

ORIGINAL RESEARCH

Effect of different industrialized acid beverages on the surface roughness of flowable composite resins: *in vitro* study

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Abstract

Flowable composite resins are materials available for restorations in pediatric dentistry. However, these materials are subject to dangerous effects in the oral environment caused by acids that deteriorate and increase their roughness. Therefore it is important to evaluate the effect of different industrialized acid beverages on the roughness of flowable composite resins. An *in vitro* experimental study, was done using a convenience sample of 132 discs of 5 mm diameter by 2 mm depth of four flowable materials (FF (Filtek™ Z350 XT Flowable), TNF (Tetric® N-Flow), PF (PermaFlo®) and GF (Grandio® Flow)) exposed to three beverages (CC (Coca-Cola), AJ (apple juice), and FM (fermented milk)) and incubated at 37 °C for 0, 15 and 30 days. The roughness (average roughness (Ra) and maximum height of profile (Rz) parameters) was measured at different intervals of time with a profilometer. For the data analysis, one-way analysis of variance (one-way ANOVA) and repeated measures analysis of variance (Repeated measures ANOVA) tests were applied ($p < 0.05$). In the roughness test before immersion, no differences were observed within the groups, with maximum roughness values for Filtek™ Z350 XT Flowable and minimum for PermaFlo®. However, at 15 and 30 days of immersion, the groups showed significant differences depending on the immersion drink, except Grandio® Flow in apple juice and fermented milk. The flowable materials studied presented specific behaviors according to the immersion period and drink used. The Filtek™ Z350 XT Flowable showed a similar increase in surface roughness independently of the drink used. Grandio® Flow was the most stable material against surface roughness changes after beverage immersion.

Keywords

Dental materials; Flowable composite resins; Industrialized acidic beverages; Surface roughness

1. Introduction

Composite resins were developed in the early 1960s as biomaterials with a reinforced polymer system used to replace the absence of dental structure. They also modify the color and contour of the teeth, thus improving the esthetics. Recently, nanocomposites [1], that enhance mechanical properties [1, 2] and clinical performance, have been incorporated into biomaterials. This continuous improvement has resulted in materials with excellent durability, resistance to wear, and superior esthetic properties [1].

Within these materials, the flowable composite resins (FCRs) have low viscosity, and small particle sizes [2, 3]. These properties allow them to be easily handled since they are dispensed with a syringe that has a needle tip [1]. Manufacturers constantly conduct laboratory tests to ensure

that dental materials meet standards for safety and efficacy. In pediatric dentistry, FCRs are indicated for restorations of classes I to V, minimally invasive restorations, sealing of fissures, bases, cavities linings, and crown fabrication [4].

Such materials in the oral environment are subject to deleterious effects caused by acids that originate from bacteria, food, and beverages. Moreover, drinks with hazardous chemicals can cause degradation of the surface of composite resin restorations and thus alter their morphology and surface roughness [5].

The consumption of acidic drinks has changed over time and has augmented worldwide in recent decades [6]. The increase is related to oral health problems such as enamel loss [7] and dental erosion [8] due to acid exposure [9, 10]. Constant interaction with acidic substances that exceed the body's defense system can cause the demineralization of hard

dental structures and alter the properties of dental materials [11]. According to previous reports, the surface roughness of dental materials increases and produces a lower resistance [11, 12].

The roughness increment in composite resins produces a more significant accumulation of bacteria on the surface of the restorations [13–15]. Therefore, they could provoke recurrent caries and a greater risk of developing periodontal diseases [16, 17].

Earlier studies [13, 18–20] have shown that exposure to specific beverages affects the roughness of composite resins [13, 21]. This depends on the type of drink, the period evaluated [22], the impact of dietary acids [21], and the characteristics of the materials [21, 22], such as the particle size [14].

However, there is a lack of studies that compare the effect of different industrialized acidic beverages on esthetic materials [13, 23, 24]. Moreover, little information exists on FCRs recommended for restorations in deciduous teeth, cervical lesions, and other small, low-or non-stress-bearing restorations [1]. Thus, it is necessary to evaluate the effect of different beverages on the roughness of FCRs. Therefore, the present work aims to assess *in vitro* the roughness of different FCRs exposed to some industrialized acidic drinks. We hypothesize (H_0) that the surface roughness of FCRs does not change after their immersion in selected industrialized acidic beverages.

2. Materials and methods

2.1 Materials selection

In vitro experimental study; the sequence of procedures and techniques applied in this work are shown in Fig. 1. Four different FCRs were selected for evaluation: FF (Filtek™ Z350 XT Flowable; 3M ESPE, Saint Paul, MN, USA), TNF (Tetric N Flow; Ivoclar Vivadent, Schaan, Liechtenstein), PF (PermaFlo®; Ultradent Products, South Jordan, UT, USA), and GF (Grandio® Flow; VOCO, Cuxhaven, Germany). Technical details of these FCRs, including their basic characteristics and composition (according to manufacturer), are shown in Table 1.

2.2 Specimen preparation

FCRs were placed in a prefabricated, 2 mm high Teflon mold with a 5 mm internal diameter. A piece of microscope glass slide was placed on top and pressed [24]. Then, the material was photopolymerized for 20 s with a third-generation light-emitting diode (LED) curing unit (Elipar™ DeepCure-L, 3M, Saint Paul, MN, USA) at a light intensity of 1470 mW/cm² with the light guide tip in direct contact with the upper glass slide. The intensity of the light source was checked every eight samples using the power checker included in the lamp's base. Next, each specimen was removed from the mold and placed in a labeled Eppendorf tube (Eppendorf® Safe-lock microcentrifuge tubes; Merck, Darmstadt, Germany) with 1.5 mL of deionized water. Immediately, they were stored in the incubator (RKI 19320, Ikemoto Scientific Technology, Tokyo, Japan) at 37 ± 2 °C for 24 hours. After that, irregularities were removed from the periphery of each disc using a strip of

sandpaper (1000-grit; Sof-Lex™ Finishing Strips, 3M ESPE, Saint Paul, MN, USA). Finally, the samples were rinsed and dried with compressed air (oil-free) for 10 s.

2.3 Experimental groups

One hundred and thirty-two discs comprised the sample, with 33 discs for each group (FF, TNF, PF and GF) of FCRs. They were randomly allocated into three subgroups (SGs (n = 11)); CC: Coca-Cola; AJ: apple juice and FM: fermented milk. The chemical composition of the industrialized acid beverages employed is described in Table 2.

2.4 Immersion in acidic drinks

The specimens were placed inside a labeled Eppendorf tube containing 1.5 mL of an industrialized acid beverage and stored in the incubator for fifteen and thirty days [24]. A digital pH meter (pH140 Conductronic, Puebla, Mexico) was used to measure the pH of each experimental drink. The vials were sealed to prevent evaporation of the solutions and the liquid content was renewed every 24 hours to avoid fungal contamination. Each Eppendorf tube was rinsed and dried before changing the beverage every day.

2.5 Surfaces roughness analysis

A profilometer (Surftest SJ-301, Mitutoyo, Tokyo, Japan) was used in this study to measure the surface roughness of each sample during three stages: at baseline (Roughness₀) and subsequently, after 15 days (Roughness₁) and 30 days (Roughness₂) of immersion in acidic industrialized beverages. Before conducting the analysis, samples were rinsed and dried with compressed air for 10 s.

The stylus tip of the profilometer was run transversely three times in the center of the specimen's exposed surface to evaluate the surface roughness. A length of 0.5 mm, a cut of 0.08 mm (λc), a velocity of 0.25 mm/s, and a Gaussian filter were used. The following roughness parameters were assessed: Ra (the average distance from the profile to the mean line over the length of assessment) and Rz (the peak-to-valley values of five equal measures within the profile) under International Organization for Standardization (ISO) ISO 4287–1997 [25]. Finally, the average values were calculated for each sample, and each group [24].

2.6 SEM evaluation

One randomly chosen specimen of each FCR was evaluated with a scanning electron microscope (JEOL, JSM-6610 LV, Tokyo, Japan) at every stage of the experiment (baseline (0 days), after 15 and 30 days of immersion). The samples were fixed to aluminum stubs using double-sided adhesive carbon tape (SPI supplies, USA). The micrographs were obtained from gold uncoated FCRs using a scanning electron microscope (JEOL, JSM-6610 LV, Japan) at low vacuum mode, detecting back-scattered electrons, a 35 Pa chamber pressure, an operating voltage of 15 kV, and an ×1200 magnification to verify any alterations in the microstructure.

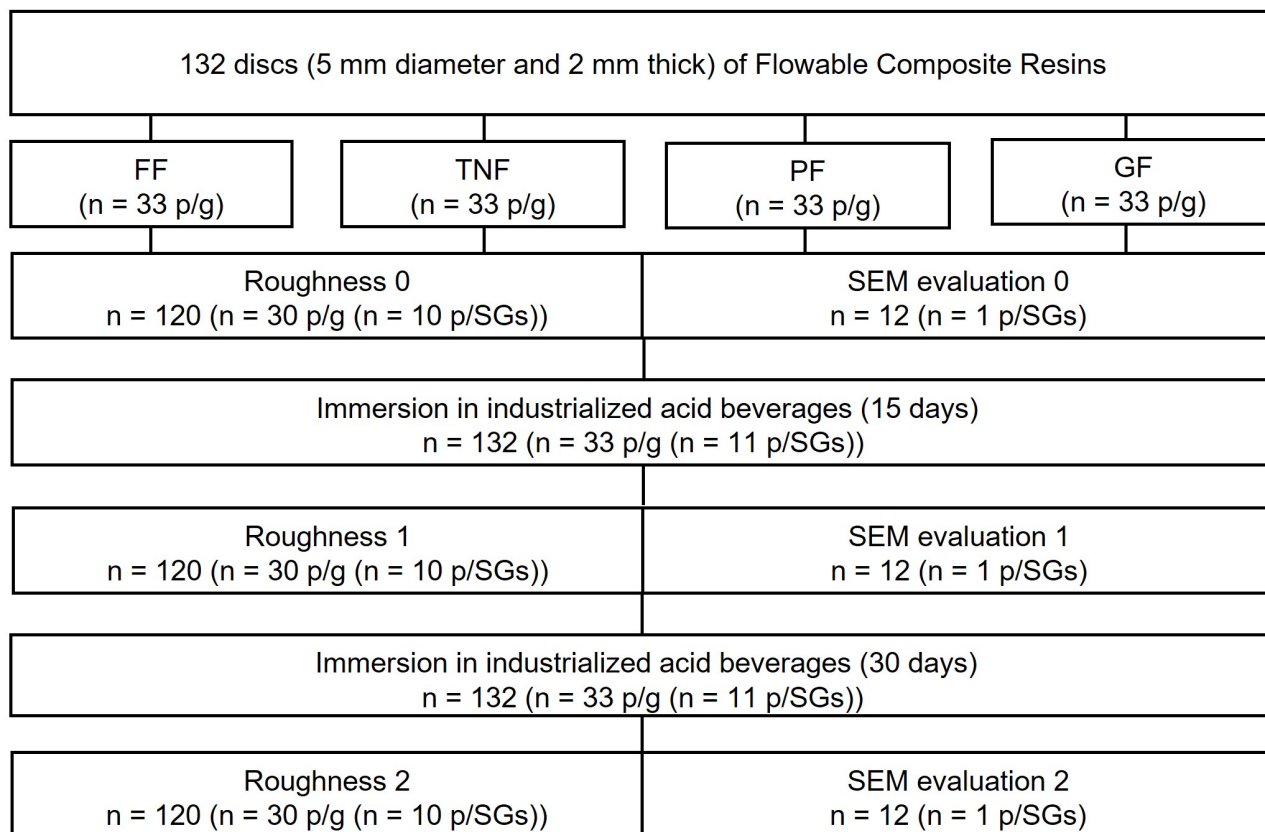


FIGURE 1. Diagram of the parameters and techniques. FF: Filtek™ Z350 XT Flowable; TNF: Tetric® N-Flow; PF: PermaFlo®; GF: Grandio® Flow; SEM: scanning electron microscopy.

TABLE 1. FCRs characteristics and composition.

Code	Name of the FCRs	Main components	Particle size	Inorganic filler wt%/vol%
FF	Filtek™ Z350 XT Flowable (3M ESPE, Saint Paul, MN, USA)	Nanofiller Bis-GMA, TEGDMA, Bis-EMA, non-agglomerated/nonaggregated silica nanofiller and zirconia nanofiller and nanocluster of agglomerated polyethylene zirconia/silica	0.6–1.4 μm	65%/46%
TNF	Tetric® N-Flow (Ivoclar Vivadent; Schaan, Liechtenstein)	Nanohybrid Bis-GMA, UDMA, TEGDMA Barium glass, ytterbium fluoride, silica	0.04–3 μm	63%/39%
PF	PermaFlo® (Ultradent Products, South Jordan, UT, USA)	Nanofiller dimethacrylates, methacrylates	1 μm	68%/NC
GF	Grandio® Flow (VOCO, Cuxhaven, Germany)	Nanohybrid Bis-GMA, TEGDMA, HEDMA, glass ceramic, nanoparticle	0.02–0.04 to 1 μm	80%/65.6%

FF: Filtek™ Z350 XT Flowable; TNF: Tetric® N-Flow; PF: PermaFlo®; GF: Grandio® Flow; Bis-GMA: bisphenol A-glycidyl methacrylate; TEGDMA: triethylene glycol dimethacrylate; HEDMA: 1,6-Hexanediol dimethacrylate; UDMA: urethane dimethacrylate; Bis-EMA: bisphenol a polyethylene glycol diether dimethacrylate; wt%: percentage of filler by weight; vol%: percentage of filler by volume; NC: information not collected.

TABLE 2. Composition of the industrialized acid beverages used.

Code	Industrialized acid beverages (brand name)	Producer	Composition	pH of beverages (mean)
CC	Coca-Cola	Coca-Cola, Atlanta, GA, USA	Water, sugar, carbon dioxide, colorant (caramel, E150d), phosphoric acid, natural flavors, and caffeine	2.71
AJ	Apple juice	Grupo Jumex, Mexico City, Mexico	Water, concentrated apple juice, citric acid	3.36
FM	Fermented milk	Yakult Honsha, Tokyo, Japan	Water, skimmed milk, glucose and fructose syrup, sugar, maltodextrin, and flavorings	3.80

CC: Coca-Cola; AJ: apple juice; FM: fermented milk.

2.7 Statistical analysis

The surface roughness values for baseline and post immersion in industrialized acid beverages were analyzed using a statistical package (SPSS, 25.0; IBM, Armonk, NY, USA). First, the data distribution was evaluated with the Shapiro-Wilk test; then, differences between materials' roughness were assessed using the one-way ANOVA test. Finally, a repeated measures ANOVA test was performed to compare the surface roughness changes through the three experimental stages. A $p < 0.05$ significance threshold was used.

3. Results

The general average and standard deviation (regardless of the industrialized acidic beverage used) for Ra and Rz parameters (μm) by material evaluated at different experimental stages are shown in Table 3. The FF group obtained the highest roughness values among groups at baseline and at the end of each immersion period ($Ra_0 = 0.019 \pm 0.002$, $Ra_1 = 0.024 \pm 0.003$ and $Ra_2 = 0.027 \pm 0.003$) ($p < 0.05$). At the same time, the PF group showed the lowest basal roughness ($Ra_0 = 0.011 \pm 0.001$) ($p < 0.05$), and after immersion in industrialized acidic beverages ($Ra_1 = 0.013 \pm 0.002$ and $Ra_2 = 0.015 \pm 0.001$) ($p < 0.001$). All groups presented significant increases in their roughness surface throughout the experiment ($p < 0.05$).

The results obtained for the Ra and Rz roughness parameters by material type and beverage are shown in Table 4. There are no statistically significant differences for both parameters in the basal roughness (0) measurements when comparing each group ($p > 0.05$). However, all groups showed significant differences for Ra_1 and Ra_2 according to the type of material and drink in both roughness parameters.

The FF, TNF and PF groups presented an increase in surface roughness values ($p < 0.001$) when immersed in CC. AJ and FM beverages at 15 and 30 days ($p < 0.001$). The group GF only had changes in roughness when submerged in the CC beverage for both experimental periods ($p < 0.001$).

GF did not present changes when immersed in AJ and FM beverages after 15 days (Ra: 0.016 to 0.018 μm and Rz: 0.100 to 0.106 μm); and at 30 days (Ra: 0.017 to 0.018 and Rz: 0.100 to 0.107 μm).

The scanning electron microscopy (SEM) micrographs of

the FCRs taken before (0 days) and after immersion in acidic beverages (15 and 30 days) are shown in Fig. 2. The baseline SEM images (0 days) showed irregular surfaces, with homogeneous dispersion of the filler particles in the polymeric matrix; the size, shape, and distribution of the particles were characteristic for each material. The largest particles were observed in the FF group; the TNF, GF and PF groups presented smaller particles.

In the SEM images taken after immersion, a superficial degradation of the materials with exposure or loss of filler particles is observed; these findings are more evident after 30 days. The FF and TNF groups presented the most irregular surface after exposure to industrialized acid drinks. All groups presented more irregular and degraded surfaces when exposed to CC drink; the FF, TNF and PF groups were only affected by the FM beverage.

4. Discussion

The following study aimed to evaluate the surface roughness of some commercial brands of FCRs before and after immersion in acidic industrialized beverages commonly consumed by children (a soft drink, fruit juice, and fermented milk). Different periods (baseline, fifteen, and thirty days) were evaluated to see the relationship between time and surface degradation. It is known that the acidic nature and sugar content of these drinks increase the risk of tooth decay and dental erosion in toddlers [26]. Additionally, four FCRs were selected for the analysis since the demand for this type of restoration materials has increased due to their aesthetic properties and their effectiveness in sealing pits and fissures of subsuperficial occlusal lesions [27]. The resins studied have different compositions in the organic matrix and the type of filler [28–31]. They were selected to determine if there is any relationship between these characteristics and the increase in roughness. The acidic beverages and the time protocol used in this research were based on previous studies [20, 24].

According to the results of this investigation, all materials became significantly rougher after they were subjected to the immersion regimen. That can be ascribed to the capacity of acid media to soften resin-based restorative materials [18, 21, 32]. A weakness of resin-based materials is the interface between the resin and filler particles which has a high sensitivity to water sorption, due to its susceptibility to

TABLE 3. General average and standard deviation of the Ra and Rz parameters (μm) at 0, 15 and 30 days of FCRs immersion in some industrialized acidic beverages.

Parameter	Group	Surface roughness			<i>p</i>
		0 day (R_0)	15 days (R_1)	30 days (R_2)	
Ra					
	FF	$0.019 \pm 0.002^{A,a}$	$0.024 \pm 0.003^{A,b}$	$0.027 \pm 0.003^{A,c}$	$p < 0.001$
	TNF	$0.017 \pm 0.002^{B,a}$	$0.023 \pm 0.002^{B,b}$	$0.025 \pm 0.002^{B,c}$	$p < 0.001$
	PF	$0.011 \pm 0.001^{C,a}$	$0.013 \pm 0.002^{C,b}$	$0.015 \pm 0.001^{C,c}$	$p < 0.001$
	GF	$0.016 \pm 0.002^{D,a}$	$0.016 \pm 0.002^{D,a}$	$0.019 \pm 0.004^{D,b}$	$p < 0.001$
	<i>p</i>	$p = 0.032$	$p < 0.001$	$p < 0.001$	
Rz					
	FF	$0.117 \pm 0.012^{A,a}$	$0.150 \pm 0.014^{A,b}$	$0.160 \pm 0.014^{A,c}$	$p < 0.001$
	TNF	$0.109 \pm 0.012^{B,a}$	$0.142 \pm 0.011^{B,b}$	$0.154 \pm 0.012^{B,c}$	$p < 0.001$
	PF	$0.072 \pm 0.008^{C,a}$	$0.083 \pm 0.009^{C,b}$	$0.100 \pm 0.009^{C,c}$	$p < 0.001$
	GF	$0.106 \pm 0.012^{D,a}$	$0.105 \pm 0.019^{D,a}$	$0.115 \pm 0.024^{D,b}$	$p < 0.001$
	<i>p</i>	$p < 0.001$	$p < 0.001$	$p = 0.019$	

Statistical analysis for each roughness parameter (Ra or Rz): the capital letters in the columns represent the comparison between the materials on the same immersion stage. Lowercase letters in a row compare the parameters at different immersion stages. Identical letters indicate that there are no statistical differences. FF: Filtek™ Z350 XT Flowable; TNF: Tetric® N-Flow; PF: PermaFlo®; GF: Grandio® Flow; *p*: *p*-value.

hydrolytic degradation [33, 34]. Due to the general results described above, the null hypothesis (H_0) of the present study was rejected and the alternative hypothesis (H_1) accepted.

Concerning the results found in this study, all FCRs presented different roughness values before the beverage immersion since the resins studied have different compositions in the organic matrix and the percentage and type of filler [28–31]. It is known that the surface roughness is related to the organic matrix structure (the monomer type) and the inorganic filler (type, size, shape, and distribution) [19].

The PF and GF groups present tiny filler particles (0.04–3 μm , 0.02–0.04 and 1 μm); in contrast, TNF and FF groups have higher filler particles (0.6–1.4 μm , 0.04–3 μm respectively); hence, PF group had the lowest roughness value, followed by GF group. The one with the highest roughness value was the FF group. The results are in line with the literature, low baseline surface roughness values were found in resin based restorative materials (composite resin, giomer, flowable composite resin compomer and resin modified glass ionomer cements (RMGIC)) with small particle sizes but higher in restorative materials with large particle sizes [22]. All the materials evaluated could be classified as nanohybrids due to the size range of their particles, even though filtek resin is described in the literature as a nanofiller because it has nanofiller particles [20, 34].

The samples of each material showed homogeneous roughness values before the immersion period. This homogeneity is an essential requirement for appropriate comparisons. FF group presented the most significant increase in roughness values at 15 and 30 days, with no difference between the industrialized acid beverages used during the experiment. These results may be explained by the highest baseline average surface roughness of Filtek™ Z350 XT Flowable when compared

with other materials and by the higher content of nanoagglomerates/nanoaggregates and nanoclusters of silica and zirconia [32].

Resin-based materials such as composites with large particles show more surface roughness after beverage immersion, according to Reddy *et al.* [20]. Additionally, surface roughness usually increases after immersion in drinks. This increment is a consequence of water presence that infiltrates and modifies the mechanical properties of the polymer matrix by expanding and reducing the friction between the polymer chains. Such is the case when TEGDMA is incorporated which causes increased water absorption on Bis-GMA in resins-based materials [23, 35].

In TNF and PF groups the surface roughness also increased over time in CC, AJ and FM beverages. This could be due to the presence of nanofillers in the composition and the low pH of the drinks evaluated. It is known that nanofiller particles contained in resin composites give a high solubility and water absorption to the materials, making them more prone to ion leaching and hydrolysis of the coupling agent. The former phenomena produces molecular loss by separating the solutes from the matrix solid [33, 34]. Resins contain a percentage of an organic matrix, which is related to the increase in water absorption and disintegration in an aqueous environment [23]. With respect to pH, it plays an essential role in the stability of surface roughness of composite resins [36]. The literature points out that industrialized drinks with low pH (pH of 2.85 and 3.49) have a positive correlation with the roughness and solubility of dental materials [37]. The CC, AJ and FM beverages present low pH in the range of 2.71 to 3.80. Hence, all FCRs were affected.

It has been previously shown that the GF group was the only fluid resin that presented a different behavior compared to the

TABLE 4. Mean and standard deviation of the roughness parameters Ra and Rz (μm) of FCRs at 0, 15 and 30 days of immersion in some industrialized acidic beverages.

Parameter	Groups	Surface roughness			<i>p</i>	
		0 day (R_0)	15 days (R_1)	30 days (R_2)		
Ra	FF	FF_CC	$0.018 \pm 0.002^{A,a}$	$0.026 \pm 0.003^{A,b}$	$0.028 \pm 0.003^{A,c}$	$p < 0.001$
		FF_AJ	$0.018 \pm 0.002^{A,a}$	$0.023 \pm 0.003^{A,b}$	$0.026 \pm 0.003^{A,c}$	$p < 0.001$
		FF_FM	$0.019 \pm 0.002^{A,a}$	$0.025 \pm 0.002^{A,b}$	$0.026 \pm 0.002^{A,c}$	$p < 0.001$
		<i>p</i>	$p = 1.000$	$p = 0.383$	$p = 0.973$	
	TNF	TNF_CC	$0.018 \pm 0.002^{A,a}$	$0.023 \pm 0.002^{A,b}$	$0.025 \pm 0.002^{A,c}$	$p < 0.001$
		TNF_AJ	$0.017 \pm 0.002^{A,a}$	$0.022 \pm 0.001^{B,b}$	$0.023 \pm 0.002^{B,c}$	$p < 0.001$
		TNF_FM	$0.017 \pm 0.001^{A,a}$	$0.023 \pm 0.002^{C,b}$	$0.026 \pm 0.002^{C,c}$	$p < 0.001$
		<i>p</i>	$p = 0.521$	$p = 0.044$	$p = 0.003$	
	PF	PF_CC	$0.011 \pm 0.001^{A,a}$	$0.012 \pm 0.001^{A,b}$	$0.015 \pm 0.001^{A,c}$	$p < 0.001$
		PF_AJ	$0.011 \pm 0.001^{A,a}$	$0.013 \pm 0.002^{B,b}$	$0.015 \pm 0.002^{B,c}$	$p < 0.001$
		PF_FM	$0.011 \pm 0.001^{A,a}$	$0.013 \pm 0.001^{C,b}$	$0.015 \pm 0.002^{C,c}$	$p < 0.001$
		<i>p</i>	$p = 0.997$	$p = 0.021$	$p < 0.001$	
	GF	GF_CC	$0.017 \pm 0.002^{A,a}$	$0.018 \pm 0.002^{A,b}$	$0.023 \pm 0.002^{A,c}$	$p < 0.001$
		GF_AJ	$0.016 \pm 0.002^{A,a}$	$0.018 \pm 0.003^{A,a}$	$0.018 \pm 0.003^{B,a}$	$p = 0.405$
		GF_FM	$0.016 \pm 0.002^{A,a}$	$0.016 \pm 0.002^{B,a}$	$0.017 \pm 0.001^{B,a}$	$p = 0.168$
		<i>p</i>	$p = 1.000$	$p < 0.001$	$p < 0.001$	
Rz	FF	FF_CC	$0.118 \pm 0.011^{A,a}$	$0.156 \pm 0.014^{A,b}$	$0.166 \pm 0.014^{A,c}$	$p < 0.001$
		FF_AJ	$0.115 \pm 0.012^{A,a}$	$0.148 \pm 0.015^{B,b}$	$0.155 \pm 0.015^{B,c}$	$p < 0.001$
		FF_FM	$0.119 \pm 0.013^{A,a}$	$0.147 \pm 0.010^{B,b}$	$0.158 \pm 0.010^{B,c}$	$p < 0.001$
		<i>p</i>	$p = 0.735$	$p = 0.003$	$p = 0.003$	
	TNF	TNF_CC	$0.109 \pm 0.019^{A,a}$	$0.142 \pm 0.009^{A,b}$	$0.153 \pm 0.011^{A,c}$	$p < 0.001$
		TNF_AJ	$0.109 \pm 0.008^{A,a}$	$0.139 \pm 0.013^{B,b}$	$0.152 \pm 0.012^{B,c}$	$p < 0.001$
		TNF_FM	$0.108 \pm 0.007^{A,a}$	$0.144 \pm 0.010^{C,b}$	$0.157 \pm 0.013^{C,c}$	$p < 0.001$
		<i>p</i>	$p = 1.000$	$p = 0.028$	$p = 0.040$	
	PF	PF_CC	$0.071 \pm 0.010^{A,a}$	$0.080 \pm 0.008^{A,b}$	$0.101 \pm 0.011^{A,c}$	$p < 0.001$
		PF_AJ	$0.074 \pm 0.009^{A,a}$	$0.084 \pm 0.011^{B,b}$	$0.100 \pm 0.008^{B,c}$	$p < 0.001$
		PF_FM	$0.071 \pm 0.005^{A,a}$	$0.085 \pm 0.007^{C,b}$	$0.100 \pm 0.008^{C,c}$	$p < 0.001$
		<i>p</i>	$p = 0.607$	$p = 0.003$	$p < 0.001$	
GF	GF_CC	$0.107 \pm 0.009^{A,a}$	$0.121 \pm 0.018^{A,b}$	$0.145 \pm 0.012^{A,c}$	$p < 0.001$	
	GF_AJ	$0.104 \pm 0.014^{A,a}$	$0.106 \pm 0.017^{B,a}$	$0.107 \pm 0.015^{B,a}$	$p = 0.608$	
	GF_FM	$0.106 \pm 0.013^{A,a}$	$0.100 \pm 0.016^{B,a}$	$0.100 \pm 0.010^{B,a}$	$p = 0.318$	
	<i>p</i>	$p = 0.890$	$p < 0.001$	$p < 0.001$		

Capital letters in a column represent the comparison between roughness values (Ra or Rz) of the same material immersed in different industrialized acidic beverages. Lowercase letters in a row compare the parameters at different immersion stages. Identical letters indicate that there are no statistical differences $p \leq 0.05$.

FF: Filtek™ Z350 XT Flowable; TNF: Tetric® N-Flow; PF: PermaFlo®; GF: Grandio® Flow; CC: Coca-Cola; AJ: apple juice; FM: fermented milk; FF_CC: Filtek™ Z350 XT Flowable-Coca-Cola; FF_AJ: Filtek™ Z350 XT Flowable-Apple juice; FF_FM: Filtek™ Z350 XT Flowable-Fermented milk; TNF_CC: Tetric® N-Flow-Coca-Cola; TNF_AJ: Tetric® N-Flow-Apple juice; TNF_FM: Tetric® N-Flow-Fermented milk; PF_CC: PermaFlo®-Coca-Cola; PF_AJ: PermaFlo®-Apple juice; PF_FM: PermaFlo®-Fermented milk; GF_CC: Grandio® Flow-Coca-Cola; GF_AJ: Grandio® Flow-Apple juice; GF_FM: Grandio® Flow-Fermented milk.

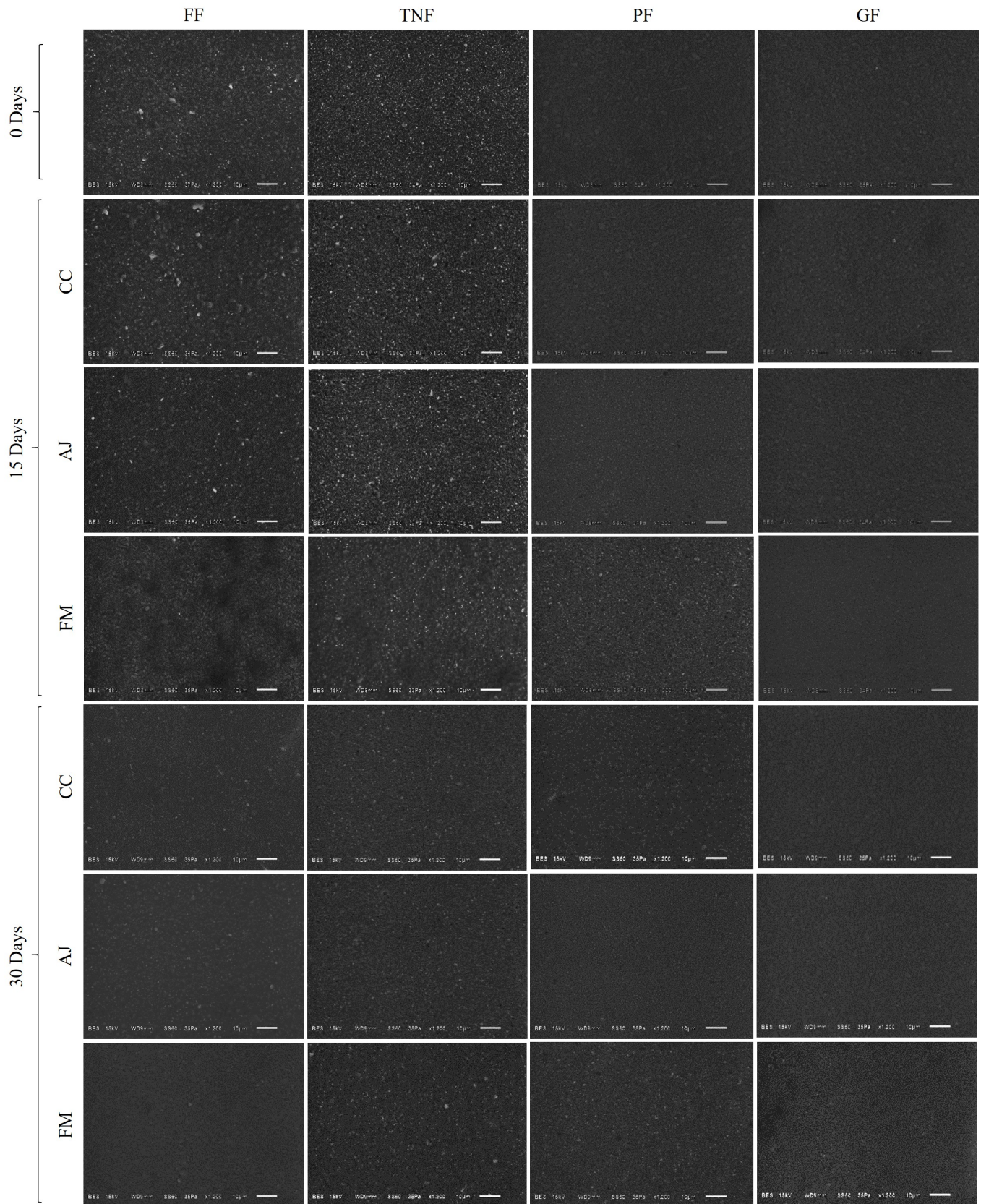


FIGURE 2. Representative SEM images of FCRs surfaces before and after immersion in acidic industrialized beverages. (Original magnification $\times 1200$); scale bar = 10 μm . FF: Filtek™ Z350 XT Flowable; TNF: Tetric® N-Flow; PF: PermaFlo®; CC: Coca-Cola; AJ: Apple juice; FM: fermented milk.

other materials studied. It showed changes in its surface roughness when embedded in CC beverage. Coca-Cola drinks cause surface degradation of the resin matrix and surface erosion of the filler content. The latter is a consequence of the former and varies according to the content and distribution of fillers and the composition of the matrix resin [13, 20, 24]. Nanohybrid-containing resins exposed to acids are expected to undergo considerable degradation [13, 22]. Another reason is that Coca-Cola contains phosphoric acid (to impart a tangy flavor), orthophosphoric acid (as an acidity regulator), carbonic acids, colorings, flavorings, and caffeine [13, 24]. It has inherent acidity (pH 2.71), due to the presence of acid components, that erodes the surface of filler contents and enhances surface degradation of the resin matrix [20], thus altering the surface of the GF group.

The roughness stability of the GF group during immersion in AJ and FM beverages may be due to the higher filler content present in its composition (80%). According to the literature, the higher the filler content, the less water absorption, which leads to less surface degradation [35]. Moreover, fruits juice and fermented milk cause less surface roughness than Coca-Cola in other resin-based materials [20, 24]. Additionally, fermented milk called Yakult is considered a non-erosive beverage in various tooth-colored restorative materials (Glass Ionomer Cement, Composite, and Compomer) [38].

In general, all the FCRs analyzed changed their surface roughness and presented different behavior patterns related to the commercial brand and the acid drink used. This could be related to the differences in the composition of each material studied (comprised of organic and inorganic compounds) since this influences its behavior against acid attack, according to Tărăboanță *et al.* [39]. On the other hand, the alterations produced by different beverages depend on the characteristics of the materials, the type of beverage, and the period evaluated. Generally, a prolonged immersion period makes a more significant impact on the properties of the resin, such as the surface roughness [23, 24]. The results of this study revealed that the initial roughness could be a determining factor for the final roughness.

As reported by Guler and Unal [22], SEM qualitative results were consistent with the surfaces' roughness. SEM analysis provides detailed information on surface roughness and supports profilometry results; however, because it is an expensive method that consumes a lot of time to carry out and due to the high difficulty to find the same area in repeated measurements, it was only carried out in a single sample for each group of material and drink. The samples were not covered with gold to guarantee their direct contact with acidic beverages during the stages of the experiment, and consequently to avoid biased results. For this purpose, a low vacuum microscope was used and the operating parameters were modified according to the study of non-conductive specimens (dental enamel) subjected to sequential studies [40].

All the intact FCRs showed irregular surfaces with specific characteristics related to their chemical composition, size and percentage of filler particles. After exposure of all FCRs in industrialized acid beverages, their surfaces were affected depending on the time and beverage used. Matrix resin decomposition by deformation of polymerized filler structures,

dissolution of polymers, removal or exposure of fillers at different levels as were observed by Guler *et al.* [22], are phenomena that explains the changes in surface roughness in the FCRs.

Some studies report that the roughness of the surface is the critical factor for the formation of Biofilm and is consequently responsible for the increased risk of caries around restorations [13, 14]. Although the fluid resins showed increased surface roughness values after exposure to acidic industrialized beverages, these did not exceed the value considered as a threshold that favors bacterial adherence ($0.20 \mu\text{m}$). However, the roughness threshold value is a controversial issue in the literature, other reports have found no appreciable differences in plaque on surfaces with Ra values that ranged from 0.07 to $1.4 \mu\text{m}$ [41]. The resin composite surfaces evaluated may be considered to have demonstrated a smooth surface, from the clinical point of view, which presents no risk of plaque accumulation.

Finally, the results obtained are difficult to compare with those reported in previous studies due to the difference in parameters and protocols used, and the scarce information available on the behavior of FCRs subjected to immersion in acidic beverages. Therefore, the results of this *in vitro* study must be interpreted with a certain degree of caution.

One limitation of this study was that the conditions of the oral environment could not be replicated precisely. Saliva can modulate the pH of some drinks due to its buffering effect; its flow can be stimulated by some drinks and thus counteract their harmful effects on the studied materials. Therefore, it is recommended to carry out *in situ* studies that evaluate the effects of beverages on the clinical performance of dental materials.

5. Conclusions

Within the limitations of this study, the following conclusion were made:

- The immersion in industrialized acid beverages of the evaluated materials, except for the GF group, produced changes in the roughness of their surface. Pronounced changes were observed the longer the material was exposed to the beverage.
- The type of drink did not influence the increase in the average roughness of the FF group, only the immersion time.
- TNF group did present greater changes in its surface roughness under immersion in FM beverage.
- PF group showed more pronounced affections by AJ and FM beverages in the Ra and Rz parameters, respectively.
- GF group was the flowable resin most resistant to changes in surface roughness caused by immersion in acidic beverages, only increasing under the influence of CC beverage.

AVAILABILITY OF DATA AND MATERIALS

Not applicable.

AUTHOR CONTRIBUTIONS

LAM and LERV—designed the research study. LAM—performed the research. LERV and MAMB—analyzed the data. LAM, RCB and BTC—wrote the manuscript. All authors contributed to editorial changes in the manuscript. All authors read and approved the final manuscript.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

The study was conducted following the Declaration of Helsinki and its later amendments or comparable ethical standards and approved by the Research Ethics Committee at the Dental Research and Advances Studies Center, School of Dentistry at the Autonomous University of the State of Mexico (CEICIEAO-2020-020); date of approval 19 March 2021.

ACKNOWLEDGMENT

To Emilio Ramírez-Rodríguez for his support in editing the text.

FUNDING

This research received no external funding.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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How to cite this article: Lorena Albarran-Martínez, Laura Emma Rodríguez-Vilchis, Rosalía Contreras-Bulnes, María de los Angeles Moyaho-Bernal, Bernardo Teutle-Coyotecatl. Effect of different industrialized acid beverages on the surface roughness of flowable composite resins: *in vitro* study. *Journal of Clinical Pediatric Dentistry*. 2023; 47(5): 152-161. doi: 10.22514/jocpd.2023.065.