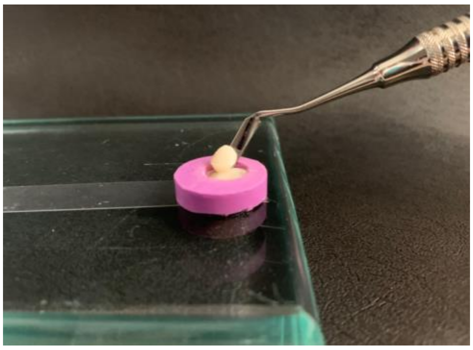

Figura 2. Inserção do material na cavidade da matriz

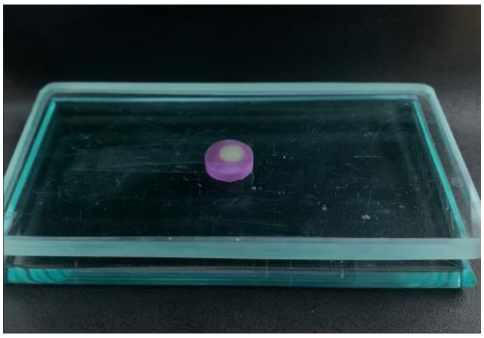

Figura 3. Planificação da superfície da amostra

Figura 4. Fotopolimerização das amostras

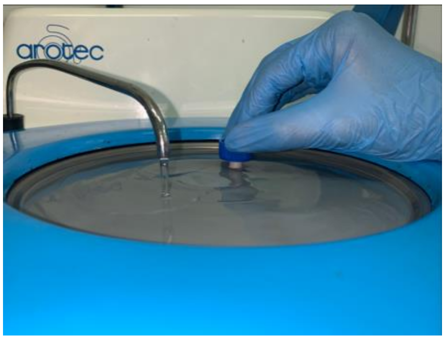

Figura 5. Polimento das amostras em politriz sob irrigação constante

Figura 6. Checagem das dimensões da amostra com especímetro

MICRODUREZA SUPERFICIAL

O valor de Dureza Knoop (HK) de cada amostra foi avaliado usando a máquina de microdureza superficial Future Tech FM-ARS (Future-Tech Corp., Tóquio, Japão). Foi aplicado um peso de 10gf por 10 segundos em cinco áreas distintas de cada face das amostras (cinco medições no topo e cinco na base), e a média dessas cinco medidas foi considerada como o valor final de Knoop.

Entre as análises de microdureza e tração diametral, as amostras ficaram armazenadas a seco a 37±1° C, em abrigo de luz.

TRAÇÃO DIAMETRAL

Após análise de microdureza, as mesmas amostras (n=10) foram medidas quanto ao diâmetro e altura com o auxílio de um especímetro, garantindo que as dimensões continuassem na proporção estipulada pela ADA nº 27 (Figura 6). Após essa verificação, as amostras passaram pelo ensaio mecânico de resistência à tração diametral que foi realizado pela Máquina de Ensaios Mecânicos Universal (Instron, 4411, Canton, MA, Inglaterra), com célula de carga de 5.000N com velocidade de 1 mm/min. As amostras foram posicionadas de lado entre as placas paralelas da máquina de teste, sendo as mesmas carregadas continuamente em compressão a 1cm/minuto até o ponto de ruptura. Em seguida, os dados obtidos foram aplicados na fórmula: $R = 2L/\pi Dh$, em que R = Valor da tração diametral; L = Força aplicada (N); D = Diâmetro da amostra (mm); h = altura da amostra (mm).

Para a avaliação do grau de conversão, 28 amostras foram confeccionadas como descrito em "confecção das amostras" e analisadas pela Espectroscopia Infravermelha Transformada por Fourier (FTIR) antes (n=2) e após (n=5) a fotoativação durante 1 minuto. Para isso, as amostras foram fotopolimerizadas com LED de 3ª geração (VALO Ultradent Products Inc., South Jordan, UT, EUA) com intensidade de luz de $1000mW/cm^2$ de acordo com o tempo recomendado pelo fabricante para cada resina. O anteparo para padronizar a distância, de aproximadamente 1mm, da ponta do aparelho à superfície da amostra foi utilizado mais uma vez, de modo a reproduzir as mesmas condições em que as amostras para a microdureza e tração diametral foram confeccionadas. Contudo, apenas a superfície de base das amostras ficou em contato com o cristal, e, portanto, os resultados do grau de conversão são referentes apenas a essa superfície. Todos os espectros foram obtidos em uma faixa de 1800-1500 cm^{-1}, utilizando-se 12 scans a 4 cm^{-1} de resolução em modo de transmissão e 2.8mm/s de velocidade. A altura do pico de absorbância foi determinada após a subtração da linha de base e o processo de normalização, utilizando-se o software Origin. O grau de conversão foi calculado a partir das amostras polimerizadas e não polimerizadas pela seguinte equação:

$$GC_{fotopolimerizado} = 100 \times 1- \left[\frac{\left(alifático / aromático \right)_{fotopolimerizado}}{\left(alifático / aromático \right)_{não\ curado}} \right]$$

ANEXOS

Anexo 1 – Verificação de originalidade e prevenção de plágio

AVALIAÇÃO COMPARATIVA DE RESINAS BULK-FILL: MICRODUREZA KNOOP, RESISTÊNCIA À TRAÇÃO DIAMETRAL E GRAU DE CONVERSÃO

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Anexo 2 – Iniciação Científica

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Bolsista: Bruna Scarcello Strini– RA 194975

Orientador(a): Prof.(a) Dr.(a) FLÁVIO HENRIQUE BAGGIO AGUIAR

Projeto: "Influência do grau de conversão nas propriedades de microdureza e tração diametral de resinas do tipo Bulk-Fill."

Bolsa: PIBIC/CNPq
Vigência: 01/08/2019 a 31/08/2020
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PARECER

Conclusão do Parecer:
A aluna cumpriu com todas as etapas exigidas de uma Iniciação Científica, mostrando amadurecimento ao longo dessa trajetória. O bom rendimento acadêmico foi mantido durante a realização da IC.

Aprovado

Pró-Reitoria de Pesquisa, 14 de setembro de 2021.

Mirian Cristina Marcançola
PRP / PIBIC - Unicamp
Matr. 299062

148542/2019-0

Anexo 3 – Comprovante de submissão do Artigo

Sergio Eduardo de Paiva Gonçalves <bds.... 17:53 (há 1 hora)
para mim, Renata, Danielle, Vanessa, Priscila, Flávio

Hello,

Joyce Marques has submitted the manuscript, "Comparative evaluation of Bulk-Fill resins: Knoop microhardness, diametral tensile strength and degree of conversion" to Brazilian Dental Science.

If you have any questions, please contact me. Thank you for considering this journal as a venue for your work.

Sergio Eduardo de Paiva Gonçalves
_ Brazilian Dental Science Institute of Science and Technolgy of São José dos Campos Universidade Estadual Paulista - UNESP http://ojs.ict.unesp.br/ ATENÇÃO: Mensagem de envio automático. Por favor, não responda.