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Effect of acid challenge and photoactivation distance on microhardness and roughness of flow bulk-fill composite resins

Efeito do desafio ácido e distância de fotoativação na microdureza e rugosidade de resinas compostas bulk-fill flow

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Resumo

Introdução: as resinas compostas são indicadas para reconstrução de paredes proximais e a avaliação das propriedades das resinas compostas bulk-fill flow expostas ao desafio ácido mostra-se necessária. **Objetivo:** avaliar a microdureza e rugosidade em diferentes profundidades de fotoativação de resinas compostas bulk-fill flow (Filtek Bulk Fill Flow; SureFil SDR Flow; Tetric N Ceram Bulk fill) e convencional (controle, Filtek Z350 XT) submetidas ao desafio ácido. **Material e método:** quarenta amostras retangulares (3x3x4 mm) foram confeccionadas utilizando uma matriz de poliacetal. Para simular o desafio ácido, as amostras foram imersas em uma solução desmineralizante. Cada amostra teve a microdureza Knoop (KHN) e rugosidade (Ra) avaliadas em três profundidades (superficial, média e cervical), considerando a superfície lateral da amostra. Os dados foram submetidos aos testes de Kruskal-Wallis, Friedman e Dunn com nível de significância de 5%. **Resultado:** comparando as resinas compostas entre si, nas regiões superficial (p=0,693), média (p=0,053) e cervical (p=0,176), não houve diferença nos valores de KHN. Também não houve diferenças na rugosidade entre os materiais nas regiões superficial (p=0,356), média (p=0,734) e cervical (p=0,207). Apenas o Filtek Z350 XT (p=0,027) apresentou menor diferença de KHN na região intermediária causada pelo desafio ácido. As mudanças na rugosidade mostraram que a maior diferença foi na região cervical para Bulk Fill Flow SDR (p=0,014) e Tetric N-Ceram Bulk Fill (p=0,003), com aumento após o desafio ácido. **Conclusão:** após desafio ácido, as resinas compostas bulk-fill flow apresentaram alterações semelhantes às apresentadas pela resina composta convencional nanoparticulada.

Descritores: Resinas compostas; restauração dentária; cárie dentária.

Abstract

Introduction: composite resins are indicated to the reconstruction of proximal walls and the evaluation of properties of flow bulk-fill composite resins exposed to acid challenge is necessary. **Objective:** to evaluate the microhardness and roughness at different depths of photoactivation of bulk-fill flow composites (Filtek Bulk Fill Flow; SureFil SDR Flow; Tetric N Ceram Bulk fill) and conventional composite resin (control, Filtek Z350 XT) subjected to acid challenge. **Material and method:** forty composites brick shaped specimens (3x3x4 mm) were made using a polyacetal matrix. To simulate pH challenges, the samples were immersed in a demineralizing solution. Each sample had Knoop microhardness (KHN) and roughness (Ra) evaluated at three depths (superficial, medium, and cervical), considering the lateral surface of the sample. Data were submitted to Kruskal-Wallis, Friedman's and Dunn's tests with a significance level of 5%. **Result:** comparing the

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composites among themselves, in superficial (p=0.693), medium (p=0.053) and cervical (p=0.176) regions, there was no difference in the KHN values. There were also no differences in roughness between the composites in superficial (p=0.356), medium (p=0.734) and cervical (p=0.207) regions. Only the Filtek Z350 XT (p=0.027) showed less difference in KHN in the middle region caused by acid challenge. Changes in roughness showed that the greatest difference was at the cervical region for Bulk Fill Flow SDR (p=0.014) and Tetric N-Ceram Bulk Fill (p = 0.003) with an increase after acid challenge. **Conclusion:** after acid challenge, bulk-fill flow composites showed alterations similar to those presented by the conventional nanoparticulate resin composite.

Descriptors: Composite resins; dental restoration; dental caries.

INTRODUCTION

Since the introduction of composite resins more than 50 years ago, they have undergone constant development and have proven to be clinically effective[1]. It is estimated that more than 290 million restorations are made each year worldwide and among the most frequently used restorative materials are composite resins[2]. Clinical protocols recommend the insertion of conventional composite resins in increments of no more than 2 mm thickness, to proportionally reduce the volumetric contraction of the material, reduce the level of tension developed by polymerization and, therefore, reduce the occurrence of crack formation in the interface with the dental cavity[3]. However, restoration procedures of extensive Class II proximal boxes are challenging mainly because of the difficulty for the light from the photoinitiator to access the area, often compromising polymerization[4,5].

On the other hand, there are negative points in the incremental insertion technique: execution time is increased, empty spaces or bubbles can be incorporated into the restoration body, risk of moisture contamination between the layers increases and working in small cavities can become more difficult[6]. Thus, restorations involving large cavity preparations are time-consuming procedures for the operator and often uncomfortable for the patient[7]. In this sense, composite resins with different technologies have been developed to reduce the effects caused by polymerization shrinkage stress of composite resins and to reduce operating time for posterior teeth restorations[8]. Bulk-fill composites were developed to enable 4-5mm single layer insertions into dental cavities[2,9]. These materials offer greater translucency, allowing for increased light transmission through the material, have more reactive photoinitiators, which allow a greater depth of polymerization and include monomers that act as polymerization reaction modulators, thus achieving low polymerization shrinkage[10].

In addition, bulk-fill resin composites can be found in two forms: high viscosity bulk-fill resin composites and fluid bulk-fill resin composites (flow, low viscosity). High viscosity bulk-fill composite resins can be used to recover the entire cavity, including the occlusal surface as they are more resistant and contain more inorganic loading[1]. Bulk-fill flow composite resins have greater fluid characteristics and, therefore, allow placement by means of syringes facilitating insertion and adaptation in less accessible cavities and generally adapt better on cavity walls, especially on uneven surfaces however, they must be covered with a final, occlusal layer of a high viscosity composite. In the proximal region of the restoration, however, bulk-fill flow composites are exposed to the oral environment[11,12].

Effective marginal adaptation is of great concern with regard to posterior restorations. The main reasons for the clinical failure of direct composite resin restorations on posterior teeth over time are secondary caries and restoration fractures[10]. Marginal seal involves several factors: cavity configuration, physical-mechanical and viscoelastic properties of the composite resin, adhesion, restoration technique, polymerization method, among others[4,7]. In addition, in contact with the oral environment, composite resins suffer wear and

influence from chemicals that act superficially on the material structure[3]. Accumulated biofilm on the restoration can also produce acidic substances that result in degradation of the surface and can lead to softening of the material, reducing its hardness and increasing its roughness[13,14].

Other than that, photopolymerization of composites is one of the main factors for clinical success of a restoration[15,16]. Dental product manufacturers rely on placing the tip of the curing light as close as possible to the composite surface, but in clinical situations, this positioning is often difficult or impossible to achieve. For example, the distance between the tip of the cusp and the base of the interproximal box may exceed 7 mm, a distance that will significantly reduce the intensity of the available light for photoactivation of the composite[16]. As a result, the manufacturer's photoactivation times may underestimate the times needed to properly polymerize the composite resin at the bottom of the interproximal box[17].

The transmittance of light varies between products due to differences, for example, in inorganic loading, in composition of the composite matrix and in the presence of pigments[15]. Flowable composite resins differ from universal composite resins mainly due to the lower load and also their light transmitting properties can vary[15]. Specifically in bulk-fill composite resins, refraction coefficients and light attenuation of monomers change during polymerization, allowing light to be better transmitted through the composite resin[15].

Mechanical properties, such as surface hardness and abrasion resistance of bulk-fill flow composite resins, are inferior to those of paste consistency composites[8]. Occlusal surface of a Class II restoration, using a bulk-fill flow composite resin base, is covered by a final layer of conventional composite resin, however, the proximal wall and the interface between the gingival wall and the bulk-fill flow composite resin restoration can remain exposed to the oral environment. Thus, flow bulk-fill composite resins are susceptible to degradation when exposed to the various incidents that normally occur in the oral cavity[1]. Bearing in mind that the manufacturers of flow bulk-fill composite resins, indicate the reconstruction of proximal walls completely with this material, it would be interesting to check the surface characteristics and microhardness of flow bulk-fill composite resins when subjected to acid challenge, simulating what occurs on the proximal faces of the restorations. The aim of this study was to evaluate microhardness and surface roughness of bulk-fill flow composite resins submitted or not to acid challenge.

MATERIAL AND METHOD

Sample preparation

The experimental units were samples of bulk-fill flow composite resin with dimensions of 3 mm x 3 mm by 4 mm in height. Four composites were used: a) Bulk fill flow (SDR flow Dentsply); b) Bulk fill N Ceram flow (Ivoclar Vivadent); c) Filtek Bulk fill flow (3M ESPE); nanoparticulate composite (Filtek Z350XT, 3M ESPE). Surface roughness and microhardness were evaluated in each sample before and after acid challenge at three photoactivation distances on the side of the sample: a) superficial; b) medium and c) cervical. Changes in microhardness and surface roughness at each region of each sample were calculated. Samples had their surface characteristics examined using scanning electron microscopy. Table 1 describes the composition and way of use of the materials from the study.

Thirty brick-shaped specimens, simulating a proximal cavity of posterior tooth, of bulk-fill composite resins were made using a polyacetal matrix, with dimensions of 3 x 3 x 4 mm in height, following manufacturers' recommendations (Table 1). A split polyacetal matrix was used, positioned on a set of glass plate and polyester strip. Then, the bulk-fill resins were inserted as a single increment in the matrix, and another polyester strip and a weight of 500 grams was placed

for 15 seconds. The samples were photoactivated according to the manufacturers' instructions for 40 seconds with an LED photoactivation device (Valo, Ultradent Products Inc, S. Jordan, UT, USA). The minimum light intensity was 1000mW/cm^2.

Table 1. Materials, manufacturers, composition and way of using the main materials from the study

Material/Manufacturer	Composition (percentages by weight)	Instructions
SureFill SDR Flow Dentsply Caulk, Milford, DE, USA Lot: 00013282	Matrix: Dimethacrylated urethane (<10%), polymerizable dimethacrylate (<10%), ethoxylated bisphenol A, dimethacrylate, pigments, photoinitiator. Inorganic loading: barium, boron aminofluorsilicate (<50%)	Single increment of 4 mm photoactivation for 40 seconds.
Tetric N Ceram Bulkfill Ivoclar Vivadent AG, Schaan, Liechtenstein Lot: x18772	Organic matrix: Bis GMA (<10%); UDMA (<10%), Bis EMA; Dimethacrylates. Inorganic loading: barium glass, prepolymer, ytterbium trifluoride and mixed oxides (<10%).	4 mm layer insertion, photoactivation for 40 seconds.
Filtek Bulk Fill Flow 3M / ESPE, St Paul, MN, USA Lot: 1817000639	UDMA (10-20%); substituted dimethacrylate; BisGMA; BisGMA; benzotriazole; TEGDMA and ethyl 4-dimethylaminobenzoate. Ytterbium fluoride (1-10%), treated silanized ceramics (50-60%)	Application with the product tip in a single 4 mm layer insertion, photoactivation for 40 seconds.
Filtek Z350 XT 3M ESPE, Saint Paul, MN, USA Lot: 1816300690	Organic Matrix: Bis-GMA, UDMA,Bis-EMA, PEGDMA and TEGDMA, Fluorescent Agents, Pigments, Stabilizers, Initiators.Inorganic matrix (load): Zirconia / Silica: 3µm or less, Zirconia / Silica Agglomerate, Silica with treated surface: 20nm 67.8%	Inserted incrementally, each of these 2 mm increments thick and 20 seconds photoactivation in each layer.

Legend: BisGMA - Bisphenol A glycidyl dimethacrylate; TEGDMA - triethylene glycol dimethacrylate; UDMA - Urethane dimethacrylate; MDP - Dihydrogen phosphate methacrylate; HEMA - hydroxyethyl methacrylate; Bis-EMA - Bisphenol-A dimethacrylate ethoxylate; PEGDMA - Polyethylene glycol dimethacrylate.

The specimens of nanoparticulate composite resin were obtained in the same manner, but in two increments of 2 mm individually activated. The distance between the light source and the specimens was standardized using a polyester strip. After preparation of the samples, they were stored in an environment with relative humidity, for 24 hours at 37 ^{o}C.

Acid challenge

To simulate cariogenic challenges, a demineralizing solution (pH 4.3) was used, dynamic physicochemical models of caries have used this solution*, which contains 2 mM calcium and 2 mM phosphate, in acetate buffer 74 mM. Considering that after 10 daily cariogenic challenges, for 14 days, that is, after 140 cariogenic challenges, the composite resin presents a significant reduction in its hardness[18], and that the drop in the saliva and microenvironment pH biofilm remains below 5.5 for approximately 45 minutes, in this study to simulate a total of 14 days, the samples were immersed in 2.5 mL of the demineralizing solution for 6300 minutes (105 hours), with daily changes every 12 hours.

Microhardness evaluation

The lateral surface of each sample was divided into three equal regions representing the superficial region (close to the occlusal surface), the middle region and the cervical region. After that, samples had their microhardness (microhardness testing machine - Pantec, digital HVS - 1000)

*Featherstone JDB, O'Really MM, Shariati M, Brugler S. Enhancement of remineralization *in vitro* and *in vivo*. In: Leach SA, editor. Factors related to demineralization and remineralization of the teeth. Oxford: IRL; 1986. p. 23-34.

measured at the three levels (superficial, medium and cervical) and values were obtained in KHN (Knoop hardness number). The test was carried out by applying a load of 25 grams for 5 seconds. Each sample was positioned in a standardized manner on the microhardness testing machine and fixed with wax. In each region, a random point was established in the center of the region and three equidistant indentations at 100 µm were made for each evaluation, with a Knoop indentator.

Surface roughness assessment

The surface roughness assessment was performed using a contact profilometer (Mitutoyo). The evaluation was carried out in a 2.5 mm path divided between three 0.25 mm cut-offs, in sequential mode. The average roughness (Ra) of each reading path was evaluated, with 3 readings in each region of the samples (superficial, medium, cervical). The path speed of the profilometer needle was 0.05 mm/s.

Scanning electron microscopy

Samples from each group were observed in Scanning Electron Microscopy (SEM) to characterize the surface, samples were placed in a metallizer (Quorum Q150R ES), the surface was covered with gold and after observed under SEM (Tescan model Vega LM 3) where a current of 15.0 kV was used. The images were obtained in 1000X magnification.

Statistical analysis

For the analysis, changes in Knoop microhardness and surface roughness were obtained, calculated by the difference between the final and initial values of each sample. Bearing in mind that the data did not adhere to homogeneity of variance and normal distribution, even with transformation procedures, Kruskal-Wallis and Friedman non-parametric analysis were applied, followed by Dunn tests. SPSS 23 program (SPSS Inc., Chicago, IL, EUA) was used to process the calculations, adopting a 5% significance level.

RESULTS

Comparing the composite resins using Kruskal-Wallis tests, it was observed that on the surface ($p = 0.693$), the middle ($p = 0.053$), and cervical ($p = 0.176$) regions, there was no statistically significant difference in Knoop microhardness values. Having in mind that the p value for the findings in the middle region was borderline, it can be suggested that there was a greater change in microhardness after acid challenge in the composite resin Z350 XT (Table 2). The percentage of microhardness changes indicated a decrease in all composites and regions evaluated after acid challenge.

For surface roughness data, there were also no statistically significant differences between the composite resins, at any of the surface ($p = 0.356$) middle ($p = 0.734$) and cervical ($p = 0.207$), as noted in Table 2. The percentage of change in roughness demonstrated that, after acid challenge, there was an increase in roughness in the cervical third of all materials.

Table 2. Mean values and standard deviations of the initial and final values. and of the absolute and relative (%) alteration of Knoop microhardness and surface roughness of composite resins as a function of the occlusal distance

Property	Composite Resin	Before acid challenge			After acid challenge			Alteration (final – initial)#			Alteration (%)#		
		Superficial	Medium	Cervical	Superficial	Medium	Cervical	Superficial	Medium	Cervical	Superficial	Medium	Cervical
Knoop microhardness (Kg/mm²)	Bulk Fill Flow SDR	28.26 (4.64)	26.77 (2.76)	26.80 (4.24)	22.16 (2.89)	22.60 (7.89)	21.12 (6.24)	-6.10*a (5.62)	-4.18*a (9.39)	-5.69*a (7.69)	-19.71 (15.88)	-13.38 (38.25)	-19.57 (26.70)
	Tetric N-Ceram Bulk Fill	34.41 (7.47)	34.46 (6.31)	37.19 (6.13)	25.32 (7.23)	26.96 (8.66)	27.50 (13.07)	-9.10*a (9.93)	-7.49*a (11.96)	-9.70*a (13.33)	-24.03 (25.91)	-17.79 (37.00)	-25.19 (33.28)
	Filtek Bulk Fill Flow	40.37 (15.90)	37.61 (13.40)	39.99 (8.50)	32.62 (10.93)	33.90 (13.77)	23.70 (6.32)	-7.75*a (14.22)	-3.71*a (22.21)	-16.29*a (10.95)	-14.95 (29.46)	0.96 (51.66)	-37.83 (23.34)
	Filtek Z350 XT	97.65 (27.47)	95.91 (33.56)	93.99 (30.64)	75.93 (27.88)	61.79 (22.22)	64.65 (24.24)	-21.72*a (29.30)	- 34.12*b (43.55)	-29.33*ab (42.91)	-20.97 (25.68)	-30.21 (31.28)	-25.02 (34.88)
Surface roughness (µm)	Bulk Fill Flow SDR	0.607(0.146)	0.559 (0.144)	0.586 (0.238)	0.447 (0.113)	0.606 (0.231)	1.045 (0.435)	-0.160*a (0.151)	0.047*a (0.208)	0.458*b (0.420)	-23.41 (21.02)	9.81 (39.11)	94.29 (93.36)
	Tetric N-Ceram Bulk Fill	0.523 (0.141)	0.502 (0.100)	0.485 (0.122)	0.454 (0.130)	0.509 (0.234)	0.647 (0.277)	-0.069*a (0.136)	0.008*a (0.179)	0.162*b (0.239)	-10.23 (23.70)	-0.43 (31.69)	34.17 (49.31)
	Filtek Bulk Fill Flow	0.560 (0.091)	0.551 (0.181)	0.570 (0.155)	0.428 (0.120)	0.486 (0.127)	0.737 (0.307)	-0.132*a (0.135)	- 0.065*a (0.230)	0.167*a (0.411)	-22.22 (23.34)	-1.65 (47.27)	46.14 (92.59)
	Filtek Z350 XT	0.796 (0.260)	0.864 (0.299)	0.821 (0.142)	0.722 (0.109)	0.820 (0.197)	0.928 (0.224)	-0.075*a (0.195)	- 0.045*a (0.315)	0.107*a (0.208)	-4.15 (20.64)	2.40 (37.12)	14.37 (28.13)

Legend: Standard deviations are in parentheses. Averages followed by asterisks indicate the absence of a statistically significant difference between composite resins, considering each occlusal distance separately (comparisons within each column), whether for microhardness or roughness changes; averages followed by distinct lowercase letters indicate a statistically significant difference between the values obtained in each distance, considering each composite resin separately (comparisons within each line). #Average of change obtained from the individual calculation of each sample in each group. Negative values of absolute or relative change indicate reduction of microhardness / roughness after acid challenge; positive values of absolute or relative change indicate an increase in microhardness / roughness after acid challenge.

When comparing the Knoop microhardness values at the different distances on the lateral face of the sample, Friedman tests indicated that there was no difference at the surface, middle and cervical regions when considering Bulk fill Flow SDR (p = 0.670), Tetric N -Ceram Bulk fill (p = 0.905), and Filtek Bulk fill Flow (p = 0.273). As for the composite resin Filtek Z350 XT (p = 0.027), on the surface microhardness was significantly lower than in the middle region, while in the cervical region intermediate values were observed, which did not differ from those found on the surface and in the middle region (Table 2).

Friedman tests also revealed that while for the Filtek Bulk Fill Flow (p = 0.150) and Filtek Z350 XT (p = 0.497) composite resins, there was no statistically significant difference in roughness at the surface and medium and cervical regions for Bulk materials Fill Flow SDR (p = 0.014) and Tetric N-Ceram Bulk Fill (p = 0.003), the roughness was significantly affected by occlusal distance. For both composite resins (Bulk Fill Flow SDR and Tetric N-Ceram Bulk Fill), in relation to the surface and the middle region, significantly higher changes in roughness occurred in the cervical region, in which there was an increase in roughness after acid challenge (Table 2).

Figure 1 and Figure 2 show SEM images obtained before acid challenge and Figure 3 and Figure 4 show SEM images after acid challenge, characterizing the superficial, middle, and cervical thirds of the proximal wall. All tested composite resins showed similar surface morphology characteristics before and after immersion in demineralizing solution. When the resins are compared to each other, it is observed that the surface changes are similar between them, except for the cervical and middle region of the Z350 XT resin, which showed greater change. When the comparison takes place between the thirds of each composite individually, it is observed that all images are also similar to each other.

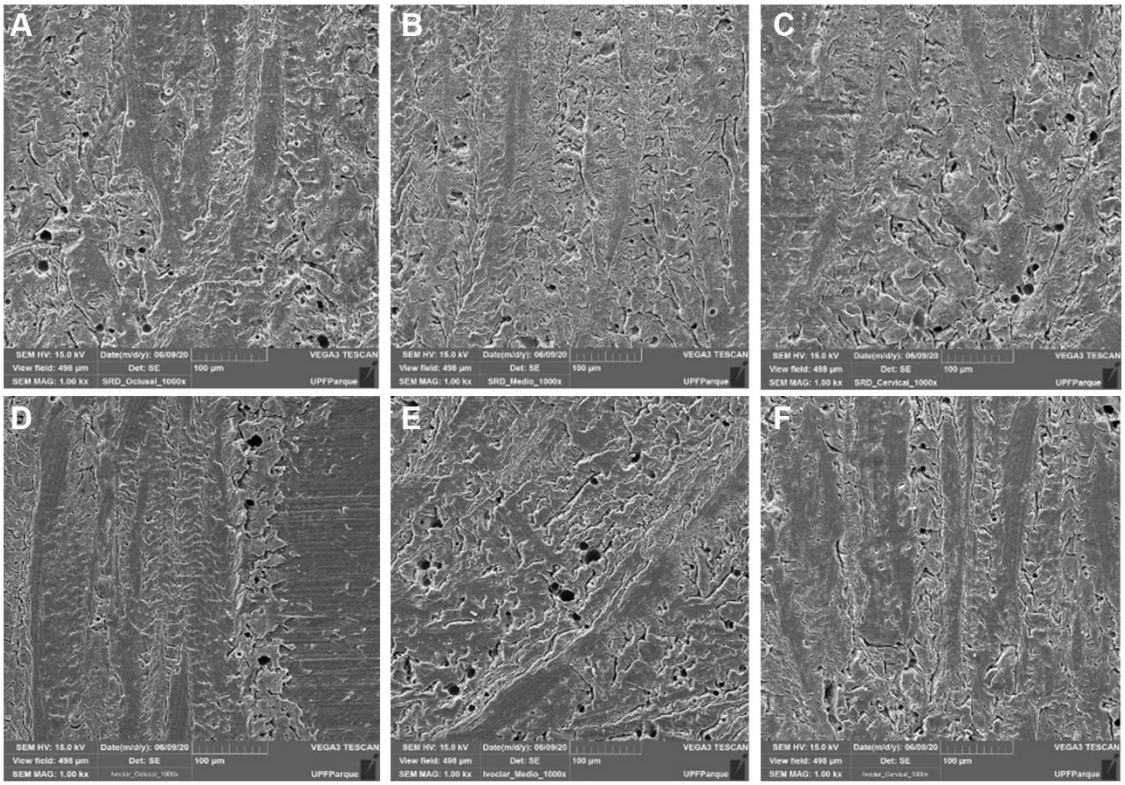

Figure 1. Scanning electron microscopies (1000x) of Bulk fill Flow SDR (A, B, C) and Tetric N-Ceram Bulk fill (D, E, F) composite resins in the superficial (A, D), middle (B, E) and cervical (C, F) thirds of the proximal wall before acid challenge.

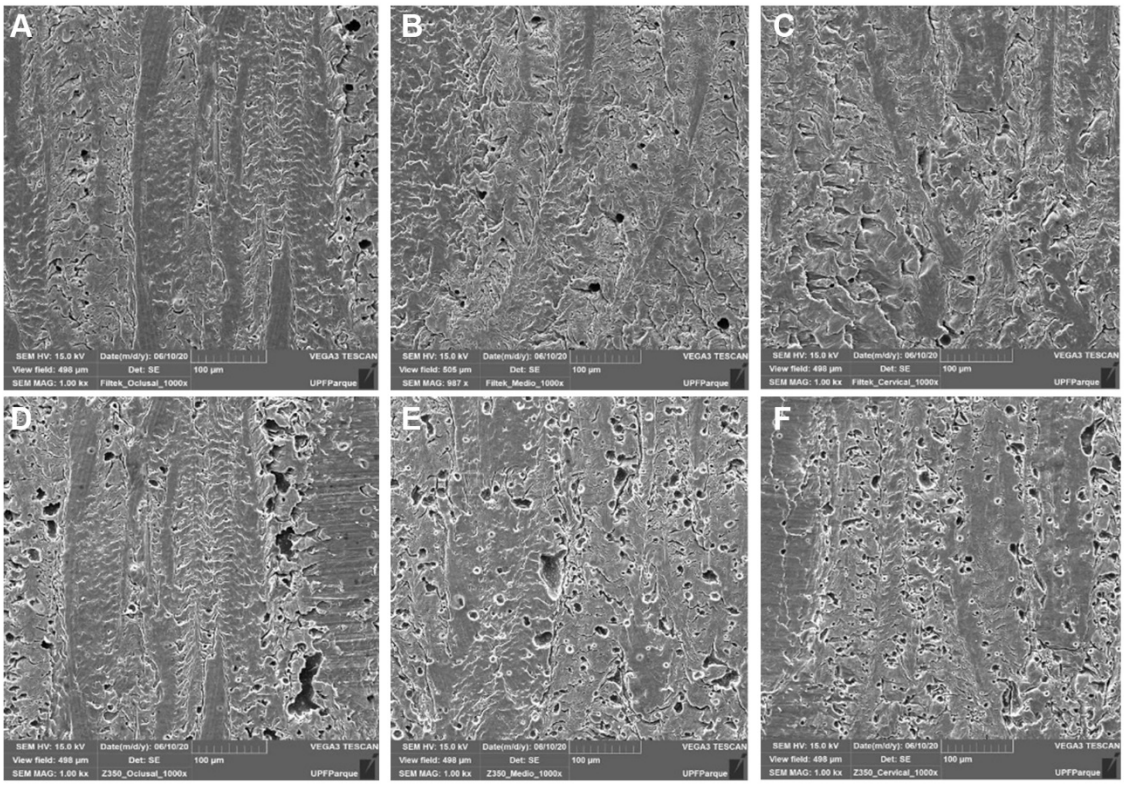

Figure 2. Scanning electron microscopies (1000x) of Filtek Bulk fill (A, B, C) and Filtek Z350 XT (D, E, F) composite resins in the superficial (A, D), middle (B, E) and cervical (C, F) thirds of the proximal wall before acid challenge.

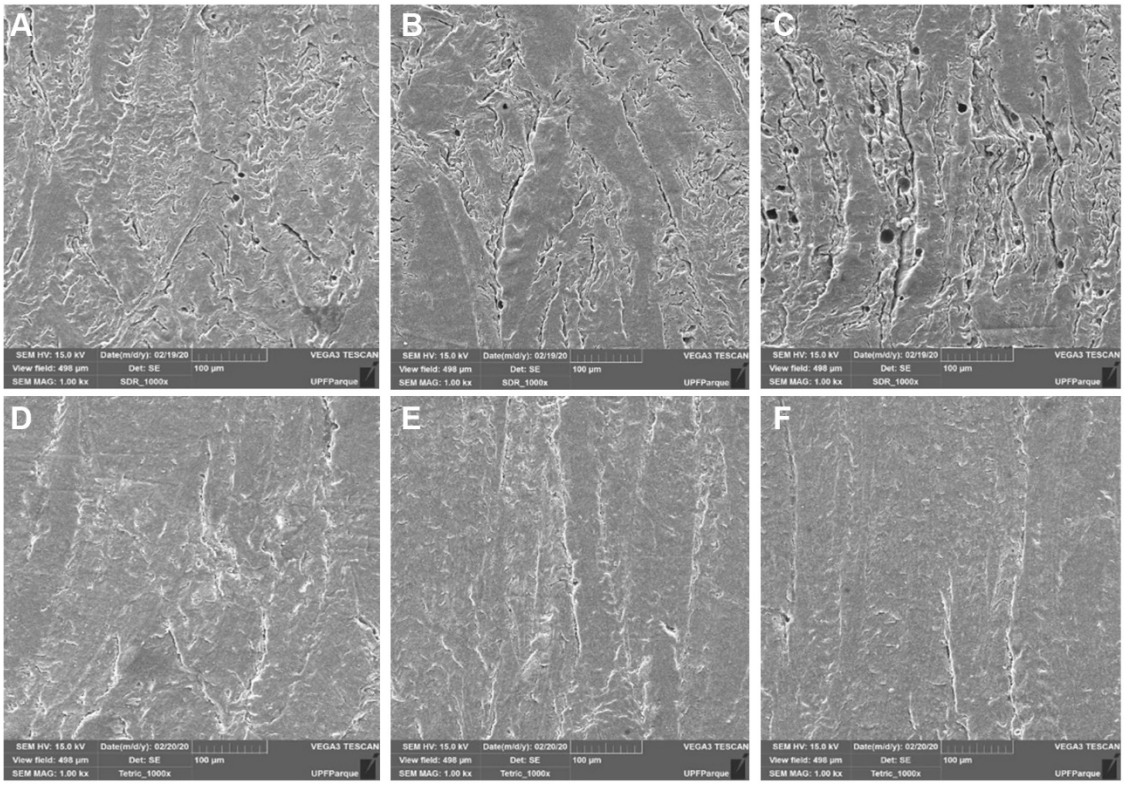

Figure 3. Scanning electron microscopies (1000x) of Bulk fill Flow SDR (A, B, C) and Tetric N-Ceram Bulk fill (D, E, F) composite resins in the superficial (A, D), middle (B, E) and cervical (C, F) thirds of the proximal wall after acid challenge.

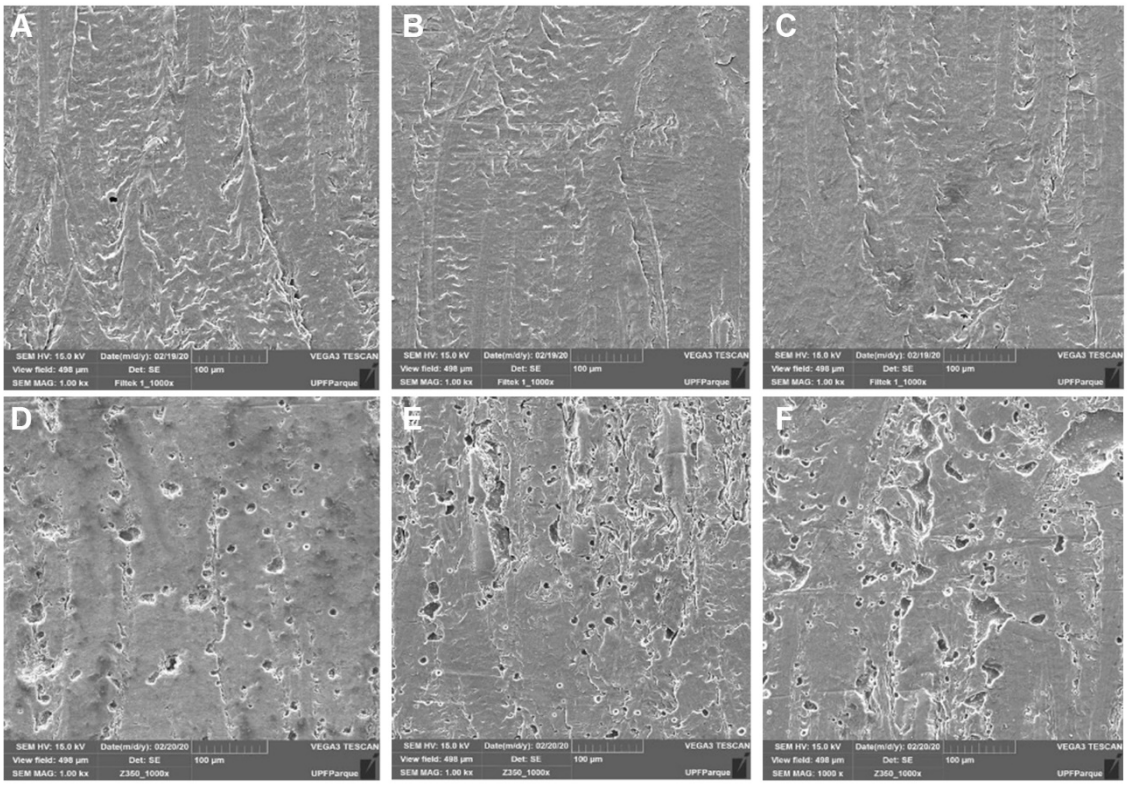

Figure 4. Scanning electron microscopies (1000x) of Filtek Bulk fill (A, B, C) and Filtek Z350 XT (D, E, F) composite resins in the superficial (A, D), middle (B, E) and cervical (C, F) thirds of the proximal wall after acid challenge.

DISCUSSION

The results showed that there was no difference in the microhardness values between low viscosity bulk-fill composite resins as well as between the studied depths. There was also no difference in surface roughness presented between the composites or when the different depths were evaluated. This similar behavior between materials may be due to the composition of the materials. Manufacturers have used several strategies in the formulation of bulk-fill composites, including the use of more reactive photoinitiators that are capable of absorbing irradiation energy, even in deeper layers, decreasing the amount of filler particles, improving translucency to facilitate light transmission, using singular monomers that act as stress relievers and incorporating different types of loading particles, such as prepolymerized particles and fiberglass-based segments[19,20].

Although announced as a new class of materials, bulk-fill composites do not fundamentally differ in their filler particle composition from nanohybrid, microhybrid and nanoparticulate composite resins. They are basically constituted by an organic matrix and load particles chemically linked by silane. Regarding the organic matrix, it is mainly formulated with dimethacrylate monomers, such as BIS-GMA, TEGDMA or UDMA, which constitutes the composite body and can influence the handling capacity and material properties, on the other hand, silica and glass are loading agents generally used to modify the aesthetic, physical and mechanical characteristics of composites[13,21].

In the SDR Bulk fill flow composite there was a change in polymerization reaction dynamics through the incorporation of a photoactive group in the urethane dimethacrylate monomer that controls the polymerization kinetics and allows the insertion of increments of up to 4 mm. This technology demonstrated a 60 to 70% decrease in polymerization shrinkage tension of this material[21,22]. One of the mechanisms used to reduce contraction stress is prolonging the initial

phase of polymerization (pre-gel) which is characterized by the formation of more flexible polymeric chains allowing the material to deform from the free adhesion walls and, thus, compensating internal material stresses generated by polymerization shrinkage[22].

The Filtek Bulk fill Flow resin (3M / ESPE) which has a fluid consistency has other monomers in addition to Bisphenol A glycidyl methacrylate (BisGMA) such as urethane dimethacrylate (UDMA), urethane aromatic dimethacrylate (AUDMA) and fragmentation monomers. During polymerization, some monomers can fragment at the ends of the polymer, promoting less polymerization shrinkage and alleviating its effect on the restoration walls[3]. Additionally, the presence of the AUDMA monomer requires fewer monomeric bonds to form the polymer due to its higher molecular weight, which favors the reduction of polymerization shrinkage. There is also less light scattering allowing the entire composite resin to be photoactivated even in deeper areas[23]. Even though insertion techniques were different, single increment for bulk-fill composites and horizontal increments for the nanoparticulate composite resin, the composition characteristics may have guaranteed good performance of the microhardness and roughness of the composite after the acid challenge.

Finally, Tetric N-Ceram contains tertiary amine/camphorquinone initiation system, but also contains a photo-initiator system called Ivocerin, based on germanium, which has been added to the material. Ivocerin initiator is considered more effective than camphorquinone alone, as it allows a greater and constant polymerization depth of 4 to 5 mm[2]. The main advance in polymerization depth for the latest generation of bulk-fill composites is the increase in translucency which improves the dispersion and penetration of blue light in depth. In addition, more rounded charge particles are used which helps increase translucency of this material[2,8,24].

Regarding photopolymerization, the fact that when light is applied over a composite, irradiance decreases as it is reflected, dispersed, and attenuated by the surface layers is well known. Thus, deeper layers are generally less polymerized. Uniform distribution of emitted energy in all layers of restorative material has been reported to be crucially important to produce enough free radicals for proper polymerization[25]. In this context, the bulk-fill composites used in this study have indications for use in layers of up to 4 mm and the nanoparticulate resin was used in 2 mm increments. Additionally, the LED light used emits more than one wavelength which can favor photoactivation of materials that have other photoinitiators different from camphorquinone[26]. Therefore, these factors related to the correct insertion technique and the power of the light unit used may have contributed to the similarity between microhardness and roughness results obtained.

The proposed cariogenic challenge, because of its acidity, could influence the increase in dissolution, plasticize the polymeric matrices and dislodge the charge particles, resulting in a surface strength and hardness decrease, increasing the degree of erosion in composite resin materials. Resin matrices can also promote the displacement of load particles from the external surface, resulting in a rapid increase in the surface roughness, decrease in the microhardness of these composites and facilitate degradation[27]. Although the effect of acid challenge was similar in altering microhardness of the tested materials, a percentage reduction in microhardness was observed after immersion in the demineralizing solution for all materials in all evaluated depths. Another factor that can be related to the decrease of microhardness in addition to the erosive effect of the acid character solution, was the water sorption by the composites that were immersed for 14 days. It is known that the interface between the organic matrix and load fillers can allow paths for the diffusion of water, which can have consequences of degradation after acid challenges[20].

The results showed that the nanoparticulate composite resin showed significant change in microhardness in the middle third after acid challenge. This is a nanoparticulate composite and was used in horizontal increments of 2 mm. Z350 XT composite resin contains PEGDMA and TEGDMA, which have low molecular weight, and contribute to an elevated number of double bonds per unit weight and creates a high degree of crosslinking,

generating a rigid composite with relatively high shrinkage. Even though 2 mm increments were used, a decrease in the microhardness at the middle third was observed after acid challenge. It is known that an acid medium can influence the increase in dissolution, plasticize the polymeric matrices and dislodge the load particles, resulting in a decrease in surface strength and hardness, and an increase in the degree of erosion in composite resin materials[27]. In addition, it is considered that the union between the increments used may have occurred in the middle third of the cavity and the lower average of microhardness in this region reflects the interface between the increments.

The results also showed that the SDR Bulk fill flow and Tetric N Ceram composites displayed greater roughness in the cervical third after acid challenge. This result can be explained by the inorganic components of the material compositions. Tetric N Ceram resin contains prepolymerized agents that are largely organic, which means that the proportion of inorganic fillers is lower. In general, incorporation of lower amounts of inorganic filler particles in the polymer matrix can increase the free volume available for water absorption. As the material content of the inorganic matrix decreases, the interface between the organic matrix and load particles tends to become larger, providing easy paths for the diffusion of water which can have degradation consequences after acid challenges[20]. In addition, the size of the load particles has been associated with the surface properties of composite resins. In this sense, the SDR Bulk fill flow resin, which has typical handling characteristics for fluid composites, has a particle size of around 4.2 μm, larger than nanoparticulate composite resins such as Filtek bulk fill 0.004–0.1 μm. Large load particles are known to have rougher surfaces than smaller load particles[2,21].

Additionally, it is necessary to consider that the roughness values found in this study were above the clinically acceptable limit, 0.2 μm[28]. Clinically, smoother surfaces are less likely to accumulate biofilm. In this sense, it is important to note that no finishing and polishing was performed on the samples as the proximal wall of the restorations that are in contact with the metallic matrix was simulated. However, the matrix used in the study was composed of polyacetal that presents lower polishing than the metallic matrix (used clinically), this fact may have reflected in the roughness of the samples. The similarity of composition between the materials may be responsible for the similarities found in the photomicrographs performed before and after the acid challenges. In addition, it can be speculated that the similarities in the irregularities presented by the materials may reflect the polyacetal matrix used in the making of the specimens.

Performance with regards to microhardness and roughness of the bulk-fill flow composite resins tested in this study did not differ significantly from the nanoparticulate composite resin Z350 XT. Therefore, considering these physical properties, bulk-fill flow composite resins appear to be an attractive alternative for posterior restorations, mainly due to the simplification of the technique and lower clinical time[29].

CONCLUSION

The present study demonstrated that, after acid challenge, the bulk-fill flow composite resins showed changes similar to those presented by the nanoparticulate composite resin: reduced microhardness and increased surface roughness at the different depths evaluated.

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CONFLICTS OF INTERESTS

The authors declare no conflicts of interest.

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