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Original Research

Effect of hydroelectrolytic beverages on the roughness and microhardness of bulk fill resin composites

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Aim: This study aimed to assess the effects of a hydroelectrolytic beverage on the surface roughness and microhardness of different bulk fill resin composites. **Methods:** Sixty resin composite samples were prepared and divided into three groups: FiltekTM Z350 (C), FiltekTM One Bulk Fill (BF-F), and Opus Bulk Fill APS (BF-O). These were further subdivided to analyze two beverage groups: GatoradeTM Lemon (GL) and distilled water (control). Surface roughness (Ra), surface microhardness (KHN), and scanning electron microscopy (SEM) results were evaluated (n=10). The data were subjected to two-way ANOVA (for roughness) or three-way ANOVA (for microhardness), followed by Tukey's post hoc test (α=0.05). **Results:** The C group exhibited higher microhardness than the BF-F and BF-O groups, regardless of the solution used. A reduction in microhardness was observed in all groups when comparing initial and final time points. SEM images revealed variations in the size, amount, and arrangement of particles among the different resin composites. **Conclusion:** The study concluded that the microhardness of different bulk fill resin composites decreases when exposed to either the hydroelectrolytic beverage or distilled water. However, the type of immersion liquid did not influence the surface roughness of the evaluated composite resins.

Keywords: Composite resins. Isotonic solutions. Hardness tests.

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Introduction

The consumption of hydroelectrolytic beverages has increased significantly in recent years, especially during physical activities. Although these beverages are intended to replenish electrolytes and hydration, studies have shown that frequent consumption can cause changes in tooth structure and restorative materials in the oral cavity[1,2]. Many of these beverages have a low pH, often below 5.5, which can lead to tooth enamel demineralization and affect the properties of resin composites[3].

Resin composites are commonly used for direct restorations in dentistry. However, their surface hardness and roughness can be influenced by acidic foods and beverages, which frequently come into contact with these materials[3,4]. Therefore, the impact of pH on these composites needs continuous investigation. A decrease in composite hardness can lead to material fatigue and compromise the restoration[1]. On the other hand, an increase in surface roughness can promote greater adhesion of bacterial biofilm[5], leading to gingival disease, marginal leakage of restorations, and caries lesions.

The quest for restorative materials with excellent clinical performance and ease of use has led to the development of various types of commercially available bulk fill resin composites. These composites are classified based on their consistency as either fluid or regular. Their low degree of polymerization shrinkage allows them to be placed in 4-5 mm increments, optimizing clinical work time[6]. Different monomers have been incorporated into these composites to modulate the polymerization reaction, enabling stress control and providing strong adhesion between the restorative material and the tooth structure[7,8]. However, these modifications may cause the resins to respond differently when exposed to various types of beverages.

Hydrolytic beverages have been found to affect color stability[9,10], surface hardness, and roughness of resin composites due to their composition, among other factors. Given the increasing consumption of hydroelectrolytic beverages and the constant evolution of new restorative materials, this study aimed to perform an in vitro evaluation of the effects of a hydroelectrolytic beverage on the surface of different types of bulk fill resin composites. The hypotheses tested are that different resin composites would show different [i] microhardness and [ii] roughness values after immersion in a hydroelectrolytic beverage.

Materials and method

Experimental Design

The factors researched in this in vitro study (3x2 factorial design) were resin composite at 3 levels: Filtek™ Z350XT (C) (3M, Sumaré, São Paulo, Brazil), Filtek™ One Bulk Fill (BF-F) (3M, Sumaré, São Paulo, Brazil), and Opus Bulk Fill (BF-O) (FGM Santa Catarina, Brazil), further subdivided into 2 levels of beverages: Gatorade™ Lemon Lime (Gatorade Co., AMBEV) and distilled water (control). Twenty samples were fabricated for each resin composite brand and divided according to the beverage tested (n=10). The response variables were surface microhardness (KHN) and surface roughness (Ra). Additionally, qualitative analyses using scanning electron microscopy were performed on all groups.

Samples preparation

The samples were prepared using a standardized method, employing a matrix with a 6 mm diameter and 4 mm thickness to fabricate 60 resin composite discs, shade A2. The bulk fill resins were inserted in a single increment of 4 mm, while the conventional resin was inserted in increments of 2 mm, using a dual titanium spatula (Millennium, Golgan, SP, Brazil). A polyester strip and a glass plate with a 500 g load were placed on the resin composite surface for 30 seconds to remove excess material and ensure a smooth, standardized surface. Subsequently, the samples were polymerized for 20 seconds and light-activated with a light-polymerizing device at 1000 mW/cm² standard power (Valo, Ultradent Products, South Jordan, USA).

The samples were finished and polished using abrasive discs #600, #1200, and #4000 (Norton Abrasivos, Vinhedo, SP, Brazil), polishing pastes, and a felt polisher (Arotec, São Paulo, Brazil)[11]. The specimens were cleaned in an ultrasound bath (Cristófoli, Campo Mourão, PR, Brazil) for 5 minutes between each stage and after polishing to remove surface debris.

Each resin composite tested was divided into two groups according to the beverage used (n=10): Gatorade or distilled water (control).

Detailed information on the resin composites and materials used is provided in Tables 1 and 2.

Table 1. Presentation of the resin composites researched in the study (Filtek Z350XT, Filtek One Bulk Fill and Opus Bulk Fill)

Resin	Manufacturer	Matrix	Inorganic filler
FILTEK™ Z350XT	3M	Bis-GMA UDMA Bis-EMA TEGDMA	Nanoparticulate Silica 20nm Zirconia 5 nm 78.5% weight
FILTEK™ ONE BULK FILL	3M	UDMA DDDMA AUDMA AFM	Nanoparticulate Silica 20nm Zirconia 4 to 11nm Ytterbium trifluoride 100nm 76.5% weight 58.5% volume
OPUS BULK FILL	FGM	Urethane dimethacrylate	Nanoparticulate Special System of Initiators (APS) Filler of silanized silicon oxide, barium aluminosilicate (BAS) glass 10µm, 2µm, 1µm and 0.7µm 79% weight

Table 2. Beverages used in the study and their compositions

Beverage	Composition	Manufacturer
Gatorade Lemon	Sucrose Syrup, Glucose Syrup, Fructose, Citric Acid, Natural Flavors, Sodium Chloride Salt, Monopotassium Phosphate Ester Gum, Yellow Flavors	The Gatorade Co., Chicago, IL
Distilled water	--------------------------------	-------------------

Sample Immersion in Beverages

Each resin composite sample was individually immersed in 20 mL of either a hydro-electrolytic fluid-replenishment solution (Gatorade group) or distilled water (control group) for 5 minutes, three times a day, over a period of 15 days[2]. After each immersion, the samples were rinsed with distilled water and then stored in an incubator at 37°C.

Surface Roughness Analysis

Surface roughness analysis was performed with a rugosimeter (SJ 301, Mitutoyo, Kanagawa, Japan) after 15 days of sample immersion in the beverages. The surface of each sample was analyzed using a sampling length of 1.25 mm and a cut-off of 0.25 mm (λc) for the measurements. This analysis was performed in triplicate for each specimen, and the mean of the three measurements was used to determine the surface roughness in Ra (µm) for each specimen.

Surface Microhardness

Surface microhardness was analyzed before and after 15 days of contact with the beverages using a Knoop penetrator with a load of 25g and time of 5s, in a microdurometer (HMV-2000, Shimadzu, Tokyo, Japan). Three indentations were made in each sample, at intervals of 100 µm, and the mean was calculated to determine the Knoop hardness (KHN).

Scanning Electronic Microscopy (SEM)

Three specimens from each group were analyzed using SEM. Initially, they were placed in an ultrasound bath, followed by immersion in 100% alcohol for 15 minutes. Afterwards, the specimens were mounted on stubs and sputter-coated with a layer of gold-palladium alloy using a vacuum sputter coater. The most representative area of each group was photographed at 3000x magnification.

Statistical Analysis

The surface roughness and microhardness data were assessed for normality with the Shapiro-Wilk test, and for homogeneity of variance, with the Levene test. The results were submitted to two-way (roughness) or three-way (microhardness) ANOVA, followed by Tukey's *post hoc* test with a preset alpha of α=0.05. All the statistical analyses were carried out using SPSS for Windows software (IBM SPSS Statistics 21).

Results

In terms of roughness analysis (Table 3), the group C exhibited higher roughness values compared to the bulk fill resins after immersion in distilled water. However, there was no significant difference compared to BF-O resin after immersion in Gatorade. When each resin was evaluated separately, no differences in surface roughness were observed between the two immersion liquids (Gatorade or distilled water).

Table 3. Mean (±SD) roughness according to each resin composite and beverage.

	(BF-O)	(BF-F)	(C)
Gatorade	0.080 (0.038)Aab	0.058 (0.013)Ab	0.124 (0.052)Aa
Distilled water	0.069 (0.034)Ab	0.084 (0.049)Ab	0.139 (0.062)Aa

Uppercases letters in columns compare beverages. Lowercase letters in rows compare resins.

The results of the surface microhardness analysis are presented in Table 4. There were no statistically significant differences found between the types of beverages concerning the resin composites at any of the evaluated time periods ($\rho > 0.05$). The group C exhibited higher microhardness than the BF-F or BF-O resin composite groups at both the initial and final time periods. All resin composites showed statistically significant differences across the time periods, indicating lower values were observed at the final time point, regardless of the type of beverage.

Table 4. Mean (±SD) microhardness, according to the resin composites, beverages, and time periods evaluated.

		(BF-O)	(BF-F)	(C)
Gatorade	Initial	69.15 (6.11)Ba	65.56 (4.40)Ba	78.59 (3.79)Aa
	Final	52.05 (5.40)Bb	50.84 (1.53)Bb	57.63 (3.20)Ab
Distilled water	Initial	70.40 (3.72)Ba	67.32 (4.20)Ba	78.19 (3.89)Aa
	Final	51.77 (3.56)Bb	51.21 (3.19)Bb	56.30 (3.53)Ab

Uppercase letters compare the resins with each time period and beverage. Lowercase letters compare the time periods with each resin and beverage.

The SEM analysis (Figure 1) revealed variations in filler particle size, number, and arrangement among the different resin composites. The images of BF-F and C resins exhibited greater similarity in particle shape and size compared to BF-O resin, which showed a pattern of smaller and more closely packed particles. When comparing the images of each resin individually under different immersion conditions (distilled water or Gatorade), no significant differences were observed.

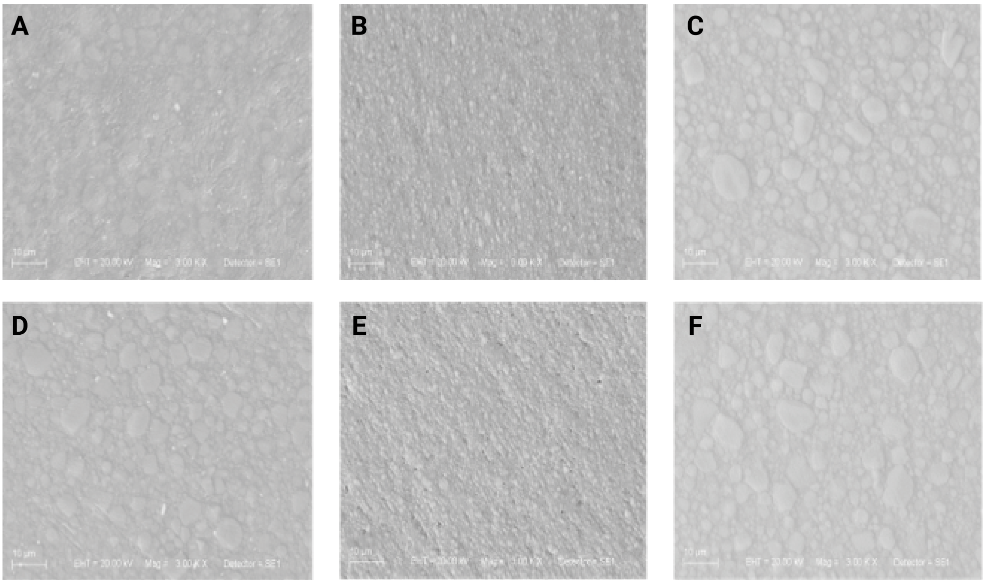

Figure 1. Representative images of the scanning electron microscopy analysis in the different groups: A. Filtek One Bulk Fill + Gatorade; B. Opus Bulk Fill + Gatorade; C. Z350XT + Gatorade; D. Filtek One Bulk Fill + Distilled Water; E. Opus Bulk Fill + Distilled Water; F. Z350XT + Distilled water.

Discussion

The results of the surface microhardness analysis showed that the C resin yielded higher values than the Bulk fill resins at all time points, regardless of the immersion beverage. The organic matrix of the C resin composite is composed of BIS-GMA, BIS-EMA, and UDMA, with small amounts of TEGDMA. This composition directly influences the polymerization, the degree of conversion of monomers into polymers, and the viscosity of the material[12], hence affecting the microhardness results.

The first hypothesis was accepted, since there was a decrease in the surface microhardness of all the evaluated resin composites; however, there was no statistically significant difference between the influence of the distilled water (control) or the hydroelectrolytic beverage on this microhardness. The decrease in the microhardness of these resinous materials was probably due to water sorption, which affected the polymeric network and reduced the frictional forces among the polymer chains[2]. According to Yap et al.[13] (2001), the presence of certain monomers in the composition of resin composites can increase their susceptibility to softening after exposure to some beverages. In this study, no differences were observed between the immersion results of the two liquids on the resins, likely because the exposure period was not long enough to affect hardness. However, other studies have shown that beverages with an acidic pH can indeed cause changes in hardness over time[1,14,15].

Regarding the roughness analysis, evaluating the behavior of Bulk Fill and conventional composite resins after exposure to Gatorade or distilled water, similar roughness values were observed within each group. Since roughness is a surface property of the restorative material, it can be affected by water sorption, a complex phenomenon that depends on the resin matrix, the filler particles, and the interfacial bond between the particles and the matrix. The filler particles of a resin composite can influence water absorption to a greater or lesser degree, depending on the filler, potentially affecting the surface roughness to varying degrees[16]. No difference being observed between the immersion results of each resin individually after 15 days may also have been influenced by the duration of the study. The study by Hemalatha and Nagar[17] (2018) indicates that resins exposed to beverages such as Gatorade showed statistically significant differences that increased progressively over 60 days.

Studies have shown that water sorption is higher for polymers with higher concentrations of the TEGDMA monomer, because of the presence of hydrophilic ether bonds[18], and this monomer is present in the organic matrix of the C resin, what may justify the higher roughness values when comparing the groups for each immersion liquids. The conventional composite resin (C) showed higher roughness values compared to the BF-F group when immersed in distilled water or Gatorade, and compared to the BF-O group when immersed in distilled water. Furthermore, the fact that the resin increments in group C (2mm) are different from those in groups BF-O and BF-F (4-5mm) may also justify the difference observed between the groups. Since this conversion occurs at 4-5mm and in group C it occurs at 2mm, it can be hypothesized that differences in the degree of conversion may help justify the differences found between the control groups and the bulk fill resins. An increase in the roughness of restorative materials is an important factor to be evaluated, since it can lead to plaque accumulation, which facilitates caries recurrences from microleakage, gingival irritation and stains on restored teeth[19].

SEM analysis was performed to visualize the morphological changes in the surface of the resin composites caused by the tested beverages. The images show different filler particle sizes and arrangements according to the different resins researched in this study. However, the images from the immersion results of distilled water and Gatorade showed no differences for any of the resins. This outcome corroborates the finding that roughness and microhardness were not affected by the immersion solutions, as no differences were found. Alencar et al.[20] (2020) observed that the SEM images for all the tested resins (including BF-F and C) showed mostly smooth surfaces, with small porosities (similar to those of the present research). The authors' objective was to observe the reactions of resins subjected to citric acid, hydrochloric acid and deionized water. Their results showed that citric acid and hydrochloric acid caused no more erosive damage than distilled water.

The combined analysis of surface hardness, surface roughness, and surface microscopy is crucial for assessing the quality of restorative materials. It is important to note that this study was conducted in an extraoral environment, with the specimens kept in an incubator at 37°C. Therefore, further studies are needed to evaluate the effects in an intraoral environment, where the restorative material is subjected to masticatory forces, the presence of saliva, brushing, and varying temperatures, among other factors that can cause direct changes. The specification of monomers, details regarding the quantity or percentage of material content, are not fully disclosed by manufacturers, and this information is necessary for a more thorough discussion of composite resin studies[21].

Thus, within the limitations of this study, it can be concluded that all composites experienced a reduction in microhardness when in contact with hydroelectrolytic beverages or distilled water, but the type of immersion liquid did not influence the roughness of each of the evaluated composite resins.

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Conflict of interest

The authors have no conflict of interest to disclose.

Data availability

Datasets related to this article will be available upon request to the corresponding author.

Author Contribution

Caio Castro Grigoletto: conception and design of the study; study conduct; drafting and writing the manuscript. **Laura Nobre Ferraz**: data collection; critical revision of the manuscript. **Rafael Pino Vitti**: analysis of data and statistics; critical revision of the manuscript. **Renata Siqueira Scatolin**: conception and design of the study; study conduct; drafting and writing the manuscript. All authors read and approved the final manuscript.

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