

# Microhardness Evaluation of Microhybrid Versus Nanofilled Resin Composite After Exposure to Acidic Drinks

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**ABSTRACT** **Objective:** To investigate the effect of two acidic agents on microhardness of nanofilled and microhybrid resin composite materials. **Materials and Methods:** 70 resin composite discs (10 mm diameter and 2 mm thick) were prepared from 2 resin composites (35 from each type), nanofilled (Z350XT) and microhybrid (Z250), 3M/ESPE. Ten discs (5 from each group) were used as control (tested at 1 h before immersion). Each group was divided into 3 subgroups ( $n = 10$ ) according to the storage media: distilled water (control), box-type cola and orange juice. Each subgroup was further subdivided into 2 divisions ( $n = 5$ ) according to microhardness testing at 24h and 7days after immersions. Digital Vickers Microhardness Tester (FM-7, Future Tech, Tokyo, Japan) was used to measure surface microhardness with a Vickers diamond indenter. The surface of the specimens received a load of 200 g for 10 seconds. Three indentations not less than 1 mm from each other were placed on the surface of all specimens. Vickers hardness number (VHN) was calculated for each indentation Data were statistically analyzed using one-way ANOVA followed by Newman-keuls tests ( $P \leq 0.05$ ). **Results:** Orange juice showed statistically significantly the lowest VHN mean value (92.7) followed by the Cola group (95.15) then the water group (104.02) compared to the control group (117.4). Microhybrid composite groups showed statistically significant higher VHN mean value (108.1) than the nanofilled composite (100.2). The 7days groups showed statistically significant lower VHN mean value (97.3) than 24h groups (106.6). **Conclusions:** All storage media reduced hardness of resin composites with orange juice showing the highest reduction in hardness values. Microhybrid is more resistant to degradation than nanofilled composite. Over time, microhardness of resin composites decreased progressively.

**KEYWORDS:** Acidic drinks, composite resins, microhardness

## INTRODUCTION

Resin composites are becoming more popular in restorative dentistry because of their superior esthetic outcomes and good mechanical properties. It is considered to be mandatory for these restorations to have a long lifespan performance inside the oral. Hence there are different forms of destructive processes that can affect tooth surface irreversibly other than caries in the oral environment.<sup>[1]</sup> which can be referred to as abfraction, abrasion, demastication, erosion and resorption.

Acidic soft drinks can cause demineralization of the tooth surface; thus consumption of citric fruits, acidic drinks, and liquid medications are considered to be the main etiological and aggravating causes for dental erosion. The consumption of soft drinks and fruit juices increased substantially in adolescents and children.

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Resin composite filling materials are susceptible to softening by organic acids and various food and liquid constituents.<sup>[2]</sup> They are exposed to many compounds (alcohols, acids, salts, alkalis, etc.), while eating and drinking, moreover it is directly affected by the frequency of consumption of these drinks.<sup>[3]</sup> This may change microhardness which is an extremely critical property of restorations, that directly affects the physicochemical properties as compressive strength and abrasion resistance. Thereby, undermining the quality of restoration and accelerates the necessity of replacement.<sup>[4,5]</sup> Basic properties of dental restorations composite materials are directly affected by noxious factors inside the patient’s mouth. These factors are either thermal, mechanical, or chemical. Chemical factors could be classified into internal (gastric acids in frequent vomiting) and external (e.g., acidic nutrients, acids from the air or chlorinated water in the swimming pool). These acidic beverages with low pH of can lead to erosive wear on the composite materials.<sup>[6]</sup>

The mechanical properties of resin composites are greatly depending on the concentration and particle size of the filler.<sup>[7,8]</sup> Hybrid and microhybrid resin composites have a broad range of particle sizes, macrofillers of an average size of 0.1 6.0 µm and microfillers with a particle size of 0.01-0.05 µm.<sup>[9]</sup> Thus allow high filler loading leading to higher strength.<sup>[10]</sup>

Recently nanofilled composites were developed to offer optimized physical and mechanical properties, so it can fulfill the need for a universal restorative material; thus they were indicated for posterior and anterior teeth. the distribution and packing of nanofillers in these classifications of composites can be attributed to the

great improve their resistance to chemical challenges in the oral environment.<sup>[11]</sup>

Several *in vitro* studies were done to examine the erosive capabilities of different beverages which are considered to have a mixture of acids. Different methodical approaches as profilometric evaluation of surface loss, electron microscopy, or determination of microhardness.

Therefore the purpose of this study is to evaluate and compare the effect of two acidic beverages on microhardness of a microfilled and nanofilled resin composites and investigate the effect of aging during storages time on microhardness of both composite resins. Two null hypotheses were tested:

- 1- There would be no influence for the type of acidic beverage on the microhardness of composite resin materials.
- 2- There would be no difference in microhardness values for both tested composite resin materials.

## MATERIALS AND METHODS

Two groups of 70 resin composite discs were prepared from two composite resin materials (35 from each type), nanofilled (Z350XT) and microhybrid (Z250), 3M/ESPE, St Paul, MN, USA of shade A2 [Table 1]. Ten discs (5 from each group) were used as baseline (tested at 1h before immersion). Each group was divided into 3 subgroups (*n* = 10) according to the storage media: distilled water (control), box-type cola (Coca-cola company, Riyadh, KSA) and orange juice (Al Rabie Saudi Foods Co., KSA), with a daily renew of the acidic beverage [Table 2]. Each

**Table 1: Composite resin materials**

Resin Composite Classification	Filtek Supreme Z350XT, (3M/ESPE) Nanofilled	Filtek Z250, (3M/ESPE) Microhybrid
Monomer	Bis-GMA, Bis-EMA, UDMA, and TEGDMA	Bis-EMA, UDMA, and Bis-GMA
Filler type	ZrO2/SiO2 nanocluster, SiO2 nanofiller	Zirconia/Silica
Filler Size µm	5-20 nm with 20 nm silica filler	0.6 µm
Filler weight (%)	78.5	82

**Table 2: Grouping of specimens**

70 composite resin discs (10mm diameter and 2mm thick)			
35 nanofilled (Z350XT) discs		35 microhybrid (Z250) discs	
5 as control	1H	5 as control	1 H
10 in distilled water (PH 5.8)	5 discs 24 H 5 discs 7 days	10 in distilled water (PH 5.8)	5 discs 24 H 5 discs 7 days
10 in box type orange (PH 3.5)	5 discs 24 H 5 discs 7 days	10 in box type orange (PH 3.5)	5 discs 24 H 5 discs 7 days
10 in box type cola (PH 2.5)	5 discs 24 H 5 discs 7 days	10 in box type cola (PH 2.5)	5 discs 24 H 5 discs 7 days

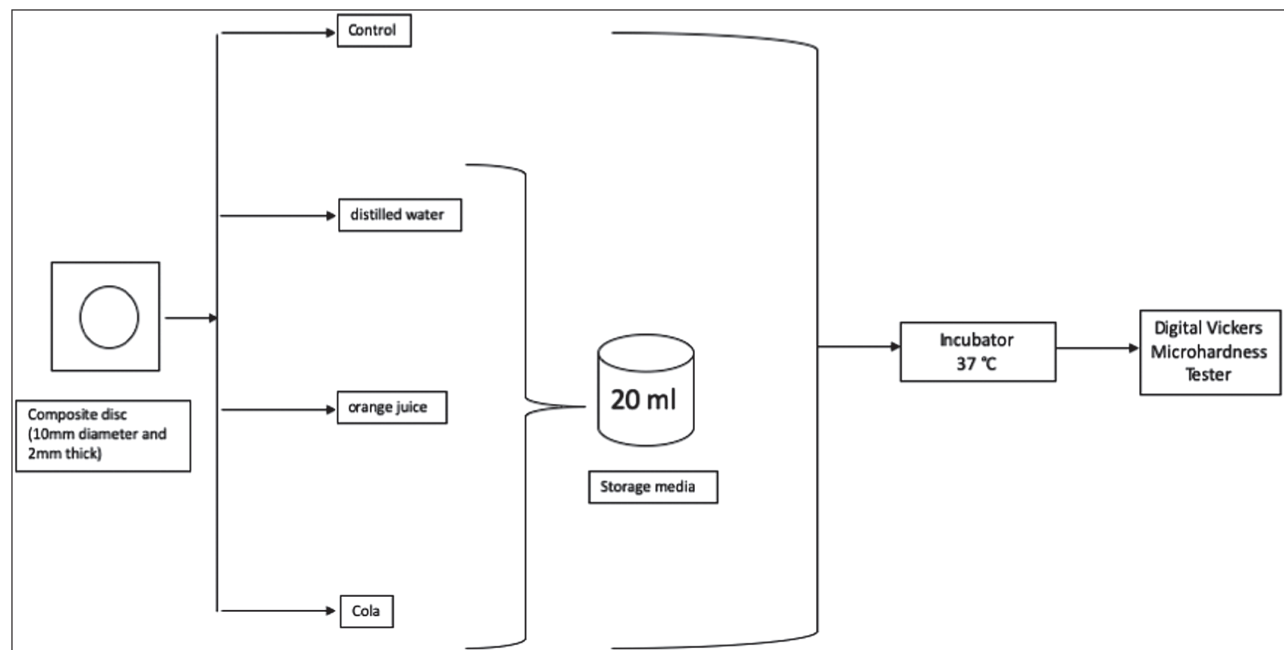


Figure 1: Schematic illustration of the research method used in this study

subgroup was further subdivided into 2 divisions ( $n = 5$ ) according to microhardness testing at 24h and 7days after immersion. The composite discs were stored in 20ml of immersion media in an incubator to ensure standardization at 37°C for 1h, 24hr or 7 day [Figure 1].<sup>[12]</sup> The pH information of the acidic bottled beverages used in this study were not provided by the manufacturers. Hence, a digital pH meter was used to calculate the pH of each solution. The measured pH values for the orange juice and cola were 3.5 and 2.5 respectively, both of which were less than the critical pH value of 5.5. The storage media were changed and the pH measured every 24 hours.<sup>[13]</sup>

#### PREPARATION OF SPECIMENS

The disc shaped specimens were prepared using a Teflon mold (10mm diameter and 2mm thick). Mylar Strips covered the top and bottom sides of the mold, then a thin glass slide was added to obtain a flat composite resin surface. Excess material was removed by applying pressure on the glass slide. Then polymerization of the specimens was carried out after removal of the glass slide for 20 seconds, according to the manufacturer's recommendations, employing a light emitting diode (LED) device (Blue phase; Ivoclar Vivadent, Schann, Lichtenstein) with a light intensity of 600 mW/cm<sup>2</sup>, measured with a light meter. The light curing tip was placed at zero distance over each specimen after removal of the glass slide.

Three grades of Sof-Lex discs (3M ESPE, St.Paul, MN, USA) were used with a low-speed handpiece

under a wet environment to polish the top surface of the specimens. The top surface was marked to facilitate identification during the microhardness measurements.

At the end of each storage period, the top surface of the samples in contact with the erosive action of the oral cavity was subjected to microhardness testing.<sup>[14]</sup>

#### MICROHARDNESS MEASUREMENTS

Digital Vickers Microhardness Tester (FM-7, Future Tech, Tokyo, Japan) was used to measure surface microhardness of the specimens. A number of three indentations were made with a diamond indenter using a load of 200g for 10 seconds each, 1 mm apart from each other. VHN was calculated for each indentation, then the mean of three values for each specimen was recorded.<sup>[15]</sup>

#### STATISTICAL ANALYSIS

Data were statistically analyzed using one-way ANOVA for comparing variables (Composite resin, immersion solutions and time) affecting mean values. One way ANOVA test was followed by Newman-keuls to detect significance between subgroups. The significance level was set at ( $P \leq 0.05$ ). Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for Windows.

#### RESULTS

Mean VHN values and standard deviations obtained 1 hour after fabrication (baseline); after 24-hours and 7-days in different storage media, and the difference between the assessments are presented in [Table 3].

**Table 3: VHN results (Mean ± SD) for both composite materials as function of immersion solutions and time**

Variables		Composite resin			Statistics
		Nano-filled composite	µ-Hybrid composite	Total	
		Mean± SD	Mean± SD	Mean± SD	
<i>Control</i>		116.93 <sup>A</sup> ±6.6	117.85 <sup>A</sup> ±2.3	117.39 <sup>A</sup> ±4.5	0.7694 ns
<i>Water</i>	<i>24 hr</i>	107.98 <sup>B</sup> ±3.2	114.66 <sup>A</sup> ±4.97	111.32 <sup>B</sup> ±4.1	0.007*
	<i>7 days</i>	99.49 <sup>B</sup> ±4.1	108.55 <sup>B</sup> ±8.9	104.02 <sup>C</sup> ±6.5	0.0112*
<i>Cola</i>	<i>24 hr</i>	104.15 <sup>B</sup> ±8.8	106.42 <sup>B</sup> ±14.2	105.28 <sup>C</sup> ±11.5	0.6942 ns
	<i>7 days</i>	89.89 <sup>C</sup> ±2.4	100.41 <sup>C</sup> ±3.6	95.15 <sup>D</sup> ±3	<0.0001*
<i>Orange</i>	<i>24 hr</i>	91.66 <sup>C</sup> ±2.3	100.41 <sup>C</sup> ±3.6	103.24 <sup>C</sup> ±5.1	<0.0001*
	<i>7 days</i>	81.49 <sup>D</sup> ±2.6	93.91 <sup>D</sup> ±9.6	92.7 <sup>D</sup> ±6.1	0.5466 ns
<i>Total</i>		100.23±4.3	108.1±7.3		0.0350*
<i>Statistics</i>	<i>P value</i>	<0.0001*	<0.0001*	0.0355*	

different letter in same column indicating significant difference ( $p < 0.05$ ) ns; non-significant ( $p > 0.05$ ); \*; significant ( $p < 0.05$ )

Orange juice showed statistically significantly the lowest VHN mean value (92.7) followed by the Cola group (95.15) then the distilled water group (104.02) compared to the control group (117.4). Microhybrid composite groups showed statistically significant higher VHN mean value (108.1) than the nanofilled composite (100.2). The 7days groups showed statistically significant lower VHN mean value (97.3) than 24h groups (106.6).

## DISCUSSION

This study was intended to evaluate the surface microhardness change of the top surface of two composite resin materials after storage in two different acidic beverages: Coca-Cola (pH = 2.5) and orange juice (pH = 3.5) for 1 day and 7 days, at 37°C.<sup>[14,16]</sup> The storage in the acidic medium was continual, in order to demonstrate long-term exposure to acidic drinks in the oral cavity. The consumption of Coca-Cola for a month was found to be similar to storage for 1 day.<sup>[17]</sup>

Composite resin specimens were prepared 2mm thick to ensure maximum polymerization. Adequate polymerization is critical for the success of composite resin restorations, as incomplete curing is accountable for water sorption, reducing wear resistance and strength, and leaching of residual monomer<sup>[16,18]</sup>

The use of distilled water in the present study, was an attempt to simulate the moist oral environment provided by the saliva. Distilled water was used to mimic the flushing action of salivary flow as the artificial saliva does not reflect a clinically relevant surrounding.<sup>[19]</sup> On the other hand, Turssi, *et al*,<sup>[20]</sup> studied the influence of immersion media on the surface characteristics of composite resin restorations and found comparable outcomes for distilled water and artificial saliva.

This study showed that the immersion of both types of resin composite discs in distilled water decreased the microhardness significantly after 24 hours for the

nanofilled composite and after 7 days for both nanofilled and microhybrid compared to the control group that can be due to water sorption that has been shown to be the main reason for the onset and propagation of microcracks, surface flaws, debonding of filler particles, release of unreacted monomers, and plasticization over time. In addition, the hydrophilic characteristics of Bis-GMA, UDMA and Bis-EMA may contribute to the reduction in surface hardness of specimens stored in distilled water.<sup>[21,22]</sup>

On the other hand, composite resin materials exposed to acidic beverages reported matrix decomposition, surface erosion and dissolution.<sup>[23]</sup> The acids present in these beverages penetrated the resin matrix which soften the Bis-GMA and facilitate the release of unreacted monomers.<sup>[2]</sup> UDMA, TEGDMA AND Bis-GMA are very susceptible to absorption and solubility which may lead to softening and degradation of the resin matrix.<sup>[24]</sup>

The results of this study revealed that the two composite resin materials exhibited a significant decrease in surface microhardness values after the 24-hours and 7-days storage periods, irrespective of the beverage used. However, specimens that were stored in acidic beverages showed higher surface microhardness decrease when compared to the specimens stored in distilled water after both storage periods. These results were in agreement with Khan *et al.* and Al-Shekhli and Aubi.<sup>[25,26]</sup> Thus, the first null hypothesis, which proclaimed that “there would be no influence for the type of acidic beverage on the surface microhardness of composite resin materials”, was rejected.

In this study, the orange juice with higher pH value than cola showed statistically significant lower microhardness values compared to distilled water and cola groups.

According to previous studies, acidic solutions result in degradation of restorative materials,<sup>[27-30]</sup> and

consequent decrease in their hardness. It has been noted that the erosive potential of a solution is not dependent only on the low pH, the titratable acidity, acid concentration, type of acid, the immersion time in the acidic beverages and the composition of the beverage are also of great consideration.<sup>[31]</sup> The titratable acidity of orange juice was found higher than that of the cola drink which is an important factor that can alter the material surface microhardness.<sup>[32]</sup>

In other studies, it has been shown that the erosive potential of the acidic beverages may depend on their chelation properties and the frequency and duration of consumption.<sup>[33]</sup> In citric acid solutions, the degradation depends on the diffusion and the chelation between the acid anions and the eluted particles.<sup>[34]</sup>

The results were also in agreement with Ortengren *et al.* who attributed the significant increase in solubility of composite resin materials immersed in citric acid to the pH of the solution and the storage time.<sup>[35]</sup> On the other hand, the immersion of composite resin in organic acids promotes polymer softening caused by leaching of filler particles.<sup>[36,37]</sup>

Also the results of this study were in accordance with Nicholas *et al.*,<sup>[38]</sup> who found the immersion in orange juice reduced the microhardness values more than the immersion in Coca-cola soft drink although the latter's pH was less than that of orange juice. They claimed that phosphoric acid appeared relatively less aggressive than citric acid and able to buffer aqueous acid solutions and the extent of this buffering capability varied with storage duration.

In general, regardless of the type of low pH beverages used, both composite resin restorative materials showed statistically significant reduction in surface microhardness results after 7-days immersion duration than after 24-hours. This may be due to liquid absorption, and the plasticizing effect of water molecules inside the resin matrix,<sup>[13]</sup> leading to expansion of the matrix and softening of the resin polymer, decreasing the frictional forces between the polymer bands.<sup>[39,40]</sup>

Two different types of resin composites were evaluated, a nanofilled type that provides better brightness, good optical properties and lower wear rates, and a microhybrid type which provide proper wear resistance and satisfactory mechanical properties.<sup>[41]</sup>

The results of this study showed significant decrease in microhardness of nanofilled and microhybrid composite resin when stored in different acidic beverages for 24 hours and 7 days compared to the control and distilled water group which was in agreement with Maganur *et al.*,<sup>[30]</sup> they stated that the

low pH drinks have deleterious effects on the long survival of the restorative materials. Other investigators have concluded that the exposure of composite resin to acidic beverages can have a detrimental impact on their mechanical properties.<sup>[42-44]</sup>

The nanofilled composite showed statistically significant lower microhardness values compared to the micro hybrid when immersed in orange juice that may be due to the high amount of organic matrix in nanofilled composites that may result in higher ability to water sorption and material disintegration.<sup>[45]</sup> In addition, zirconia/silica fillers were found to be liable to water attack, and the smaller surface area of spherical shaped zirconia/silica fillers bonded to the resin matrix leached more easily.<sup>[46,47]</sup> Thus, the second null hypothesis, which proclaimed that “there would be no difference in microhardness values for both tested composite resin materials” was rejected.

On the other hand, researchers have claimed that the type of storage media and the constituents of dental resins are critical aspects concerning the degradation of composite resins.<sup>[48]</sup>

Therefore, the longevity of composite resins is highly influenced by the inherent properties of the materials and the surrounding media.<sup>[49]</sup> Composite resin restorations are exposed to disintegration resulting from the effect of chemical constituents in the saliva, food, beverages, and daily mouthwashes, even in the absence of mechanical loads or abrasive forces.<sup>[50]</sup>

## CONCLUSIONS

Under the conditions of this study:

1. All storage media reduced hardness of resin composites with orange juice showing the highest reduction in hardness values.
2. Microhybrid is more resistant to degradation than nanofilled composite.
3. Over time, microhardness of resin composites decreased progressively

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Not applicable.

## FINANCIAL SUPPORT AND SPONSORSHIP

Nil.

## CONFLICTS OF INTEREST

There are no conflicts of interest.

## AUTHORS CONTRIBUTIONS

Not applicable.

## ETHICAL POLICY AND INSTITUTIONAL REVIEW BOARD STATEMENT

Not applicable.

**PATIENT DECLARATION OF CONSENT**

Not applicable.

**DATA AVAILABILITY STATEMENT**

All data generated or analyzed in this study are included in this article.

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