

## Effect of acidic solutions present in the diet on the surface roughness of microhybrid composite resins

Efeito de soluções ácidas presentes na dieta na rugosidade superficial de resinas compostas micro-híbridas

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### Abstract

The objective of this study was to evaluate in vitro the roughness and surface morphology of composite resins (CRs) stored for 180 days in acidic solutions present in the diet. Three micro-hybrid composite resins were used in this study (Charisma, Opallis, and Esthet-X). Fifty-one specimens were made of each material with 4 mm in diameter and 2 mm thick with the aid of an elastomer matrix. After photoactivation for 40 seconds (Elipar Free-Light / 3M ESPE), the specimens were stored in distilled water for 24 hours at 37°C for subsequent surface polishing with Soft Lex / 3M ESPE discs in fine and extra-fine granulation. Then, the specimens were dried with absorbent paper and the surface roughness was measured using a rugosimeter. The specimens were then randomly divided into five groups (n=10) according to the tested solutions: DW - Distilled water (pH 5.30); RW - Red Wine / Trápiche Syha Ind. Argentina red wine (pH 3.68); CK - Coke / Coca Cola Co. (pH 2.73), OJ - Orange juice / Purity Cocamar (pH 3.58) and LBS - lactate buffer solution (pH 2.74). The tested solutions were changed daily. The surface roughness test was repeated after 15, 90 and 180 days of immersion. The average values of surface roughness were submitted to LSmeans analysis and Tukey-Kramer test, at a significance level of 5%. The results of surface roughness revealed that all CRs showed a significant increase in the roughness values over time for all tested solutions.

**Keywords:** Composite resins; Diet simulator solutions; Acidic solutions; Alcoholic solutions; Surface roughness.

### Resumo

O objetivo deste estudo foi avaliar in vitro a rugosidade e a morfologia superficial de resinas compostas (RCs) armazenadas por 180 dias em soluções ácidas presentes na dieta. Três resinas compostas micro-híbridas foram utilizadas neste estudo (Charisma, Opallis e Esthet-X). Cinquenta e um corpos de prova foram confeccionados de cada material com 4 mm de diâmetro e 2 mm de espessura com o auxílio de uma matriz de elastômero. Após fotoativação por 40 segundos (Elipar Free-Light/3M ESPE), os corpos de prova foram armazenados em água destilada por 24 horas a 37°C para posterior polimento superficial com discos Soft Lex/3M ESPE em granulação fina e

extrafina. Em seguida, os corpos de prova foram secos com papel absorvente e a rugosidade superficial foi medida com um rugosímetro. Os corpos de prova foram então divididos aleatoriamente em cinco grupos (n=10) de acordo com as soluções testadas: DW - Água destilada (pH 5,30); RW - Vinho Tinto / Trapiche Syha Ind. Vinho Tinto Argentina (pH 3,68); CK - Coca-Cola / Coca Cola Co. (pH 2,73), OJ - Suco de laranja / Pureza Cocamar (pH 3,58) e LBS - solução tampão de lactato (pH 2,74). As soluções testadas foram trocadas diariamente. O teste de rugosidade superficial foi repetido após 15, 90 e 180 dias de imersão. Os valores médios de rugosidade superficial foram submetidos à análise LSmeans e teste de Tukey-Kramer, ao nível de significância de 5%. Os resultados de rugosidade superficial revelaram que todos os RCs apresentaram um aumento significativo nos valores de rugosidade ao longo do tempo para todas as soluções testadas.

**Palavras-chave:** Resinas compostas; Soluções simuladoras de dieta; Soluções ácidas; Soluções alcoólicas; Rigidez superficial.

### Resumen

El objetivo de este estudio fue evaluar in vitro la rugosidad y morfología superficial de resinas compuestas (RCs) almacenadas durante 180 días en soluciones ácidas presentes en la dieta. En este estudio se utilizaron tres resinas compuestas microhíbridas (Charisma, Opallis y Esthet-X). Se fabricaron 51 especímenes de cada material de 4 mm de diámetro y 2 mm de espesor con la ayuda de una matriz elastomérica. Luego de la fotoactivación por 40 segundos (Elipar Free-Light/3M ESPE), los especímenes fueron almacenados en agua destilada por 24 horas a 37°C para su posterior pulido superficial con discos Soft Lex/3M ESPE en granulación fina y extrafina. Luego, las probetas se secaron con papel absorbente y se midió la rugosidad superficial con un rugosímetro. Los especímenes fueron luego divididos al azar en cinco grupos (n=10) de acuerdo con las soluciones probadas: DW - Agua destilada (pH 5,30); RW - Vino Tinto / Trapiche Syha Ind. Vino Tinto Argentino (pH 3,68); CK - Coca-Cola / Coca Cola Co. (pH 2,73), OJ - Zumo de naranja / Cocamar Purity (pH 3,58) y LBS - Solución tampón de lactato (pH 2,74). Las soluciones ensayadas se cambiaron diariamente. El ensayo de rugosidad superficial se repitió a los 15, 90 y 180 días de inmersión. Los valores promedio de rugosidad superficial fueron sometidos a análisis LSmeans y prueba de Tukey-Kramer, a un nivel de significancia del 5%. Los resultados de rugosidad de la superficie revelaron que todos los RC mostraron un aumento significativo en los valores de rugosidad con el tiempo para todas las soluciones probadas.

**Palabras clave:** Resinas compuestas; Soluciones de simulación de dieta; Soluciones ácidas; Soluciones alcohólicas; Rugosidad de la superficie.

## 1. Introduction

Acidic drinks have become a common practice among people, especially those looking for healthy eating and regular physical activity. These drinks help speed up metabolism, facilitating caloric expenditure and weight loss. The associated constant consumption can accelerate the surface degradation of composite resin restorations in the oral cavity (Ciuris et al., 2019).

Direct restorations with resinous composites correspond to one of the most widely performed clinical procedures today, due to technological development and improvement of the physical, mechanical and aesthetic properties of composite resins, allowing their use in both anterior and posterior teeth (Hwang et al., 2018; Tanthanuch et al., 2018; Leite, Silva, Meireles, Duarte & Andrade, 2014).

In general, the composite resins present a polymeric matrix consisting of dimethacrylate monomers, such as bisphenol A glycol dimethacrylate (Bis-GMA), urethane dimethacrylate (UDMA) and / or triethylene glycol dimethacrylate (TEGDMA) (Valinoti, Neves, Silva & Maia, 2008). The presence of hydroxyl groups (OH) in the Bis-GMA molecule it is responsible for the water absorption and solubility characteristics of composite resins. The absorption of water through hydroxyl produces the plasticization of the polymer and the consequent reduction in chemical and mechanical properties, in addition to increasing susceptibility to staining (Moszner, Fischer, Angermann, & Rheinberger, 2006; Soares et al., 2007). To reduce this problem, the replacement of OH groups with methyl groups (CH<sub>3</sub> Bis-GMA), originating the Bis-EMA monomer, which has a high molecular weight and less viscosity when compared to Bis-GMA, has been suggested because it results in material less susceptible to degradation by solvents, less water absorption and greater clinical durability (Camilotti et al., 2020). In addition, Bis-GMA monomers present in their composition ester radicals that undergo hydrolysis when subjected to acidic media, consequently generating a degradation of the resinous material (Valinoti, Neves, Silva & Maia, 2008). In addition to the

organic matrix, composed primarily of dimethacrylate monomers and ester bonds, resins compounds are constituted by an initiator-accelerator polymerization system, and particles of inorganic charge coated by a bonding agent, normally silane, which contains methyl methacrylate, and allows the union of these particles with the organic matrix (Valinoti, Neves, Silva & Maia, 2008; Yulianto et al., 2019).

The available composite resins can be classified in different ways, however, they are usually grouped according to the type of particles with inorganic charge, the size of these particles and their distribution (Kocağaoğlu et al., 2019). The inorganic particles can consist of silica and alkaline glasses (silicon dioxide, barium oxide and strontium oxide), and promote composite resins their mechanical properties, as well as optical and radiopacity properties, which vary according to their size, shape and distribution (Habib, Wang, Wang, Zhu & Zhu, 2016). Thus, according to Ferracane (2011) apud Pfeifer (2017) these composites can be classified into micro-particulate resins, consisting of silica particles with size between 0.4-0.5  $\mu\text{m}$  (40-50 nm); hybrids, composed of 10-50  $\mu\text{m}$  glass particles and 0.4  $\mu\text{m}$  (40 nm) silica particles; micro-hybrids, 0.6-1  $\mu\text{m}$  glass particles and 0.4  $\mu\text{m}$  (40 nm) silica particles; nano-hybrids, 0.6-1  $\mu\text{m}$  glass particles and 5-100 nm silica particles (Pfeifer, 2017). Besides, currently a smallest scale of particles has been used nanofillers and the composite resins with exclusivity of nanometric particles are classified as nanofilled (Vilela et al., 2021).

The composition of the composite resin, its physical properties and its degree of conversion must guarantee the resistance of the restorative material to the action of solvents in the oral environment. In this sense, the pH of the oral environment and the contact time of the solution have a high influence on the principle of degradation of the restoration surface. Acid substances presents in the oral environment can generate a surface degradation and the increase of surface roughness, since acid solutions cause the erosion of the agent coating the filler particles of the composite resin, that results in the loss of hardness (Münchow et al., 2014; Camilotti et al., 2020). Clinically this can imply to loss of restoration contour, increasing of plaque retention due the increase of surface roughness, that can generate a most susceptibility to wear and, as a consequence, caries and periodontal inflammation (Cilli, Pereira & Prakki, 2012). Thus, the objective of this study was to determine the effect of acidic and alcoholic solutions on the surface of composite resins by analyzing the roughness and morphology of the variables, composition of composite resins and time of exposure to the medium.

## 2. Methodology

Three composite resins indicated for anterior and posterior teeth restorations were selected for this study. The selected composite resins present inorganic particles with average size on the micrometric scale (0.4 - 0.6  $\mu\text{m}$ ) and the organic matrix based on Bis-GMA as the predominant monomer: Charisma (Heraeus Kulzer, São Paulo, SP, Brazil) Opallis (FGM, Joinville, SC, Brazil) and Esthet-X (Dentsply, York, Pennsylvania, USA). The technical description, including basic composition, manufacturer and batch, is shown in Table 1.

**Table 1** – Description of the composite resins used in the study.

Material	Composition	Particle size (µm)	Manufacturer	Production batch
Charisma	Bis-GMA, TEGDMA Particles: barium charge	0.7 to 3 75% by weight	Hereaus Kulzer	010306
Opallis	Bis-GMA BisEMA, TEGDMA UDMA camphorquinone, co-initiator and silane, co-initiator and silane. Particles: silanized barium-aluminum silicate glass, pigments and silicas.	0,5 75 a 77% by weight	FGM	170308
Esthet X	BisGMA, TEGDMA, BisEMA Particles: barium-fluorine-aluminum silicate, silica, initiators and stabilizers	1 a 0,4 77% by weight	Dentsply	050400137 3

Fonte: Autores.

## 2.1 Preparation of the specimens

The sample size calculation was based on the pilot study; thus, fifty specimens (n=10) were made for each material. For the preparation of the specimens, a polyvilsiloxane matrix with 4 mm in diameter and 2 mm in thickness was used. The composite resins were inserted in the matrix in two increments. A dental floss was inserted in the increments of the composite resins, 10 cm long to leave the specimens suspended in the solutions. Then, a polyester strip (K-Dent, Joinville, SC, Brazil) was placed over the composite resin and the material pressed by digital compression with the aid of a 2 mm thick glass plate. The material was photoactivated for 40 seconds through the polyester strip using the Elipar Free-Light 2 light unit (3M ESPE, St. Paul, MN, USA) with a power density of 1200 mW / cm<sup>2</sup>, measured on a radiometer (Demetron, Kerr, Orange, CA, USA). The diameter and thickness of all specimens were measured using a digital electronic caliper (Mitutoyo Corporation, Tokyo, Japan). After polymerization, the specimens were stored for 24 hours at 37°C at 100% relative humidity. Then, the specimens were subjected to the surface polishing procedure (on the top surface) with aluminum oxide discs (Soft Lex Pop On, 3M ESPE, São José do Rio Preto, SP, Brazil) mounted on a low rotation handpiece (Kavo Dental, Biberach, Germany) with fine and superfine grain. Between one sanding disc and another, the specimens were cleaned with air / water jets and in water under ultrasound for 15 minutes. After polishing was finished, the specimens were cleaned again in distilled water with the aid of ultrasound (Ultrasonic Cleaner, Cincinnati, Ohio, USA) for 30 minutes to remove the polishing debris. During the cleaning period on the ultrasound, the distilled water was changed every 10 minutes.

## 2.2 Surface roughness

Each specimen had the surface carefully dried with absorbent paper to measure the surface roughness. For this, three readings of the real profiles of the surfaces of each specimen were made, with the arithmetic mean of the readings being their representative value.

To check the surface roughness, a Rugosimeter (Surfcorder SE 1700, Kosaka Laboratory, Tokyo, Japan) was used with a diamond tip of 2 µm in diameter that covered a previously delimited surface (1.25 mm). The device features specific software that processes the information quantitatively. Thus, the results were recorded on a microcomputer that stored the information relevant to the test.

The parameter of Ra (mean roughness) was used, which translates the value of the arithmetic mean of all absolute distances of the roughness profile (R) from the central line, within the mean extension Lm (Limit of measurement = the extent considered in the reading). The Lm was 1.25 mm, cut off 0.25 mm. The tolerances (T = the extreme values to be considered in the readings) were 0.01 µm (minimum T) and 8 µm (maximum T). The Ra parameter was used in order to provide better

conditions for comparison with results of other studies carried out with this parameter.

After the diamond tip traversed the entire specimen at a speed of 0.5 mm / sec with a load of 0.7 mN, a graph was created by the device's software. To analyze the wear of the specimen, the parameter used was the reading of the actual profiles tested. After the evaluation of the roughness, a specimen was randomly separated for evaluation in the scanning electron microscope. The roughness test was performed immediately after the specimens were made and repeated after 15, 90 and 180 days of storage in the experimental solutions.

### 2.3 Storage

The specimens were randomly divided into five groups, with three specimens from each group being separated for the analysis of surface morphology (n=10): Group 1 - Water distilled; Group 2 - Red wine (Trapiche Syha, Mendoza, Argentina); Group 3- Coca-Cola classic (Coca-cola Brazil, Porto Alegre, RS, Brazil); Group 4 - Orange juice (Cocamar, Altonia, PR, Brazil); Group 5 - Lactate buffer solution. The solutions were changed daily. Each specimen was stored individually in properly identified 1.5 mL tubes. The specimens were stored in an oven at 37°C without exposure to light.

The pH values of each of the solutions were determined using a pH meter (Thermo Scientific Orion, Waltham, MA, USA) that consists of an electrode coupled to a potentiometer. Prior to the immersion of the specimens, the pH meter was placed in each solution to convert the electrode potential value into pH units. This procedure was repeated daily before changing each solution. The composition of the solutions and the pH values are described in Table 2.

**Table 2** – Description of the solutions used with the pH value.

Solutions	pH	Composition	Trade mark
Distilled water	5,30	Distilled water	_____
Wine	3,68	water, 14% alcohol, tartaric acids 5.5 and 8.5g / l., fatty acids, glycerin, sugar, amino acids, carbohydrates, mono terpenes, aldehydes and esters.	Trapiche Syha (Industria Argentina)
Coke Classic	2,73	Carbonated water, sugar: 2400g, enough water to dissolve, caramel: 37g, caffeine: 3.1g, phosphoric acid: 11g, decocainized coca leaf: 1.1g, kola nut: 0.37g	Coca-Cola classic/ Coca-cola LTDA
Orange Juice	3,58	Water, sugar, concentrated orange juice, acidulants: citric acid, flavoring: natural orange flavor, vitamins A and D, stabilizers: water gum and natural colors: turmeric and annatto. Gluten-free	Purity/ Cocamar
Lactate Buffer Solution	2,74	8.26g of lactic acid, 0.92 d of sodium lactate, deionized water	_____

Fonte: Autores.

### 2.4 SEM evaluation

A specimen of each composite resin in each solution after 24h, 15, 90 and 180 days was subjected to analysis by scanning electron microscopy (JEOL-JSM 5600, Tokyo, Japan), at a magnification of 3000 X to check the changes in the microstructure. For the analysis, the selected specimens were cleaned in ultrasound (Ultrasonic Cleaner) for thirty minutes, with the water being changed every ten minutes. Then, the specimens were mounted in a sample holder and coated with gold-palladium under high vacuum (Balzer - SCD 050).

As the same specimen was used to measure the surface roughness values for all evaluated periods, the Mauchly sphericity test was applied using a mixed model by the PROC MIXED procedure for repeated measurements of the SAS statistical program, considering the design of 3 resins x 5 solutions (with repeated measures in 4 steps). The treatment averages were compared using the LSmeans command and the Tukey-Kramer test. The level of significance considered was 5%.

### 3. Results

The average values obtained during the reading of the surface roughness of the specimens, including the standard deviation in the respective experimental groups are shown in Table 3. The statistical differences were identified with the application of the LSmeans Analysis and Tukey-Kramer test. The level of significance considered was 5%.

**Table 3** – Average values of surface roughness and respective standard deviations for composite resins as a function of the solutions, in the different reading periods (Ra -  $\mu\text{m}$ ).

Solutions	Period				Material
	Initial	30 days	90 days	180 days	
					Charisma
Water	0,223( $\pm$ 0,013)Da	0,238( $\pm$ 0,010)Ca	0,256( $\pm$ 0,014)Bab	0,266( $\pm$ 0,012)Aab	
Wine	0,224( $\pm$ 0,012)Da	0,253( $\pm$ 0,015)Ca	0,274( $\pm$ 0,017)Bab	0,304( $\pm$ 0,016)Aab	
Coke	0,236( $\pm$ 0,019)Da	0,275( $\pm$ 0,018)Ca	0,313( $\pm$ 0,016)Ba	0,347( $\pm$ 0,014)Aa	
Juice	0,212( $\pm$ 0,038)Da	0,229( $\pm$ 0,039)Ca	0,250( $\pm$ 0,038)Bb	0,258( $\pm$ 0,038)Ab	
Lactate	0,214( $\pm$ 0,08)Da	0,246( $\pm$ 0,012)Ca	0,278( $\pm$ 0,012)Bab	0,290( $\pm$ 0,014)Aab	
					Opallis
Water	*0,302 ( $\pm$ 0,035)Da	*0,319( $\pm$ 0,037)Ca	*0,339( $\pm$ 0,037)Bab	*0,349( $\pm$ 0,037)Aab	
Wine	*0,284 ( $\pm$ 0,058)Da	#0,313( $\pm$ 0,059)Ca	*0,344( $\pm$ 0,059)Bab	*0,364( $\pm$ 0,058)Aab	
Coke	0,251 ( $\pm$ 0,030)Da	0,291 ( $\pm$ 0,029)Ca	0,314( $\pm$ 0,030)Bab	0,334( $\pm$ 0,030)Aab	
Juice	0,257 ( $\pm$ 0,045)Da	0,275 ( $\pm$ 0,046)Ca	0,295( $\pm$ 0,044)Bb	0,306( $\pm$ 0,046)Ab	
Lactate	*0,309 ( $\pm$ 0,021)Da	*0,333 ( $\pm$ 0,023)Ca	*0,369 ( $\pm$ 0,024)Ba	*0,400( $\pm$ 0,025)Aa	
					Esthet-X
Water	0,210 ( $\pm$ 0,006)Da	0,225 ( $\pm$ 0,005)Ca	0,240( $\pm$ 0,006)Ba	0,249( $\pm$ 0,007)Aa	
Wine	0,218 ( $\pm$ 0,007)Da	0,232( $\pm$ 0,007)Ca	0,272( $\pm$ 0,012)Ba	0,293( $\pm$ 0,012)Aa	
Coke	0,207 ( $\pm$ 0,015)Da	0,240 ( $\pm$ 0,014)Ca	0,271( $\pm$ 0,012)Ba	0,288( $\pm$ 0,013)Aa	
Juice	0,207 ( $\pm$ 0,013)Da	0,237 ( $\pm$ 0,014)Ca	0,260( $\pm$ 0,012)Ba	0,295( $\pm$ 0,011)Aa	
Lactate	0,211 ( $\pm$ 0,009)Da	0,248( $\pm$ 0,007)Ca	0,279( $\pm$ 0,005)Ba	0,306( $\pm$ 0,05)Aa	

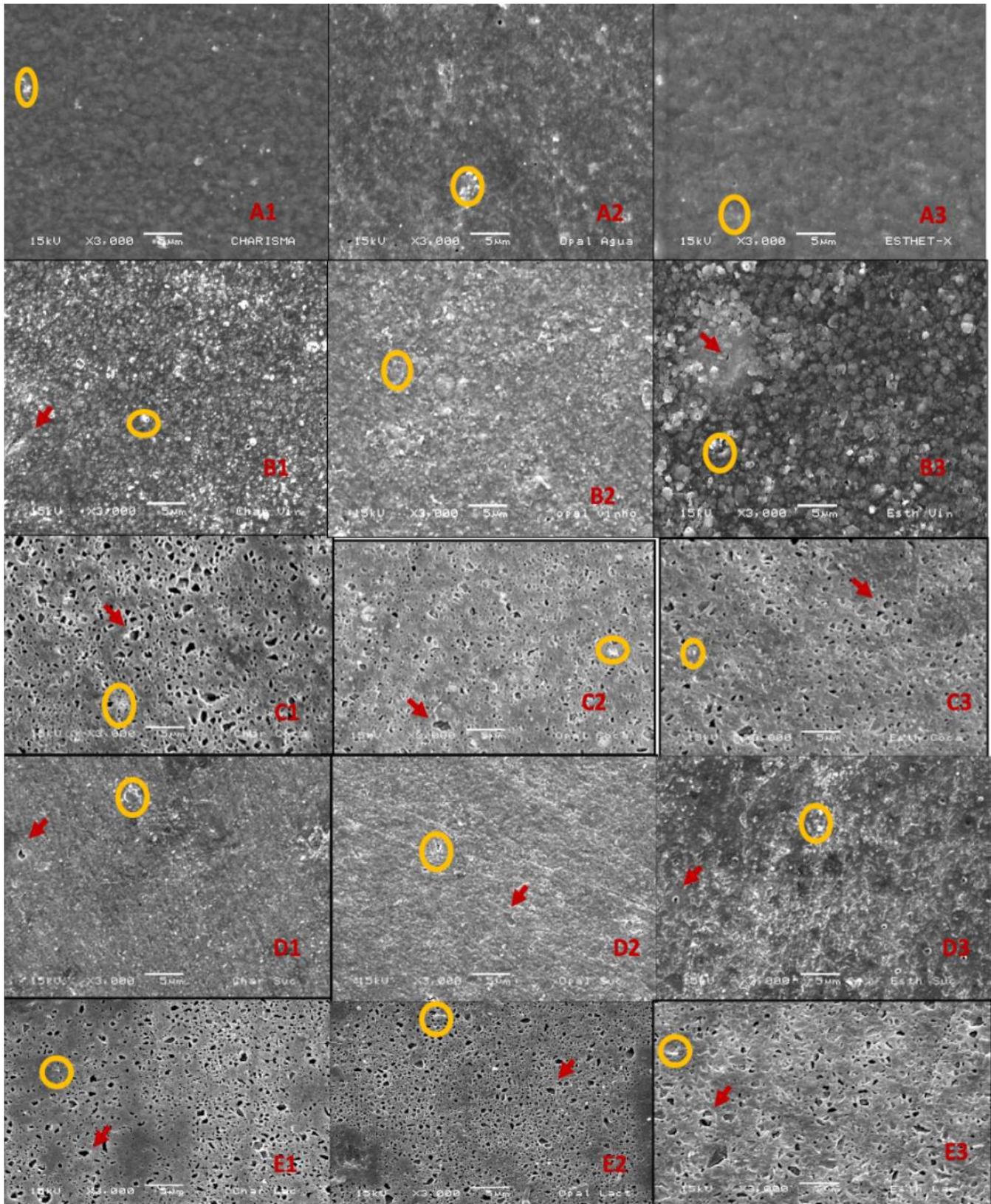
Means followed by different letters (uppercase in the horizontal and lowercase in the vertical within each material) differ from each other ( $p \leq 0.05$ ). \* It differs from Charisma and Esthet-X in the same time and solution ( $p \leq 0.05$ ). # It differs from Esthet-X in the same time and solution ( $p \leq 0.05$ ). Fonte: Autores.

The results found in the present research revealed morphological changes on the surface of the different composite resins when exposed for 180 days to acidic and alcoholic substances ingested in the daily diet. It was observed in Table 3 that the three composite resins evaluated showed a significant increase in the value of the gradual surface roughness up to the period of 180 days for all evaluated solutions. The composite resin Charisma showed statistically higher roughness values for coca-cola solution in the periods of 90 and 180 days. The Opallis composite resin had its roughness values significantly increased for the lactate solution in the periods of 90 and 180 days. The Esthet-X composite resin did not have its roughness values significantly altered due to the storage solution.

Regarding the comparison between the composite resins, it was noted that the Opallis composite resin presented the highest values of significantly roughness compared to the other composite resins evaluated for the water, lactate and wine solutions. Except for the 30-day period in red wine, there was no statistically significant difference between Opallis and Charisma composite resins.

Images A1 to A3 (Figure 1) represent the three composite resins evaluated stored in water for ninety days. The images reveal an irregular organic matrix showing the charge particles to show, this aspect being more accentuated for the Opallis (A2) composite resin. In the red wine solution (B1 to B3) it reveals dissolution of the organic matrix with outcrop of the charge particles on the surface of the materials. It is also possible to observe the irregularity of the sizes of the charge particles of the composite resins Charisma and Esthet-X. For the coke solution (C1 to C3) it shows the displacement of part of the charge particles from the material surface, leaving signs of porosity. The composite resins stored in orange juice (D1 to D3) showed discontinuity in the organic matrix and the extrusion of some particles of charge on the surface of the materials. The lactate buffer solution caused great loss of the charge particles, showing signs of porosity. In Opallis composite resin this aspect is more pronounced.

**Figure 1** - SEM photographs (3.000X magnification) of the composite resin Charisma (left column), Opallis (center column) and Esthet-X (right column) after the 90-day immersion period in water (A) ; red wine (B); coke (C); orange juice (D) and lactate buffer solution (E). The red arrows exemplify the break in the continuity of the resin matrix and the yellow circles elucidate the charge particles.



Fonte: Autores.

#### 4. Discussion

The results found in the present research revealed morphological changes on the surface of the different composite resins when exposed for 180 days to acidic and alcoholic substances ingested in the daily diet.

All composite resins evaluated showed a significant gradual increase in the surface roughness value up to the period of 180 days for all evaluated solutions, data that corroborate the study previously carried out by Camilotti et al. (2020) (Camilotti et al., 2020). Furthermore, these findings contradict the results presented in the analysis by Karaman et al. (2014) in which the surface roughness of the tested composite resins was not affected by the action of the solutions. However, these data can be explained due to the short immersion period to which the resins were subjected (only 15 days) (Karaman, Tuncer, Firat, Ozdemir & Karahan, 2014).

To assess the clinical effect of the exposure time of certain solutions on composite resin restorations, some projections were made according to the protocol by Von Fraunhofer & Rogers (2004) apud Camilotti et al. (2020) based on the consumption of two liters of water divided into 8 glasses (250 ml each) per day and resident time in the mouth of 20 seconds, the exposure would be equal to 10 hours per month. Direct contact for 24 hours for 30 days would be equivalent to 720 hours of exposure. In the present study, composite resins were exposed in storage solutions for a period of 180 days, equivalent to an approximate daily consumption of three years, a reasonable period to evaluate the chemical degradation of composite resin restorations present in the oral cavity (Camilotti et al., 2020).

Probably, the action of the different solutions on the surface of the composite resin establishes the process of chemical degradation by hydrolysis of the bond between the silane and the surface of the load contained in the composite. As a consequence, the loose cargo particle can be displaced by the mechanical and physiological processes to which restoration is subjected in the oral environment, making the surface rougher, according to the values recorded in this *in vitro* study and the one previously reported by Moszner et al. (2006) (Moszner, Fischer, Angermann, & Rheinberger, 2006).

The morphological analysis of the Opallis and Charisma composite resins presents irregularly shaped particles that can contribute to the increase in roughness when compared with the Esthet-X composite resin as shown in the images in figure 1 (B1 to B3). Sabbagh et al. (2004) evaluated the influence of the size and shape of the charge particles on the morphology of different composite resins and found that materials with more irregular charge particles showed greater changes in the surface morphology, corroborating the findings in the present study (Sabbagh et al., 2004).

The composite resins Charisma, Opallis and Esthet-X, use barium glass as the main constituent element of the inorganic particle, whose average size is between 0.5 to 0.7  $\mu\text{m}$ . In addition, the organic matrix is mainly composed of Bis-GMA. Even with the similar composition, the Opallis composite resin showed statistically higher roughness values than the other composite resins evaluated when stored in water, red wine and lactate solution. This fact can be attributed to the presence of a greater amount of diluting monomers such as TEGDMA and UDMA in the Opallis composite resin. Since the Esthet-X and Charisma composite resins present only the TEGDMA monomer as a diluent. This hypothesis can be supported by the study by Sideridou & Achilas (2005) that verified the displacement of different resin monomers stored in different immersion periods. The authors found that the UDMA and TEGDMA monomers were the most easily removed from the interior of the restorative material (Sideridou & Achilas, 2005). This process can be intensified in the presence of alcoholic solution (red wine pH 3.68), the alcohol diffuses in the composite resin, plasticizes the matrix polymer causing reduction of some mechanical properties such as hardness, wear resistance and smoothness, acting directly on the matrix particle interface (Sideridou & Achilas, 2005). Valinoti et al. (2008) also reported higher values of surface roughness for Opallis composite resin when compared to different micro-hybrid composite resins (Valinoti, Neves, Silva & Maia, 2008).

The intensity of chemical degradation may also be associated with the acidity of the storage solutions. According to Han et al. (2008) an alcoholic and / or acidic environment generates the superficial degradation of composite resins (Han, Okamoto, Fukushima & Okiji, 2008). Furthermore, Shabaniyan & Richards (2002) reported that the lower the pH of a solution, the more damage it will bring to the mechanical performance of composite resins (Shabaniyan & Richards, 2002).

The erosive effect of the lactate buffer solution on the evaluated composite resins, especially for the Opallis composite resin, can be elucidated by the hydrolysis of the ester groups present in the organic matrix responsible for the union of Bisphenol A to methacrylate groups. In particular, the hydrolysis of the ester groups could initiate the formation of free carboxylic acids that can decrease the pH values inside the polymer and intensify hydrolytic degradation (Santerre, Shajii & Leung, 2001; Prakki, Cilli, Mondelli, Kalachandra & Pereira, 2005). In contrast, Munack et al. (2001) reported that neither acidity nor the storage time influenced the surface roughness of different polyacid-modified composite resins after one year of storage (Munack, Haubert, Dogan & Geurtsen, 2001). As these authors changed the solutions weekly, the acidity of the solution decreased and, consequently, its erosive potential was reduced. In this study, solutions were exchanged daily. With that, the pH of the solutions were maintained and also the potential for degradation, as explained by Owens & Kitchens (2007) (Owens & Kitchens, 2007).

When the composite resins were stored in Coke (pH 2.73) and orange juice (3.68), the damage caused to the surface of these materials was similar to each other. This confirms the results found by Franciscone et al. (2008) who observed that micro-hybrid composite resins did not show statistically significant differences in surface roughness values when stored in juice solution or Cokeclassic (Franciscone et al., 2008).

The Esthet-X composite resin did not show significant changes in the surface roughness values due to the different storage solutions. It can be attributed to the presence of a less soluble organic matrix with stronger cross-links compared to the other composite resins evaluated, making it difficult to leach residual monomers (Sideridou & Achilas, 2005). Similar results were found by Mohamed-Tahir & Yap (2004) (Mohamed-Tahir & Yap, 2004). The authors observed that the Esthet-X composite resin did not change the roughness values due to the acidity of the storage solutions (Mohamed-Tahir & Yap, 2004).

It was possible to verify that the solutions that increased the surface roughness values of some composite resins were the lactate buffer solution and the red wine. Regarding the material, the Esthet-X composite resin showed better results both in the surface roughness values and in the analysis of the surface morphology for the different solutions and evaluation periods. This shows that the surface roughness and chemical degradation are dependent on the composition of the composite resins, as observed by Yazici, et al. (2010) (Yazici, Tuncer, Antonson, Onen & Kilinc, 2010). Clinically the effects of acidic and alcoholic solutions on composite resins may be different. The exchange of the components of the solutions interacting with the active components of other products of the diet and oral hygiene, can interfere in the chemical degradation of the composite resins. Further studies should be carried out to evaluate these parameters, making associations of agents that can be aggressive to direct restorative materials, trying to similar the condition of the oral environment.

## 5. Conclusion

According to the results found in this study, it can be concluded that the acidic and alcoholic solutions can negatively interfere with the results of surface roughness in micro-hybrid composite resins, and that the composition and commercial brand influenced the behavior towards the solutions. Opallis composite resin was the one that suffered the most damage, both in roughness and surface analysis, with storage in the tested solutions.

## References

- Camilotti, V., Mendonça, M. J., Dobrovolski, M., Detogni, A. C., Ambrosano, G. M. B., & De Goes, M. F. (2020). Impact of dietary acids on the surface roughness and morphology of composite resins. *Journal of Oral Science*, 63 (1), 18-21.
- Cilli, R., Pereira, J. C., & Prakki, A. (2012). Properties of dental resins submitted to pH catalysed hydrolysis. *Journal of Dentistry*, 40, (12), 1144-1150.
- Ciuris, C., Lynch, H. M., Wharton, C., & Johnston, C. S. (2019). A Comparison of Dietary Protein Digestibility, Based on DIAAS Scoring, in Vegetarian and Non-Vegetarian Athletes. *Nutrients*, 10, (12): 3016.
- Francisconi, L. F., Honório, H. M., Rios, D., Magalhães, A. C., Machado, M. A., & Buzalaf, M. A. (2008). Effect of erosive pH cycling on different restorative materials and on enamel restored with these materials. *Operative Dentistry*, 33 (2), 203-208.
- Habib, E. G., Wang, R., Wang, Y., Zhu, M., & Zhu, X. X. (2016). Inorganic Fillers for Dental Resin Composites - Present and Future. *ACS Biomaterials Science & Engineering*, 2 (1), 1-11.
- Han, L., Okamoto, A., Fukushima, M., & Okiji, T. (2008). Evaluation of Flowable Resin Composite Surfaces Eroded by Acidic and Alcoholic Drinks. *Dental Materials Journal*, 27 (3), 455-465.
- Hwang, S., Chung, S. H., Lee, J. T., Kim, Y. T., Kim, Y. J., Oh, S., & Yeo, I. L. (2018). Influence of Acid, Ethanol, and Anthocyanin Pigment on the Optical and Mechanical Properties of a Nanohybrid Dental Composite Resin. *Materials*, 11 (7), 1234.
- Karaman, E., Tuncer, D., Firat, E., Ozdemir, O. S., & Karahan, S. (2014). Influence of Different Staining Beverages on Color Stability, Surface Roughness and Microhardness of Silorane and Methacrylate-based Composite Resins. *Journal of Contemporary Dental Practice*, 15 (3), 319-325.
- Kocaağaoğlu, H., Aslan, T., Gürbulak, A., Albayrak, H., Taşdemir, Z., & Gumus, H. (2017). Efficacy of Polishing Kits on the Surface Roughness and Color Stability of Different Composite Resins. *Nigerian Journal of Clinical Practice*, 20 (5), 557-565.
- Leite, M. L. A. S., Silva, F. D. S. C. M., Meireles, S. S., Duarte, R. M., & Andrade, A. K. M. (2014). The effect of drinks on color stability and surface roughness of nanocomposites. *European Journal of Dentistry*, 8 (3), 330-336.
- Mohamed-Tahir, M. A., & Yap, A. U. (2004). Effects of pH on the surface texture of glass ionomer based/containing restorative materials. *Operative Dentistry*, 29 (5), 586-591.
- Moszner, N., Fischer, U. K., Angermann, J., & Rheinberger, V. (2006). Bis-(acrylamide)s as new cross-linkers for resin-based composite restoratives. *Dental Materials*, 22 (12), 1157-1162.
- Munack, J., Haubert, H., Dogan, S., & Geurtsen, W. (2001). Effects of various storage media on surface hardness and structure of four polyacid-modified composite resins ("compomers"). *Clinical Oral Investigations*, 5 (4), 254-259.
- Münchow, E. A., Ferreira, A. C., Machado, R. M., Ramos, T. S., Rodrigues-Junior, S. A., & Zanchi, C. H. (2014). Effect of acidic solutions on the surface degradation of a micro-hybrid composite resin. *Brazilian Dental Journal*, 25 (4), 321-326.
- Owens, B. M., & Kitchens, M. (2007). The erosive potential of soft drinks on enamel surface substrate: an in vitro scanning electron microscopy investigation. *Journal of Contemporary Dental Practice*, 8 (7), 11-20.
- Pfeifer, C. S. (2017). Polymer-Based Direct Filling Materials. *Dental Clinics of North America*, 61 (4), 733-750.
- Prakki, A., Cilli, R., Mondelli, R. F., Kalachandra, S., & Pereira, J. C. (2005). Influence of pH environment on polymer based dental material properties. *Journal of Dentistry*, 33 (2), 91-98.
- Sabbagh, J., Ryelandt, L., Bachérius, L., Biebuyck, J. J., Vreven, J., Lambrechts, P., & Leloup, G. (2004). Characterization of the inorganic fraction of resin composites. *Journal of Oral Rehabilitation*, 31 (11), 1090-1101.
- Santerre, J. P., Shajii, L., & Leung, B. W. (2001). Relation of dental composite formulations to their degradation and the release of hydrolyzed polymeric-resin-derived products. *Critical Reviews in Oral Biology & Medicine*, 12 (2), 136-151.
- Shabaniyan, M., & Richards, L. C. (2002). *In vitro* wear rates of materials under different loads and varying pH. *Journal of Prosthetic Dentistry*, 87 (6), 650-656.
- Sideridou, I. D., & Achilias, D. S. (2005). Elution study of unreacted Bis-GMA, TEGDMA, UDMA, and Bis-EMA from light-cured dental resins and resin composites using HPLC. *Journal of Biomedical Materials Research Part B: Applied Biomaterials*, 74 (1), 617-626.
- Soares, L. E., Cesar, I. C., Santos, C. G., De Cardoso, A. L., Liporoni, P. C., Munin, E., & Martin, A. A. (2007). Influence of coffee on reflectance and chemistry of resin composite protected by surface sealant. *American Journal of Dentistry*, 20 (5), 299-304.
- Tanthanuch, S., Kukiattrakoon, B., Eiam-O-Pas, K., Pokawattana, K., Pamanee, N., Thongkamkaew, W., & Kochatung, A. (2018). Surface changes of various bulk-fill resin-based composites after exposure to different food-simulating liquid and beverages. *Journal of Esthetic and Restorative Dentistry*, 30 (2), 126-135.
- Valinoti, A. C., Neves, B. G., Silva, E.M., & Maia, L. C. (2008). Surface degradation of composite resins by acidic medicines and pH- cycling. *Journal of Applied Oral Science*, 16 (4), 257-265.

Vilela, A.L.R., Machado, A.C., Queiroz, L.L., Batista, P.H.M., Faria-E-Silva, A.L., Menezes, M.S. (2021). Effect of Interval Time between Corrosive and Abrasive Challenges on a Nanoparticulate Composite Resin. *European Journal of Dentistry*, 15 (4), 607-611.

Yazici, A.R., Tuncer, D., Antonson, S., Onen, A., Kilinc, E. (2010). Effects of delayed finishing/polishing on surface roughness, hardness and gloss of tooth-coloured restorative materials. *European Journal of Dentistry*, 4 (1), 50-56.

Yulianto, H.D.K., Rinastiti, M., Cune, M.S., de Haan-Visser, W., Atema-Smit, J., Busscher, H.J., van der Mei, H.C. (2019). Biofilm composition and composite degradation during intra-oral wear. *Dental Materials*, 35 (5), 740-750.