Surface hardness evaluation of different composite resin materials: influence of sports and energy drinks immersion after a short-term period

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ABSTRACT

Objectives: This study evaluated the effect of sports and energy drinks on the surface hardness of different composite resin restorative materials over a 1-month period.

Material and Methods: A total of 168 specimens: Compoglass F, Filtek Z250, Filtek Supreme, and Premise were prepared using a customized cylindrical metal mould and they were divided into six groups(N=42; N=7 per group). For the control groups, the specimens were stored in distilled water for 24 hours at 37°C and the water was renewed daily. For the experimental groups, the specimens were immersed in 5 mL of one of the following test solutions: Powerade, Gatorade, X-IR, Burn, and Red Bull, for two minutes daily for up to a 1-month test period and all the solutions were refreshed daily. Surface hardness was measured using a Vickers hardness measuring instrument at baseline, after 1-week and 1-month. Data were statistically analysed using Multivariate repeated measure ANOVA and Bonferroni's multiple comparison tests (α =0.05).

Results: Multivariate repeated measures ANOVA revealed that there were statistically significant differences in the hardness of the restorative materials in different immersion times (p<0.001) in different solutions (p<0.001). The effect of different solutions on the surface hardness values of the restorative materials was tested using Bonferroni's multiple comparison tests, and it was observed that specimens stored in distilled water demonstrated statistically significant lower mean surface hardness reductions when compared to the specimens immersed in sports and energy drinks after a 1-month evaluation period (p<0.001). The compomer was the most affected by an acidic environment, whereas the composite resin materials were the least affected materials.

Conclusions: The effect of sports and energy drinks on the surface hardness of a restorative material depends on the duration of exposure time, and the composition of the material.

Key words: Surface hardness. Composite resins. Sports drinks. Energy drinks.

INTRODUCTION

The consumption of sports and energy drinks has gained high popularity among the adolescent population, especially 18- to 35-year-olds in recent years^{14,25}. Although the purpose of those drinks is to enhance performance and endurance and prevent dehydration for individuals involved in physical activity, they are being widely consumed by the

general population instead of carbonated drinks⁸. However, previous studies have shown that these beverages potentially cause dental $erosion^{8,15}$ and, due to their acidity, may be detrimental to the properties of restorative materials.

The use of resin-based restorative materials in dentistry has substantially increased over the past few years because of their good aesthetic appearance, improvements in formulations,

ease of handling, and ability to establish a bond to dental hard tissues^{5,18}. The mechanical property of the dental composites largely depends on the concentration of filler particles and particle size²⁸. Recent advancements on the organic matrix and inorganic fillers have led to the development of new materials with reduced particle size and increased filler loading, and have resulted in improved mechanical properties and aesthetics on the current composite resin materials^{23,27}.

To be clinically successful, restorative materials are required to have long-term continuousness²², a quality "which is strongly influenced not only by the intrinsic characteristics of the materials, but also by the environment to which they are exposed to^{18,29,36}." But the oral cavity is a complex, aqueous environment where the restorative material is in contact with saliva^{12,22}. In addition, other factors such as low pH due to acidic foods and drinks may influence the material's mechanical and physical characteristics¹³. In a clinical environment, a material's decrease of hardness may contribute to its deterioration²⁶. However, "under *in vivo* conditions, composite resin materials may be exposed either discontinuously or continually to chemical agents found in saliva, food, and beverages^{32,36,37}." Consequently, in the short- or long-term, these conditions may have a deleterious effect on the polymeric network, modifying its structure physically and chemically³⁴.

Physical characteristics of restorative materials are an important concern when determining suitable restorative materials because they strongly influence the clinical longevity of restorations³. One of the most important properties is the material's hardness, which correlates well with compressive strength, resistance to intra-oral softening, and degree of conversion^{5,30}. A low surface hardness value is largely related to inadequate wear resistance²⁴ and proclivity to scratching, which can compromise fatigue strength and lead to failure of the restoration¹⁸.

It has been reported that lengthy contact time with coffee, tea, mouthwashes, acidic food and low pH drinks may affect the surface hardness of resin- based composite materials^{13,20,33}. The increased consumption of sports and energy drinks among the general population has raised questions about these drinks' erosive potential on the dental hard tissues, as well as their effects on the clinical performance of restorative materials. Until now, however, there have been no studies on the effect of sports and energy drinks on the surface hardness of resin- based restorative materials. Given their high rate of consumption among younger patients, especially those who engage in physical activity, and the growing use of resin-based restorative materials for this group, this is an important omission.

Against this background, the aim of this *in vitro* study was to evaluate the effect of short-term immersion in sports and energy drinks on the surface hardness of polyacid-modified composite resin, compomer, microhybrid composite, and nanofilled composites. The tested null hypothesis were that (1) there would be no significant effect of the type of solutions on the surface hardness of the restorative materials and (2) two minutes daily exposure time in

the tested solutions for a 1-month evaluation period would have no significant effect on the surface hardness of resin-based restorative materials.

MATERIAL AND METHODS

Preparation of specimens

In the present study four types of resin-based composite materials namely; a polyacidmodified composite resin-compomer (Compoglass F, Ivoclar- Vivadent, Schaan, Liechtenstein), a microhybrid composite resin (Filtek Z250, 3M ESPE, St. Paul, MN, USA) and two nanocomposites (Filtek Supreme, 3M ESPE, St. Paul, MN, USA; Premise, Kerr-Hawe, Orange, CA, USA) of A2 shade were tested. The list of the materials with their compositions is provided in Figure 1. A total of 168 disc-shaped specimens (n=42 for each composite), 8mm in diameter and 2 mm thick, were prepared using a customized cylindrical metal mould. In order to obtain a flat polymerized surface without bubble formation, the specimens were covered on both sides (top and bottom) with a polyester matrix strip (Mylar Strip, SS White Co., Philadelphia, PA, USA) and a thin, rigid glass microscope slide (1-mm thick). Finger pressure was applied on the slide to extrude the excess material. The composite material was then polymerized through the glass slide and polyester matrix strip for 20 s, according to the manufacturer's recommendations, using a halogen light curing unit (VIP, Bisco Inc., Schaumburg, IL, USA) operating in standard mode and emitting not less than 600 mW/cm², as measured with a light meter that was placed on the curing unit before beginning the polymerization. The guide of the light curing unit was placed perpendicular to the specimen's surface and the distance between the light source and the specimen was standardized by using a 1-mm glass slide. Afterward, all specimens were stored in distilled water in a lightproof container for 24 hours at 37°C to ensure complete polymerization. The top surfaces of all specimens were serially polished with a series of three grades (medium, fine, and super-fine) of Sof-Lex disks(3M ESPE, St.Paul, MN, USA) with a slow-speed handpiece under dry conditions for 30 seconds before the surface hardness evaluation. After each polishing step, the specimens were thoroughly rinsed with water for 10 seconds to remove debris, air dried for 5 seconds, and then polished with another disk of lower grit for same period of time before the final polishing.

Surface hardness evaluation

Forty-two specimens of each restorative material were divided into six groups (n=7/group). The hardness value of each specimen was determined using a microhardness tester (Micromet 5114, Buehler, Lake Bluff, IL, USA) with a diamond Vickers indenter. Three indentations were made and measurements were obtained at different points on each specimen, with a 200 g load for a 15 s dwell time. The average value was converted into a Vickers Hardness Number (VHN). The measurements were taken automatically 1 mm from each other.

Immersion of specimens in solutions

After the baseline microhardness evaluation, 7 specimens from each experimental group were individually stored in vials containing 5 mL of distilled water (pH 6.58) for 24 hours, and kept in an incubator at 37°C as a control solution and the distilled water was renewed daily up to 1-month. The other specimens from each experimental group were individually

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immersed (n=7) in vials containing 5 mL of Powerade sports drink (The Coca-Cola Co., Atlanta, GA, USA; pH 3.79), Gatorade sports drink (The Gatorade Co., Chicago, IL, USA; pH 3.27), X-IR energy drink (Nice Trading Inc., Istanbul, Turkey; pH 3.15), Burn energy drink (The Coca-Cola Co., Atlanta, GA, USA; pH 2.67) and Red Bull energy drink (Red Bull GmbH, Am Brunnen, Austria; pH 3.54) for 2 minutes daily at room temperature $(23\pm1^{\circ}C)$. After the immersion period in the test solutions, the samples were washed with distilled water and the specimens were maintained in distilled water at 37°C during the rest of the day. The vials were sealed to prevent evaporation of both the control and test solutions. All the solutions were refreshed and the pHs of the solutions were measured daily with a pH meter (HI 221, Hanna Instruments Inc., Woonsocket, RI, USA) before immersing the specimens. For the entire experimental period, newly opened test solutions were used for each day. Thereafter, in order to evaluate the change in surface hardness over time, the microhardness test was carried out 1-week and 1-month after the start of storage for the control and immersion for the experimental groups.

Restorative	Classification	Filler	Filler	Filler Type	Filler Size	Monomer	Shade
Material		Weight	Volume			Composition	
		(%)	(%)				
Compoglass F	Poly-acid	77	55	SiO ₂ , YbF ₃ , (Ba)	1 µm	Bis-GMA,	A2
(Ivoclar-Vivadent,	modified resin			FAISi		UDMA,	
Schaan,	composite					TEGDMA,	
Liechtenstein)	(Compomer)					CDCDMA	
Filtek Z 250	Minifilled hybrid	82	60	Zirconia/silica	0.6 µm	Bis-EMA,	A2
(3M ESPE, St Paul,						UDMA,	
MN, USA)						Bis-GMA	
Filtek Supreme	Nanofilled	78.5	59.5	ZrO ₂ /SiO ₂	5-20 nm	Bis-GMA, Bis-	A2B
(3M ESPE, St Paul,				nanocluster,SiO ₂	with 20 nm	EMA,	
MN, USA)				nanofiller	silica filler	UDMA,	
						TEGDMA	
Premise	Nanohybrid	84	69	Barium alumino	Glass: 0.4	Bis-GMA, Bis-	A2
(Kerr-Hawe,				borosilicate glass,	µm, silica:	EMA, TEGDMA	
Orange, CA, USA)				silica nanofiller,	0,02 µm		
				PPF, barium			
				glass, discrete			
				nanofiller			

Figure 1- Composition of the restorative materials used in the study

Statistical analysis

Statistical analysis was performed using the Statistical Package for Social Sciences (SPSS) software, version 17.0 (SPSS Inc., Chicago, IL, USA). This study was performed to evaluate the effect of immersion times (1-week and 1-month) and different solutions on the surface hardness of different composite resins as the main effects and all possible combinations of these variables as the interaction effects (immersion times*solutions). The results were primarily analysed using the Kolmogorov-Smirnov test to determine the existence of a normal distribution. Since the data was normally distributed, the statistical analysis was performed by using a multivariate repeated measure ANOVA test on the surface hardness values of different composite resins after immersion in different solutions for 1-week and 1-month. One- way ANOVA was used to compare the effect of different solutions on the

surface hardness values of each restorative material for each evaluation period. The means were then compared by *post- hoc* pairwise Bonferroni's multiple comparison tests. Values are expressed as mean \pm SD. The statistical significance level was established at P<0.05.

RESULTS

The mean and standard deviations in the surface hardness values for the four restorative materials before and after storage in distilled water for 24 hours and immersion in any of the test solutions for two minutes daily at 1-week and 1-month are summarized in Table 1. Multivariate repeated measures ANOVA revealed that there were statistically significant differences in the hardness of the restorative materials in different immersion times (p<0.001) in different solutions (p<0.001). The interaction of the immersion time and solution was found to be significant for the Compoglass F (p=0.002) and Premise (p=0.005) restorative materials (Table 2). The results of this study reveal that the mean surface hardness values of all the restorative materials before storage in distilled water were lower than those after 1-week of storage. However, sports and energy drinks decreased the mean surface hardness values of all restorative materials after 1-week of immersion when compared to the baseline. After the 1-month evaluation period, all restorative materials presented a significantly lower surface hardness in comparison to 1-week for both the control and test solutions utilized (p<0.001).

The effect of different solutions on the surface hardness values of the restorative materials were tested using Bonferroni's multiple comparison tests, and it was observed that specimens stored in distilled water demonstrated statistically significant lower mean surface hardness reductions when compared to the specimens immersed in sports and energy drinks after a 1-month evaluation period (p<0.001). Surface hardness differences for each material among the solutions for 1-week and 1-month were evaluated by using the one-way ANOVA test. According to the results of the one-way, significant differences were detected among the solutions for the Filtek Z250 and Filtek Supreme (p<0.05), and for the other restorative materials at the 1-week period (p<0.001) and for all restorative materials at the 1month period (p<0.001) (Table 1).

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Material	Test Period	Distilled water	Powerade	Gatorade	X-IR	Burn	Red Bull	F	р	
Compoglass F	Baseline	51.73±0.6241	51.76±1.54×1	51.70±0.91a,1	51.80±1.358,1	51.79±1.49×1	51.74±0.90×1			Ī
	1 Week	51.95±2.16*1	47.06±1.69 ^{5,2}	46.06±1.15b,2	46.60±1.41b,2	44.72±1.86±2	46.64±1.60°2	15.166	<.001	
	1 Month	45.05±3.00 ⁴³	38.08±1.48 ^{1,4}	37.30±0.86%	36.75±1.074	38.10±1.29°	37.98±1.62 ¹³	25.769	<,001	
Filtek Z250	Baselne	80.16±0.65%	80.21±0.5411	80.53±1.0141	79.94±0.6741	80.01±0.82 ^{c1}	80.37±1.1441			
	1 Week	81.50±5.69 ¹¹	77.25±4.13 ⁽¹⁾	75.76±2.34 ⁽¹⁾	75.59±3.70 ¹¹	73.69±2.15 ⁽²⁾	77.39±4.25 ⁴¹	3.198	0.017	
	1 Month	72.21±1.31*.3	64.46±2.48*4	64.35±1.83**	63.13±2.99×4	62.66±3.76×4	66.14±3.26*4	11.505	<.001	
Filtek Supreme	Baseline	73.53±1.83 ⁽¹	73.59±1.76 ⁽¹⁾	73.40±0.94 ¹¹	73.74±2.24 ¹³	73.77±1.33 ^{ti}	73.63±1.80 ⁽¹			
	1 Week	74.15±5.111	70.34±2.92±12	70.17±2.79 ¹²	69.92±3.52 ¹²	68.07±1.88¤2	69.80±2.80 ¹¹²	2.553	0.045	
	1 Month	65.47±2.99±3	59,37±1,40%	59.04±2.21×4	57.08±1.74 ²⁴⁵	55.03±0.74%	58.04±1.86 ²⁴⁵	23.598	<,001	
Premise	Baseline	61.09±0.87 ¹¹	61.11±1.44	61.07±1.33 ⁺	61.06±1.36 ³	61.03±1.14 ¹	61.09±1.33 ¹			
	1 Week	62.75±3.35 ¹¹	58.96±1.82°	57.27±1.09 ²	57.92±1.62 ⁰	55.81±2.81 ⁰	58.83±1.94 ⁰	7.659	<,001	
	1 Month	53.90±2.13 ^{k3}	47.61±1.23 ^{.45}	48.74±1.76 ^{.45}	46.45±1.40 ⁺⁵	47.07±2.04**3	49.74±1.10 ^{1,4}	18.613	<.001	

Within each material group for each solution the same smallletters in the same column represent statistical insignificance, whereas different small letters represents statistical significance for the Bonferroni test at p <0.05. Within each material group for each test period the same numbers in the same now represent statistical insignificance, whereas different numbers represents statistical significance for the Bonferroni test at p <0.05.

Table 1- Mean ± standard deviation surface hardness values of tested restorative materials before and after immersion in solutions

	Effect	F-value	Significance
Compoglass	Immersion time	495.287	<0.001
	Solution	22.331	<0.001
	Immersion time * solution	5.439	0.002
Filtek Z250	Immersion time	73.243	<0.001
	Solution	2.761	0.033
	Immersion time * solution	0.708	0.714
Filtek Supreme	Immersion time	244.099	<0.001
	Solution	5.814	<0.001
	Immersion time * solution	2.184	0.085
Premise	Immersion time	270.701	<0.001
	Solution	5.983	<0.001
	Immersion time * solution	2.675	0.005

Table 2- Multivariate ANOVA results of the effects of interest (immersion time and solution) and interactions between these effects (immersion time * solution)

In particular, the Compoglass F (compomer) demonstrated a significant surface hardness reduction after immersion in sports and energy drinks compared to the other resin-based composite materials after the 1-month evaluation period (p<0.05). However, surface hardness reductions among the tested resin-based composites was insignificant (p>0.05; Table 2) for the same evaluation period.

For the Compoglass, there were significant differences between the effect of distilled water and the effect of other solutions on the surface hardness (p<0.001), whereas no significant differences in surface hardness were observed among the sports and energy drink immersed samples (p>0.05).

For Filtek Z250, there were no significant differences between the effects of distilled water and sports and energy drinks on the surface hardness (p>0.05) except for Burn(p=0.05)whereas no significant differences in surface hardness were detected among the sports and energy drink immersed samples (p>0.05).

For Filtek Supreme, no significant differences in surface hardness were observed among the sports and energy drink samples (p>0.05), whereas significant differences in surface hardness were observed among the samples stored in distilled water and immersed in other solutions (p<0.05) except for Powerade(p=0.063).

For Premise, no significant differences in surface hardness among the sports and energy drink immersed samples (p>0.05); however, significant differences in surface hardness were observed between the samples stored in distilled water and other solutions (p<0.05) except for Red Bull (p=0.1140.

DISCUSSION

The present study was aimed to determine the surface microhardness of different restorative materials after exposure to different solutions.

During consumption, food or drink contacts teeth or restoration surfaces for only a short time before it is washed away by saliva. However, in previous studies, substrates usually had contact with acidic food or drink for a prolonged period of time and the situation did not account for the role of saliva^{2,33}. Therefore, in the present study, due to the acidity and erosive potential of sports and energy drinks, the restorative materials were immersed in these drinks for 2 min a day and then stored in distilled water for the rest of the day to simulate the washing effect of saliva and represent a lower frequency of intake for a short-term period (1-month). Distilled water was selected instead of artificial saliva to simulate the washing effect of saliva because the artificial saliva storage medium is not considered to be a more clinically relevant environment¹⁸. In addition, Turssi, et al.²⁹ (2002) evaluated the influence of storage media upon the micromorphology of resin-based materials and achieved similar results for distilled water and artificial saliva.

Due to the increased consumption of sports and energy drinks among the general population in recent years for the purpose of enhancing performance and endurance^{8,14,25}, the authors of this study wanted to observe the effects of the five most commonly consumed sports and energy drinks on the surface hardness of restorative materials, which are commonly used for restoring teeth that have erosive conditions.

The present results reveal that all four restorative materials showed a significant surface hardness reduction after the 1-month storage and immersion period, irrespective of the solution used. However, specimens that were immersed in sports and energy drinks demonstrated greater surface hardness reductions when compared to the specimens stored in distilled water after a 1-month evaluation period. Therefore, the first null hypothesis, which stated that " there would be no significant effect of the type of solutions on the surface hardness of the restorative materials", was rejected.

The surface hardness values of all restorative materials after 1-week of storage in distilled water are higher than the baseline surface hardness values. This could possibly be explained by the increased monomer conversion and/or additional post-curing cross-linking reactions in the resin phase over the course of time 16,18,33. However, when comparing baseline surface hardness values and after 1-week of sports and energy drink immersion, all of the restorative materials showed lower surface hardness values. This could be due to the fact that all of the restorative materials displayed a tendency to erode under acidic conditions and the acids in these drinks promoted the release of unreacted monomers by penetrating into the resin matrix, thereby resulting in lower surface hardness values^{21,37}. Previous studies have revealed that compomers release higher amounts of fluoride into acidic buffers than into neutral buffers, indicating that the structure of compomers could have been solved at a low pH^{4,11}. The compomer tested in this study showed the greatest surface hardness reduction of all the restorative materials when immersed in the acidic sports and energy drinks. In accordance with our findings, Abu-Bakr, et al.¹ (2000) evaluated the effect of low-pH drinks on the surface hardness and surface texture of tooth-colored restorative materials for 7 days, and reported that a compomer immersed in a low-pH soft drink exhibited significantly lower hardness values than a compomer immersed in deionized water for the same period.

The hardness results obtained in the present study indicate that immersion time in the solutions had a critical impact on the restorative materials. Therefore, the second null hypothesis, which stated that "two minutes daily exposure time in the tested solutions for a 1-month evaluation period would have no significant effect on the surface hardness of resinbased restorative materials", was also rejected. In general, regardless of the solutions used, all restorative materials demonstrated significantly lower values after a 1-month evaluation period than after 1-week. This is because the materials deteriorate by way of liquid absorption. Storage in distilled water decreased the surface hardness of the restorative materials studied. This can be explained by the fact that "water serves as a plasticizing molecule within the composite matrix¹⁸, causing a softening of the polymer resin component by swelling the network and reducing the frictional forces between polymeric chains^{7,21}".

The sports and energy drinks tested in the present study are low-pH beverages with a pH ranging from 2.67 to 3.79, and this could explain the lower surface hardness values of the restorative materials that were obtained in the study for the entire experimental period. It has been previously reported that resin materials immersed in "low-pH drinks have a high solubility, and this solubility causes surface erosion and dissolution, negatively affecting wear, hardness, and surface integrity by softening the matrix and causing a loss of structural ions^{26,31}". In addition, increased interaction between the solutions and resins, as well as the

water uptake and greater erosive effect of acidic conditions on restorative materials, resulted in the decreased surface hardness values observed in the present study. However, the effect of the pH of the tested solutions on the surface hardness of the restorative materials was not evaluated in the study. Further research is necessary to evaluate this effect.

A resin-based restorative material may include different types of filler particles. It has been noted that composite materials containing "zinc, barium glass, and zirconia/silica fillers were shown to be more susceptible to aqueous attack than those containing quartz fillers³⁵⁻³⁷". The resin-based composite materials used in the present study composed of high volume fraction of filler particles with a narrow particle size distribution, the average particle size being below 1 μ m. Also, the chemical composition of resin based composites may have an effect on their susceptibility to softening and degradation^{9,17,36}.

However, all three resin-based composite materials contain Bis-EMA and a reduced amount of TEGDMA, characteristics which promote better resistance to the action of chemical substances¹⁹. Therefore, differences in chemical composition among the compomer and resin-based composites might have contributed to the differences in surface hardness among these materials.

The present study evaluated the *in vitro* effects of different solutions on the surface hardness of restorative materials for 1-month; further long- term clinical and *in vitro* studies are needed to investigate and elucidate the effects of these solutions on the surface hardness of the restorative materials. Although this study could not completely replicate the complex oral cavity, it confirms the deleterious effects of some commercially available sports and energy drinks on restorative materials, effects which patients should be aware of.

CONCLUSIONS

Under the conditions of this 1-month in vitro study, the following conclusions were drawn:

Surface hardness values of the composite resin materials were significantly decreased, either immersed in distilled water or immersed in sports and energy drinks after the 1-month evaluation period.

Distilled water exhibited less reduction than the sports and energy drinks on surface hardness values of the composite resin specimens over time.

The compomer was the most affected material than the other restorative materials in terms of surface hardness change.

Duration of exposure time and the composition of the composite resin material have a significant effect on the surface hardness change of a restorative material.

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