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Physical-mechanical properties of a flowable nanofiber-reinforced resin composite

Propriedades físico-mecânicas de uma resina composta fluida reforçada com nanofibras

Propiedades físico-mecánicas de un compuesto de resina fluida reforzado con nanofibras

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Abstract
This *in vitro* study aimed to evaluate the effect of toothbrushing simulated test on wear and roughness of different low-viscosity resin composite, as well as the polymerization shrinkage stress. Thirty rectangular specimens (5 × 10 × 3 mm) were prepared and assigned into three different low-viscosity resin composites (n=10): Filtek flow Z350 (Z350); NanovaPro fill (Nanova), and SureFil SDR Flow (SDR). The specimens were brushed for 100.000 cycles using a toothbrushing testing machine with soft bristle tips (Colgate Classic, Colgate-Palmolive Co., Osasco, Sao Paulo, Brazil) and dentifrice suspension (Colgate MFP, Colgate-Palmolive Co.) in deionized water under a 300 g load. The surface roughness (Ra) (n=10) (before and after brushing) and wear (μm) (n=10) were measured by roughness tester. Also, the microhardness (KHN) (n=5) and shrinkage stress (MPa) (n=5) were evaluated. Data were analyzed by one-way for wear, microhardness and shrinkage stress data and two-way for roughness ANOVA and Tukey test (α = 0.05). The Nanova group presented higher final roughness (1.79±0.36) ($p < 0.031$), wear (13.87±3.26) ($p < 0.001$) and microhardness (52.56±1.7) than the other groups ($p < 0.006$). For tensile test, all materials showed no difference in relation to shrinkage stress (p= 0.468). The Nanova group showed higher wear and roughness than the other groups. SDR and Z350 were statistically more resistant to wear.
Keywords: Composite resins; Toothbrushing; Nanofibers.

Resumo
Este estudo *in vitro* teve como objetivo avaliar o efeito do teste de escovação simulada no desgaste e rugosidade de diferentes resinas compostas de baixa viscosidade, bem como a tensão de contração de polimerização. Trinta espécimes retangulares (5 × 10 × 3 mm) foram preparados e divididos em três diferentes resinas compostas de baixa viscosidade (n=10): Filtek flow Z350 (Z350); NanovaPro fill (Nanova) e SureFil SDR Flow (SDR). Os espécimes foram escovados por 100.000 ciclos usando uma máquina de teste de escovação com pontas de cerdas macias (Colgate Classic, Colgate-Palmolive Co., Osasco, São Paulo, Brasil) e suspensão de dentifrício (Colgate MFP, Colgate-Palmolive Co.) em água deionizada sob uma carga de 300 g. A rugosidade superficial (Ra) (n=10) (antes e após a escovação) e o desgaste (μm) (n=10) foram medidos por rugosímetro. Também foram avaliadas a microdureza (KHN) (n=5) e a tensão de contração (MPa) (n=5). Os dados foram analisados por one-way para dados de desgaste, microdureza e tensão de retração e two-way para rugosidade ANOVA e teste de Tukey (α = 0,05). O grupo Nanova apresentou maior rugosidade final (1,79□0,36) (p < 0,031), desgaste (13,87±3,26) (p <0,001) e microdureza

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(52,56±1,7) que os outros grupos (p < 0,006). Para o ensaio de tração, todos os materiais não apresentaram diferença em relação à tensão de retração (p= 0,468). O grupo Nanova apresentou maior desgaste e rugosidade do que os outros grupos. SDR e Z350 foram estatisticamente mais resistentes ao desgaste.
Palavras-chave: Resinas compostas; Escovação dentária; Nanofibras.

Resumen

Este estudio *in vitro* tuvo como objetivo evaluar el efecto de la prueba simulada de cepillado de dientes sobre el desgaste y la rugosidad de diferentes compuestos de resina de baja viscosidad, así como la tensión de contracción de polimerización. Se prepararon treinta muestras rectangulares (5 × 10 × 3 mm) y se asignaron a tres compuestos de resina de baja viscosidad diferentes (n=10): Filtek flow Z350 (Z350); Relleno NanovaPro (Nanova) y SureFil SDR Flow (SDR). Los especímenes fueron cepillados durante 100.000 ciclos usando una máquina de prueba de cepillado de dientes con puntas de cerdas suaves (Colgate Classic, Colgate-Palmolive Co., Osasco, Sao Paulo, Brasil) y suspensión dentífrica (Colgate MFP, Colgate-Palmolive Co.) en agua desionizada bajo una carga de 300 g. La rugosidad de la superficie (Ra) (n=10) (antes y después del cepillado) y el desgaste (μm) (n=10) se midieron con un rugosímetro. Además, se evaluaron la microdureza (KHN) (n=5) y el estrés de contracción (MPa) (n=5). Los datos se analizaron mediante unidireccional para datos de desgaste, microdureza y tensión de contracción y bidireccional para rugosidad ANOVA y prueba de Tukey (α = 0,05). El grupo Nanova presentó mayor rugosidad final (1,79±0,36) (p < 0,031), desgaste (13,87±3,26) (p<0,001) y microdureza (52,56□1,7) que los demás grupos (p < 0,006). Para el ensayo de tracción, todos los materiales no mostraron diferencia en relación al esfuerzo de contracción (p= 0,468). El grupo Nanova mostró mayor desgaste y rugosidad que los otros grupos. SDR y Z350 fueron estadísticamente más resistentes al desgaste.
Palabras clave: Resinas compuestas; Cepillado dental; Nanofibras.

1. Introduction

Dental caries remains a prevalent disease that affects adults and children, considered a significant oral health issue. As a dynamic process of demineralisation and remineralisation episodes, caries occurs and progress due to an imbalance that favours demineralization (Pretty & Ellwood, 2013).

Despite the use of traditional restorative treatment is considered the conventional approach for cavidated carious lesions, the management of dental caries should be based on practices and procedures before its onset. Following the principles of a minimal intervention dentistry, the use of sealants is considered a primary prevention tool for individuals at high-risk of experiencing caries, providing a mechanical barrier that protect the tooth against the biofilm accumulation, cariogenic challenges and subsequent mineral loss (Beauchamp et al., 2008; Alkilzy et al., 2011; Ahovuo-Saloranta et al., 2017). In the case of cavitation lesions, less invasive approaches should be indicated, preserving the maximum of the tissues (Featherstone & Doméjean, 2012; Frencken, 2017).

Flowable resin composites have been recommended as pit and fissure sealants and minimally invasive restorations in clinical practice, because they present some desirable characteristics such as low viscosity, low modulus of elasticity and easy to dispense, which facilitates their use in small preparations that is difficult to fill otherwise (Baroudi & Rodrigues, 2015). Although such strategy is safe, there are concerns about gaps at the restorations-tooth interface, since traditional flowable resin composites present high polymerization shrinkage which could facilitate biofilm accumulation and marginal leakage (Hevinga et al., 2007; Desai et al., 2021).

In addition, once the filler loading of flowable resins is reduced compared to conventional composites (Baroudi & Rodrigues, 2015), they could not present adequate mechanical properties to be applied in stress-bearing areas. Whereas the restorative material is also frequently exposed to wear tension during daily tooth brushing procedures, appropriate mechanical properties with microfillers can probably enhance the overall wear resistance of flowable composites, without fracture, less roughness and biofilm accumulation, improving the long-term of restorations and protect them from recurrent caries over time (Svanberg et al., 1990; Baroudi & Rodrigues, 2015; Obeid et al., 2021).

With the introduction of new restorative biomaterials, the minimal intervention dentistry has become more favorable with increasingly predictable outcomes (Mackenzie & Banerjee, 2017). The introduction of bulk-fill flowable materials promoted easier clinical setting and claimed to present enhanced curing, shrinkage, mechanical, and wear properties. Despite all desirable characteristics, flowable bulk-fill resin composites present lower mechanical strength, being more useful in shallow cavities or needing a final covering with a higher filler load in more extensive cavities (Leprince et al., 2014; Van Ende et al., 2017; Lassila et al., 2019).

As a promising new class of restorative material, fiber-reinforced flowable resin composites have been introduced, enhancing the composite properties by acting mainly as crack stoppers (Khan et al., 2015). In general, during masticatory forces, stress is transmitted onto the filler because they are more rigid than organic matrix, which generate cracks around the filler particles leading to the displacement of the fillers and resulting in wear of the material (Oliveira et al., 2012; Tian et al., 2007). When resin composites are reinforced by nanofibers, since they are more ductile than inorganic fillers, present large ratio of surface area to volume and reduced size, there is an increased between the intermolecular bonding with the resin matrix, providing good load transfer between them and, consequently, resulting in higher strength (Tian et al., 2007; Papkov et al., 2013; Velo et al., 2019).

Indeed, several studies have reported promising results regarding the incorporation of nanofibers into resin-based composites (Wang et al., 2016; Salek et al., 2018; Velo et al; Obeid et al., 2022). However, most available literature focused on conventional resin composites and limited researches were performed using flowable bulk-fill composites. Ardestani et al. (2021) demonstrated that the incorporation of 0.5% wt. of silanized nanofibers into a low-viscosity bulk-fill resin improved the flexural strength and hardness of the material. Conversely, in the study of Yancey et al. (2019), a significantly lower flexural strength, modulus of elasticity and greater volumetric shrinkage were presented by nanofiber-reinforced restorative material over conventional hybrid composites. Nevertheless, there is insufficient data available regarding their polymerization shrinkage stress and resistance to wear in association to their mechanical properties.

Although several studies discussing minimal intervention or non-intervention are available, there is a lack of information regarding flowable fiber-reinforced resin composite to make credible conclusions for such purpose. Whereas the recommendation should be based in evidence and applying a flowable resin composite for minimal intervention restorations in occlusal surfaces will be submitted to masticatory forces and wear tension during daily tooth brushing procedures, the aim of this study was to evaluate the physical-mechanical properties of a nanofiber bulk-fill flowable resin composite compared to a conventional flowable resin composite and a bulk-fill flowable resin. The null hypotheses investigated were that: (1) the nanofibers bulk-fill flow resin composite would not present higher surface microhardness when compared to the control groups; (2) the nanofibers bulk-fill flow resin composite would not present higher surface wear and roughness when compared to the control groups and, (3) the nanofibers bulk-fill flow resin composite would not present higher polymerization shrinkage stress than control groups.

2. Methodology

2.1 Experimental design

In this *in vitro* study, the factors under study were three low-viscosity materials: conventional flowable resin composite (Z350- Filtek flow, 3M-ESPE, St. Paul, MN, EUA); nanofibers bulk-fill flow resin composite (Nanova - NanovaPro fill, Columbia, MO, USA flowable bulk-fill) and (SDR- SureFil SDR Flow, Dentsply International, York, PA, USA). The materials, manufacturer, composition, shade and classification used in this study are described in Table 1. The study was divided into three groups (n=10), according to the materials used: G1=Z350, G2=Nanova and G3=SDR. All tested resin composites were analyzed by toothbrushing abrasion, roughness and wear, microhardness and shrinkage stress.

Table 1 - Manufacturer, composition, shade and classification of the flowable resin composites tested.

Materials	Manufacturer	Composition*	Shade	Classification
Filtek flow Z350XT	3M-ESPE, St. Paul, MN, EUA	Bis-GMA, TEGDMA, and Procrylat K, yttrium fluoride: 0.1 to 5.0 µm, silica: 20 nm, zirconia: 4 to 11 nm, and zirconia/silica clusters of 0.6 to 10 µm	A2	Conventional flowable resin composite
NanovaPro fill	Nanova, Columbia, MO, USA	BAFSG, barium aluminofluorosilicate glass; Bis-EMA, bisphenol A ethoxy dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; amorphous; fumed silica, titanium dioxide, hydroxyapatite	A2	Nanofibers bulk-fill flow resin composite
SureFil SDR Flow	Dentsply International, York, PA, USA	Barium-alumino-fluoro-borosilicate glass, strontium alumino-fluoro- silicate glass, modified urethane dimethacrylate resin, ethoxylated bisphenol-A dimethacrylate (EBPADMA), triethyleneglycol dimethacrylate, camphorquinone, butylated hydroxyl toluene, uv stabilizer, titanium oxide,iron oxide pigments.	A2	Bulk-fill flowable

***** Information provided by the manufacturers. Source: Authors.

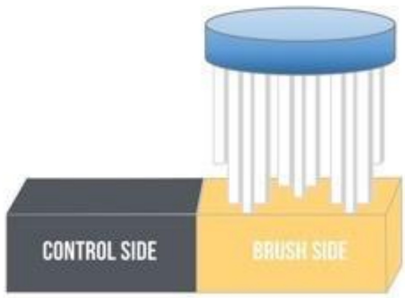
2.2 Sample preparation for toothbrushing abrasion evaluation, roughness and wear measurements

The flowable resin composites were applied to a rectangular stainless-steel mold ($10 \times 5 \times 3$ mm^3) and covered with polyester matrix strip (TDV Dental, Santa Catarina, Brazil), in order to maintain the smoothness of the material surface. All samples were polymerized with a high-power LED light-cure (Valo LED, Ultradent Products Inc, South Jordan, UT, USA) according to manufacturer's instructions, before and after removal of steel molds.

2.3 Toothbrushing abrasion

The surface of each sample (n=10) was divided into a control side and the brushed side (Figure 1). The samples were subjected to the toothbrushing test at a temperature of 37±2ºC with a soft nylon bristle toothbrush (Colgate Classic CLEAN, Colgate Palmolive Industrial Ltda., Osasco, SP, Brazil), with a load of 300g (Prakki et al., 2007; Prakki et al., 2008). Before to the toothbrushing test (ISO # 14569-1), a slurry was performed by mixing (2:1) of Colgate MFP toothpaste and deionized water (Colgate Palmolive, Osasco, SP, Brazil). Each group received 100,000 brushing strokes and the bristle toothbrush were changed after 50,000 cycles (Wang et al., 2004). After the toothbrushing test, all specimens were sonicated in deionized water for 10 min for cleaning (Prakki et al., 2008; Mondelli et al., 2015).

Figure 1 - Schematic representation of the toothbrush positioning on the sample.



Source: Authors.

2.4 Roughness and wear measurements

The surface roughness of all groups (n = 10) was assessed before (baseline) and after (final) the toothbrushing simulation test by roughness tester (Hommel Tester RT 1000, Hommelwerke, GmbH, Alte Tuttinger Strebe 20. D-7730 VS-Schwenningen, Germany). A diamond needle (Hommelwerke, T 1E, 100 5 90j 1.6–30 / 1.95 0.75/0; Art Nr.: 224160 GmbH) was used to perform five surface readings in random directions and the mean (Ra) was obtained. The reading parameters were determined at Lt (assessment length): 5 mm and Lc (cut-off): 0.25 mm.

For wear (µm) evaluation, a reading from the control side of the sample to the brushed side was performed and wear was calculated by the mean of 5 readings. The profilometer function of the same equipment was used in the parameters Lt: 10 mm and Lc: 0.00 mm.

2.5 Polymerization shrinkage stress evaluation

The shrinkage stress was measured from load cell deformation (Velo et al., 2019; Santin et al., 2021). To evaluate the polymerization shrinkage stress of composites, tensile test was performed on a Universal Testing Machine (Instron model 3342, Instron, Norwood, MA, USA) with a 50 Kg/F load cell. Samples from each group (n = 5) with 24 mm^3 volume were light-cured (Valo LED, Ultradent Products Inc, South Jordan, UT, USA) according to manufacturer's instructions. The experimental device consisted of two rectangular steel bases (6 × 2 mm^2) arranged parallel to one another. After adaptation of the composite into two metal bases (6 × 2 mm^2), samples were light-cured for 40s, per group, with a LED light-cure (Valo LED, Ultradent Products Inc, South Jordan, UT, USA) and, contraction forces were generated and captured by the Universal Testing Machine. At the end of 300 s, the contraction forces were converted to shrinkage stress (MPa). The metal base surfaces were blasted with aluminum oxide to ensure the bonding of the composite to the steel face. The polymerization shrinkage induced stress data were obtained by specific software and converted to MPa.

2.6 Microhardness testing

Disc-shaped samples measuring (2 × 2 mm^2) (n = 5) were developed by placing the composites into stainless steel molds and covered with a polyester strip. All samples were polished using decreasing grit abrasive papers (in series 600, 800, 1200, Buehler Ltd., Lake Bluff, IL, USA) for 2 min each, followed by 0.5 mm diamond paste (Buehler Ltd., Lake Bluff, IL, USA). For microhardness analysis in each group (n = 5), a microhardness tester (HMV-2000; Shimadzu Corporation, Tokyo, Japan) was used. A Knoop diamond was applied for 5 s with a 25 g load. Five random indentations were performed at a distance of 100 µm in the center of each specimen and the Knoop hardness number was obtained by the mean of the five indentations (Velo et al., 2019).

2.7 Statistical analysis

For surface wear, microhardness and shrinkage stress data, one-factor analysis of variance (ANOVA) and for roughness, two-factor analysis of variance (ANOVA) were performed. Multiple comparisons were applied for all analyses by Tukey's test ($\alpha = 0.05$). All statistical analyses were conducted using the statistical software (SPSS version 17.0; SPSS Inc).

3. Results

The two-way ANOVA revealed that there was statistically significance difference between material and period evaluated (before and after toothbrushing test) ($p < 0.001$). All groups showed increased roughness after brushing. Baseline roughness of all groups showed no difference ($p > 0.335$); however, after brushing, the Nanova group (1.79 ± 0.36) (Ra) presented highest values of roughness, while the Z350 group showed the lowest ($p < 0.031$) (Table 2).

Table 2 - Mean and standard deviation of baseline and final roughness (Ra) and wear (μm) for all tested groups.

Groups	Baseline	Final	Wear
Z350	0.13 (0.26)^{Aa}	0.50 (0.08)^{Ab}	5.59 (0.88)^{A}
Nanova	0.14 (0.22)^{Aa}	1.79 (0.36)^{Bb}	13.87 (3.26)^{B}
SDR	0.13 (0.18)^{Aa}	1.50 (0.15)^{Cb}	7.88 (2.06)^{A}

Different capital letter in the column and different lowercase letters in line indicates statistically significant difference at the 5% significance level. Source: Authors.

The one-way ANOVA test for evaluating the surface wear showed that the groups presented difference in wear ($p < 0.001$). The Nanova group had the highest wear (13.87±3.26) compared to the other groups ($p < 0.001$). The Z350 and SDR groups showed no difference ($p = 0.084$) between them (Table 2).

Table 3 - Mean and standard deviation of shrinkage stress (MPa) and microhardness (KHN).

Groups	Shrinkage stress (MPa)	Microhardness (KHN)
Z350	0.25 (0.05)^{A}	33.40 (0.46)^{B}
Nanova	0.24 (0.03)^{A}	52.56 (1.7)^{A}
SDR	0.21 (0.03)^{A}	26.32 (0.77)^{C}

Different capital letter in the column indicates statistically significant difference at the 5% significance level. Source: Authors.

For tensile test, all materials showed no difference in relation to shrinkage stress (Mpa) ($p = 0.468$) (Table 3). Regarding microhardness (KHN) results, the one-way ANOVA revealed the influence of the factors evaluated ($p < 0.001$). The Nanova group had the highest hardness value (52.56±1.7), followed by the Z350 (33.40±0.46) and SDR (26.32±0.77) group, with statistical difference between all groups ($p < 0.006$) (Table 3).

4. Discussion

Resin-based based materials undergo loss of mechanical properties and aesthetics as a function of fatigue, aging at body temperature and a moist environment (Borgia et al., 2019). Considering the limited published studies in the literature regarding nanofiber-reinforced restorative composites, this study aimed to evaluate the properties of surface wear, roughness, shrinkage stress and microhardness of the new flowable nanofiber composite resin compared to different low-viscosities resin composites. Based on our results, the first and third null hypothesis were rejected, since the nanofibers bulk-fill flow resin composite presented higher surface microhardness and similar polymerization shrinkage stress when compared to the control groups.

Indications for the use of flowable resin composites range from minimally invasive occlusal restorations, pit and fissure sealants to Class V abfraction lesions (Baroudi & Rodrigues, 2015). The composition of Nanova contains calcium phosphate (hydroxyapatite) nanofibers (NanovaPro Flow, Nanova, Columbia, MO, USA). The manufacturer's claims that this class of material has high flexural and micro-tensile bond strengths, low shrinkage stress, improving marginal quality, without sacrifice handling properties (Nanova, 2016). However, some clinical limitations still remain, due to the mechanical deficiency of flowable materials, especially from the previous generation (Attar et al., 2003).

A previous study evaluated the physical-mechanical properties of a packable NanovaPro Fill, comparing it with traditional hybrid composites (Yancey et al., 2019). It was concluded that there was not significant advantage to use this new class of material, since nanofiber composite had similar flexural strength, shrinkage, and degree of conversion than the traditional resin composite Esthet-X HD (Yancey et al., 2019). The mechanical properties of resin-based composites also influence the fracture strength of restored teeth and, therefore, higher values of surface hardness could lead to greater fracture resistance (Habekost et al., 2007; Velo et al., 2019). Our results demonstrated the highest microhardness value for the group Nanova (Table 3). Considering that the increase in filler content is expected to increase the material hardness values (Alrahlah et al., 2014; Ilie & Stark, 2015; Rodriguez et al., 2017) the higher filler content in volume (40-80%) presented by Nanova than the SDR (47.3%) and Z350 (55.6%) groups can explain such results, in addition to the reduced diameter of nanofibers, which generally implies in a significant increase in strength (Papkov et al.,2013).

On the other hand, the Nanova group had a rougher and more surface wear than the control groups (Table 2). The rougher surface of fiber-reinforced resin-based composites is related to their mechanical properties and enhanced fracture resistance. A rough surface of the composite suggests that the presence of nanofibers effectively deflect a crack propagation in a fracture presence, minimizing the dispersion of tension and increasing the strength of the material (Tian et al., 2007; Rodriguez et al., 2017; Velo et al., 2019). In a clinical scenario, being a wear resistant material plays an important role, since the structure of the composite materials is less degraded when submitted to masticatory forces and hydrolytic decomposition (de Gee et al., 1966). The chewing cyclic stress of these materials can lead to the development of restorations cracks and fracture (Ferracane, 2013). The present study observed that toothbrushing with a toothbrush was able to alter the equilibrium between the organic matrix and the filler, increasing the roughness of all groups, especially for Nanova (Table 2). In contrast, previous study has shown the lowest wear depth values for a fiber-reinforced flowable resin composite (GC Corp, Tokyo, Japan - experimental), suggesting better performance of this class of material in high stress-bearing application area (Lassila et al., 2019). In the current study, the better wear resistance of the Z350 group than the SDR group can be explained by the use of nanoparticles and nanoclusters in composition, improving particle distribution and interaction (Rizzante et al., 2019). However, it is important to note that, a restorative material should present wear properties similar to those of tooth substrates (approximately 0.02-0.04 mm in enamel of vertical loss per year) (Lambrechts et al., 1989; Dionysopoulos & Gerasimidou, 2021) and, even with the advanced in dental materials over years, wear is still a main concern for all resin composites.

Whereas flowable resin composites are still mainly indicated for minimally invasive procedures, the challenges incudes a limited access and difficulties that control excavation and fill the cavities. Therefore, the concern related to the polymerization shrinkage stress, which can result in interfacial gaps, marginal discoloration and adjacent caries was investigated herein. Anttila et al., 2008 showed that the use of fibers could reduce polymerization shrinkage. In this study, the Nanova group performed a similar polymerization shrinkage to the other groups, without significant difference (Table 3). The results were similar to Yancey et al., 2019, when compared with a nanohybrid and microhybrid composite. In addition, promising results from bulk-fill composites are related to the use of larger increments (up to 4 mm) with lower polymerization shrinkage, due to the incorporation of high molecular weight monomers and stress relievers (Garcia et al., 2014; Kruly et al., 2018; Meereis et al., 2018). However, this improvement was not observed in this study when compared to other flowable composite resins.

5. Conclusion

The fiber-reinforced flowable bulk-fill resin composite presented better mechanical properties and higher surface wear loss, without affect the polymerization shrinkage stress. Clinical trials long-term follow-up with should be conducted to provide robust data about the use of this class of material in occlusal surface, since it has been indicated as minimally preparations and sealants of pit-and-fissure.

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