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Effect of Acidic Drinks on Flexural Strength, Microhardness, and Roughness of Composite Resins

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ABSTRACT

Introduction: Currently the intake of acidic drinks has shown an important effect on dental tissues' demineralization; additionally, these beverages may also affect some properties of the composite resins. **Objective:** To compare the effect of acidic drinks on the flexural strength, microhardness, and roughness of composite resins. **Material and methods:** Forty bars of nanohybrid resin (Filtek™ z250 XT Nano hybrid universal restorative, 3M™ ESPE) were made to evaluate flexural strength (ISO 4049-2019) and sixty discs, twenty for microhardness test and forty for roughness test; the samples were distributed in 3 groups according to the type of drink (G1=distilled water, G2=Coca-Cola®, G3=Power Ade®). The samples were exposed to the drinks for 14 days for 2 hours per day and stored at 37° C. Microhardness was determined using the Vickers HV scale (50gf-30s). Roughness in the Ra parameter was measured in a roughness tester (0.25mm/s- cutoff distance 0.25mm-5×). The pH of the beverages was measured with a potentiometer to relate it to the mentioned tests. **Results:** Flexural strength did not show statistically significant differences between groups; G1- 115MPa, G2- 107MPa, and G3- 102MPa, elastic modulus showed G1- 5.4GPa, G2- 6.3GPa and G3- 6.6GPa, $p<0.05$. Microhardness results showed G1- 116HV 0.05/30, G2- 105HV 0.05/30 and G3- 113HV 0.05/30. In Ra roughness values were G1- 0.46µm, G2- 0.28µm, and G3- 0.27µm, $p<0.05$. The pH was 7.01 in G1, 2.55 in G2, and 3.44 in G3. **Conclusions:** The intake of acidic drinks does not affect the flexural strength and elastic modulus of composite resins, but they do produce a change at a micrometrical level such as surface microhardness and roughness.

Keywords: Flexural strength, microhardness, roughness, composite resin, energy drinks, carbonated beverages.

INTRODUCCIÓN

The use of composite resins has increased in recent years, making them the most popular aesthetic direct filler. Composite resins consist of inorganic filler particles coated with an active silane compound that binds these particles to the organic matrix of the polymer[1,2]. In these restorations, monomers and photoinitiators are polymerized by radicals, which result in highly aesthetic materials with excellent mechanical properties[3]. Therefore, having a high degree of conversion of monomer to polymer provides the restoration with better physical properties such as resistance to bending, compression, wear, dimensional, and color stability[4].

Frequent and prolonged exposure of teeth to acidic beverages is widespread. In dental tissues, this exposure produces decalcification, white spot lesions, and/or pigmentation[5–8]. Since soft drinks and energy drinks contain multiple types of acids and sugars[9,10], their associated pH values range from 2.3 to 3.4, which, from the start, leaves our body with harmful effects when

ingested and, consequently, impairs our general health; in addition, the salivary pH becomes more acidic and therefore the risk of enamel erosion and demineralization increases[11]. This could also occur with composite resins due to excessive consumption of these drinks. Due to the importance of physical and surface properties in the clinical results of composite resins, the objective of this work is to compare the effect of acidic drinks on the flexural strength, microhardness, and roughness of composite resins.

MATERIALS AND METHODS

An experimental study was conducted to determine the sample size; Tang's method was used to calculate the sample size needed to achieve a power of 80% and a confidence level of 95%[12,13]. The sample size "n" for each test is indicated in the respective test section. A pilot study was conducted to estimate these parameters. The power of this method is the probability of rejecting a false null hypothesis, it determines the number sample based in the *F* statistic, which divides the mean square between groups by the mean square within the groups, obtained from an ANOVA test. Each test differs depending on the degrees of freedom. The characteristics of the materials used in this study are shown in Table 1.

Table 1.
Test materials used in the study

Material	Composition	Manufacturer	Batch
Composite resin Filtek™ Z250 XT Nano hybrid universal restorative	Surface-modified silica/zirconium and 20 mm surface-modified silica particles. 81.8% inorganic filler, with a particle size of 20 nm for silica and 3 µm for silica/zirconium. BIS-GMA, UDMA, BIS-EMA, PEGDMA Y TEGDMA.	3M™ ESPE, USA	NF38178
Distilled water	Distilled water	JT Baker, Mexico.	SK28C36S
Coca-Cola®	Carbonated water, sugar, high fructose syrup, class IV caramel, phosphoric acid, sucralose flavorings (4.4 mg/100 g), and caffeine.	Coca-Cola®, Mexico	717RIQ
Power Ade®	Water, high fructose syrup, citric acid, Power Ade concentrate blackberry flavor, potassium citrate, sodium chloride, magnesium chloride, calcium chloride, tripotassium phosphate, blue 1, vitamins B3, B6, and B12.	Coca-Cola®, Mexico	886H663119

To evaluate the pH, 10 mL of each of the 3 solutions: distilled water, Coca-Cola®, and Power Ade® were placed in a 50 mL beaker, the pH of the beverages was measured with a pH meter (4-Star Benchtop pH/ISE Meters, Thermo Scientific Orion, USA) which was previously calibrated with buffer solutions at 4.0, 7.0 and 10.0 pH. Results are shown in Table 2.

Thirty samples of nanohybrid composite resin (Filtek™ z250 XT Nano hybrid universal restorative, A2 shade, 3M™ ESPE) were made with stainless-steel molds (2 mm wide × 2 mm deep × 25 mm long), according to the ISO 4049-2019 standard[14] to evaluate the flexural strength (Figure 1. A). A celluloid strip was placed between the tile and the stainless-steel mold, and a silicone oil, chemically inert separator was placed on the walls of the stainless-steel molds, with the help of a micro brush, to prevent them from adhering to the mold and allow their removal

(Figure 1. B). The resin was placed directly into the mold and compacted with a Titanium Aluminum Nitride (AlTiN) coated resin spatula (Hue-Friedy, TNCIGFT1, Ill, USA) to avoid gaps and/or bubbles inside, and to fill evenly until slightly overfilled. Another strip of celluloid was placed on top to press and remove excess (Figure 1. C). Each sample was polymerized with a LED curing lamp (Bluephase® N MC, 800 mW/cm^2, Ivoclar Vivadent®, Austria) (Figure 1. D) fully attached to the sample at 0 mm distance; the samples were polymerized in 5 zones for 20 s each and along the entire sample, so that each irradiated zone overlapped half of the previous polymerized zone. The mold was separated to release the sample and obtain a rectangular prism (Figure 1. E). The samples were lightly polished on each side with silicon carbide paper (320 grit SiC paper) until the resin edges were removed. The samples were divided into 3 groups (n=10) –G1- distilled water, G2- Coca-Cola® and G3- Power Ade®. The experimental groups were placed in the drinks for 2 hours daily and stored in distilled water for 14 days at 37° C. The flexural strength in each group was evaluated after 24h according to the ISO 4049-2019 standard[14]. They were subsequently subjected to a 3-point bending test on a universal testing machine (INSTRON, Mod. 4465, Norwood, MA, USA) at 0.75 mm/min cross-speed.

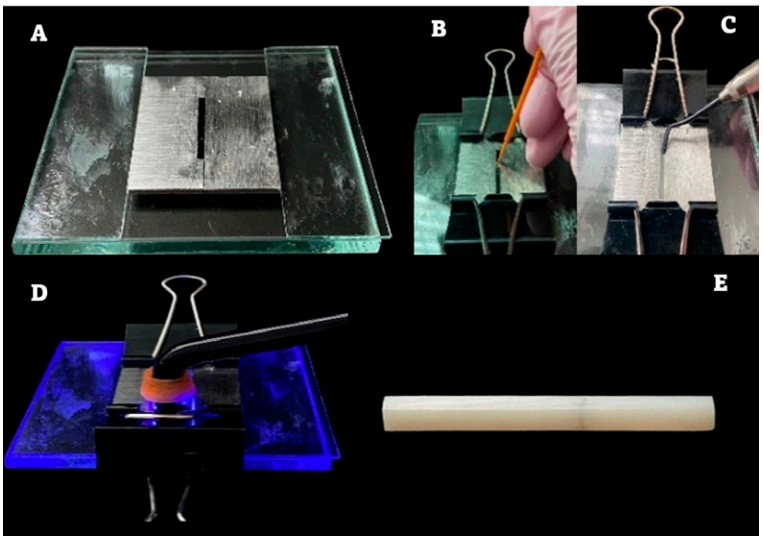

Figure 1. Preparation of flexural strength samples. A. Tile assembled with stainless steel molds. B. Silicone oil placed on the walls of the molds. C. Resin compaction in the molds. D. Direct photopolymerization. E. Final sample

To evaluate the microhardness, fifteen nanohybrid resin discs (Filtek™ z250 XT Nano hybrid universal restorative, A2 shade, 3M™ ESPE) with a diameter of 5 mm and a height of 1.5 mm were made, polymerized with the same monowave LED lamp previously mentioned. The discs were randomly divided into the 3 groups (n=5) previously described. The samples were exposed for 2 hours daily into the beverages for 14 days at 37°C, and the microhardness was evaluated on the Vickers scale with a microhardness meter (Microhardness tester MHT2, Matsuzawa Seiki Co. Japan) (Figure 2. A), exerting a test load of 50 gf for 30 seconds. Samples were placed under support pliers to ensure that the used force was only exerted on the sample, keeping results as accurate as possible (Figure 2. B). The test loads were applied in 10 different locations of the sample, per sample, to get a final n=50.

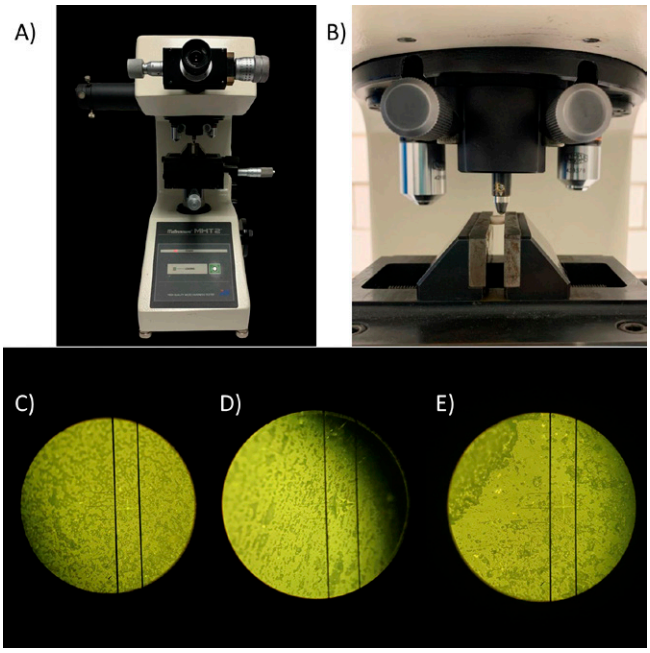

Figure 2. Microhardness test. A. Microhardness tester. B. Sample mounting. Microscope images showing the indentation during microhardness testing. C. G1=Distilled water, D. G2=Coca-Cola®, E. G3= Powerade®

Finally, for roughness evaluation, thirty discs of resin composite (Filtek™ z250 XT Nano hybrid universal restorative, A2 shade, 3M™ ESPE) of 5 mm diameter and 1.5 mm high, polymerized with an LED curing lamp (Bluephase® N MC, 800 mW/cm^2, Ivoclar Vivadent®, Austria); the samples were lightly polished with silicon carbide paper (1500 and 2000 grit SiC paper). The samples were divided into 3 groups (n=10). Ra parameter was measured on the surfaces to determine roughness with a roughness tester (Mitutoyo SJ-301, Mitutoyo American Corporation, USA) previously calibrated (Figure 3. A) at a speed of 0.25mm/s and a cutoff distance of 0.25mm-5×. The evaluated surface was fixed with double-sided tape to prevent movement of the sample and parallelized to the roughness detector tip (Figure 3. B). The measurement was carried out in triplicate in each sample, in three different areas, with a final number sample of n=30, per group.

The data were analyzed in the statistical program SPSS v.24, and the Shapiro Wilks test was used to probe the normality of data ($p > 0.05$). Subsequently, an ANOVA test was performed followed by *post hoc* Tukey test for multiple comparisons. A confidence interval of 95% was used.

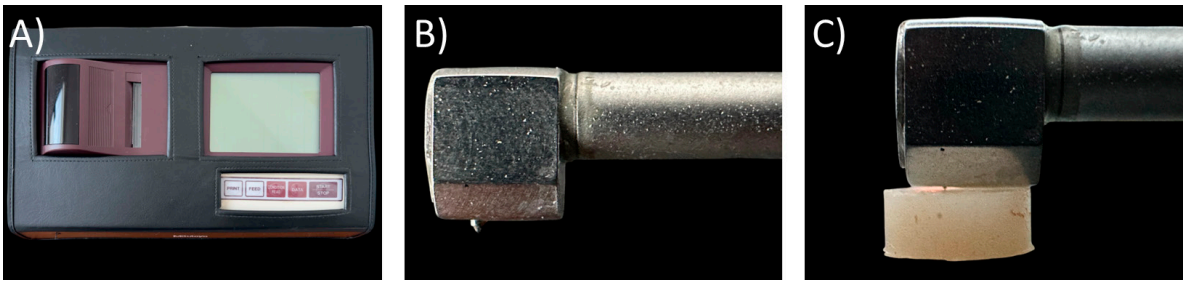

Figure 3. Roughness test. A. Roughness tester. B. Detector tip with a diamond stylus. C. Sample with detector tip.

RESULTS

The results of the evaluated properties are displayed in Table 2. When comparing the average flexural strengths (Table 2), no decrease in flexural strength was observed in the three groups. G1 obtained 109.9 MPa, G2 112.8 MPa and G3 102.9 MPa (ANOVA $p > 0.05$). Regarding the elasticity modulus, group G1 obtained 5.4 GPa, G2 showed 6.3 GPa, and G3 - 6.5 GPa (ANOVA $p > 0.05$). No statistically significant differences were found in these two tests.

Table 2.
Chemical and physical testing results

	pH	Flexural strength (MPa) n=10	Elasticity modulus (GPa) n=10	Microhardness (HV) n=50	Roughness (Ra) n=30
G1-Distilled water	7.01	109.9 ± 26.3[a]	5.4 ± 1.4[a]	116 ± 3[a]	0.46 ± 0.16[a]
G2- Coca-Cola®	2.55	112.8 ± 28.6[a]	6.3 ± 0.7[a]	105 ± 3[b]	0.28 ± 0.08[b]
G3 -Power Ade®	3.44	102.9 ± 33.9[a]	6.5 ± 1.3[a]	113 ± 6[a]	0.27 ± 0.11[b]
ANOVA	-	$p > 0.05$	$p > 0.05$	$p < 0.05$	$p < 0.05$

The mean and standard deviation are indicated. Means are compared between drinks per column, and different superscript lowercase letters indicate statistically significant differences (ANOVA $p < 0.05$, Tukey *post hoc* test $p < 0.05$).

The most representative images of the indentations of the microhardness test are observed in Figure 2C-E. In the microhardness measurements, G1 had a value of 116 0.05/30 HV, G2 showed 105 0.05/30 HV, and G3 exhibited 113 0.05/30 HV (ANOVA $p < 0.05$, *post hoc* Tukey $p < 0.05$), Table 2.

Roughness results showed that G2 and G3 are smoother in comparison to G1; G2 exhibited 0.28 µm and G3 0.27 µm, and both found statistical differences with G1 that show 0.46 µm, considering Ra measurement (ANOVA $p < 0.05$, *post hoc* Tukey $p < 0.05$), Table 2.

DISCUSSION

Currently, the prevalence of consumption of fast food and soft drinks with high sugar content has been discussed. In the United States, between 2013 and 2016, more than 36% of adults consumed fast food in a daily basis[15], and this consumption includes the intake of carbonated beverages[16]. In addition, an increase in the consumption of sports and energy drinks has been observed among the general population, which has raised questions about the erosive potential of these drinks on dental hard tissues[8,17], as well as their effects on the clinical performance of the materials used[18].

It is important to mention that composite resins are used in many restorative procedures, which can make them susceptible to being affected by such beverages. Despite the fact that these polymers are biomaterials that provide good biomechanical properties, these properties may vary depending on the filler content as it has been correlated with curing depth, color stability, hardness, flexural strength, and elasticity modulus. Considering the widespread use of resin composite and the harsh conditions in the oral environment, these resin composites must be resistant to degradation. However, they have been shown to suffer degradation over time

under acidic conditions, this can be predicted by changes in surface topography and roughness, decreased hardness and wear resistance, and loss of substance of the resin composite[19].

Gradinaru *et al*.[20] in 2023 showed that the clinical performance of composite resins depends not only on the structure of the biomaterial but also on the environment to which it is exposed. In this sense, the surface condition analysis provides relevant data on the effect of acidic beverages and structural degradation of the material. Several authors[19,21–23] have conducted various studies showing a decrease in the microhardness of the surface of different restorative materials when exposed to different acidic media, in which similar results were observed to those obtained in the present study, where the composite resin showed a decrease in hardness after immersion in acidic beverages[24,25].

Barve *et al*.[26,27] explained two main mechanisms of polymer degradation; the first one is by hydrolysis, which is passive, and the second one is by enzymatic reactions, which are active. Among them, the passive hydrolysis of the polymer is the most important. Increased water absorption by the resin composite could be responsible for its hydrolytic degradation and decreased surface hardness. Secondary causes could be sustained silica loss after matrix degradation, splitting of the matrix-filler interface, and internal damage. These deficiencies are expected to decrease the mechanical properties of the material, such as flexural strength, which is a clinically relevant property for restorative materials, as it mimics the use of composites in areas that withstand high stresses[28]. However, studies conducted by Scribante *et al*.[29,30], where they evaluated resin composite with different sizes and quantities of filler particles in terms of flexural strength and elastic modulus, found that the immersion in an acidic medium decreased both properties of the resin composite (Composite Universal 3M™ Filtek™ Supreme XTE, 3M™ ESPE), while other materials were not affected by immersion. In this sense, any significant correlation was found between the acidic environment and the elastic modulus of the materials used.

It has been mentioned that low surface hardness values are mainly related to inadequate wear resistance and in consequence affected roughness, which can cause stains, plaque buildup, gingival irritation, and recurrent caries[31]. Among the results obtained by some authors[32,33], an increase in roughness has been observed when exposing the material to different acids while others did not find any difference[34], which is consistent with the results obtained in the present study when evaluating after 14 days of exposure affect roughness and microhardness. On the other hand, Badra *et al*.[35] observed an increase in surface roughness in early periods, but it decreased over time when evaluated at 30 and 60 days. Therefore, it is important to mention that all the results are conditioned by the duration of the exposure time, the pH of the medium, and the composition of the material chosen for each evaluation, which may explain the differences in the obtained results.

On the other hand, a limitation of this *in vitro* model includes other factors, such as salivary buffer capacity and acquired film, which have not been considered to give a more realistic assessment of structural degradation. That could be a predisposing factor to bacterial colonization, and in turn, potentially increase the risk of oral diseases. Studies that focus on the real impact of the environment to which materials such as resin composite are exposed; are of utmost importance to promote improvements in formulation, and therefore, increase the degree of reliability, quality, and longevity of restorations over time.

CONCLUSIONS

Among the limitations that this study presents, it is concluded that the ingestion of acidic drinks does not affect the macro-mechanical properties such as flexural strength and elasticity modulus of composite resins; however, it does affect micromechanical properties such as microhardness and roughness.

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