Surface hardness of different restorative materials after long-term immersion in sports and energy drinks

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The purpose of this study was to evaluate the effect of sports and energy drinks on the surface hardness of different restorative materials over a 6-month period. Forty-two disk-shaped specimens were prepared for each of the four restorative materials tested: Compoglass F, Filtek Z250, Filtek Supreme, and Premise. Specimens were immersed for 2 min daily, up to 6 months, in six storage solutions ($n=7$ per material for each solution): distilled water, Powerade, Gatorade, X-IR, Burn, and Red Bull. Surface hardness was measured at baseline, after 1 week, 1 month, and 6 months. Data were analyzed statistically using repeated measures ANOVA followed by the Bonferroni test for multiple comparisons ($\alpha=0.05$). Surface hardness of the restorative materials was significantly affected by both immersion solution and immersion period ($p<0.001$). All tested solutions induced significant reduction in surface hardness of the restorative materials over a 6-month immersion period.

Keywords: Surface hardness, Resin-based composite, Energy drinks, Sports drinks

INTRODUCTION

Sports and energy drinks “contain caffeine, taurine, vitamins, herbal supplements, and sugar or sweeteners and are marketed to improve energy, weight loss, stamina, athletic performance, and concentration”¹. Although many brands and products are available in the market, they generally have similar compositions². Consumption of sports and energy drinks has garnered a large following among adolescents and young adults in many countries. In the US, they constituted the fastest growing segment of the beverage industry in 2011^{1,2}. Alarming, these popular, widely consumed beverages reportedly have the potential to cause dental erosion^{2,3}.

In recent years, composite resin materials have also garnered a large following among patients with increased aesthetic expectations and demands. Improved formulations and simplified bonding procedures have endeared these materials to both patients and dentists alike^{4–6}. Improvements in their mechanical and physical properties stem from reinforcing dental composites with filler particles, of which the filler concentration and particle size wield a significant influence⁷. Recent researches on dental composites, in which reinforcing nanoparticles are embedded in a polymeric matrix, have led to the development of a new generation of dental composites with reduced particle size and increased filler loading, hence resulting in excellent aesthetics, improved wear resistance, higher level of fracture toughness, and lower brittleness^{8,9}.

The mechanical properties of composite resins are influenced not only by their chemical compositions, but also by the environment to which they are exposed^{10,11}.

The low pH of acidic foods and drinks reportedly induced pronounced erosive wear in these materials¹². To be clinically successful, dental composites must be durable and have a high degree of wear resistance in the oral cavity^{13,14}. They are exposed either intermittently or continuously to chemical agents found in saliva, food, and beverages^{15–17} — and such exposure can soften the resin matrix of the composite resins and cause filler constituents to leach out¹⁸.

Surface hardness is an important indicator of a restorative material’s mechanical strength and resistance against intra-oral softening^{4,19}. Protracted storage in and contact time with acidic media such as coffee, tea, and mouthwashes have been shown to reduce the surface hardness of resin-based composite materials^{14,20–22}. Therefore, the low pH of sports and energy drinks might pose an erosive threat not only on dental hard tissues but also on the clinical longevity of restorative materials. However, there is little documented evidence of the effect of sports and energy drinks on the surface hardness of restorative composite resins. This is a notable omission, given the prevalence of these drinks among young adults and the increasing use of resin-based composite materials for patients of all ages.

The goal of the present study was to evaluate the effect of long-term (up to 6 months) immersion in sports and energy drinks on the surface hardness of a polyacid-modified composite resin (compomer), a microhybrid composite, and two nanofilled composites. The null hypotheses to be tested were: (1) immersion solution would have no effect on the surface hardness of composite resin restorative materials; and (2) immersion period would have a significant effect on the surface hardness of composite resin restorative materials.

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MATERIALS AND METHODS

Materials

Four types of commercial restorative materials of A2 shade were used in this study. They were: a polyacid-modified composite resin compomer (Compoglass F, Ivoclar Vivadent, Schaan, Liechtenstein), a microhybrid composite resin (Filtek Z250, 3M ESPE, St. Paul, MN, USA), and two nanofilled resin composites (Filtek Supreme, 3M ESPE, St. Paul, MN, USA; Premise, Kerr Hawe, Orange, CA, USA). Their chemical compositions are given in Table 1.

Specimen preparation

For each restorative material, 42 disk-shaped specimens (8 mm diameter, 2 mm thickness) were prepared using a customized cylindrical metal mold, rendering a total of 168 specimens for the four restorative materials. To ensure that specimens would have flat polymerized surfaces with no bubble formation after curing, the top and bottom surfaces were each covered with a polyester matrix strip (Mylar Strip, SS White Co., Philadelphia, PA, USA) and a thin, rigid, glass microscope slide (1 mm thickness). Finger pressure was applied on the glass

slide, and excess material was removed.

Composite specimens were polymerized using a quartz-tungsten-halogen (QTH) light curing unit (LCU) (VIP, Bisco Inc., Schaumburg, IL, USA) through the glass slide and polyester matrix strip for 20 s. According to manufacturer's recommendations, QTH LCU was used in standard mode, emitting at least 600 mW/cm² irradiance as measured using a built-in radiometer. Light probe tip was placed perpendicular to and in contact with the glass slide, such that the distance between the light source and specimen was standardized at 1 mm according to glass slide thickness. To ensure complete polymerization, all light-cured specimens were stored in distilled water in a lightproof container for 24 h at 37°C.

Before subjecting to baseline microhardness evaluation, the top surfaces of all specimens were sequentially polished with medium, fine, and super-fine aluminum oxide-impregnated disks (Sof-Lex, 3M ESPE, St. Paul, MN, USA) using a slow-speed handpiece under dry conditions for 30 s. Between each polishing step, the specimens were thoroughly rinsed with water for 10 s to remove debris and allowed to air-dry for 5 s.

Table 1 Compositions of the restorative materials used in this study

Resin Composite	Classification	Filler Weight (%)	Filler Volume (%)	Filler Type	Filler Size	Monomer Composition	Shade
Compoglass F (Ivoclar Vivadent, Schaan, Liechtenstein) Batch # K36230	Polyacid- modified resin composite (Compomer)	77	55	SiO ₂ , YbF ₃ , (Ba)F ₂ Si	1 µm	Bis-GMA, UDMA, TEGDMA, CDCDMA	A2
Filtek Z250 (3M ESPE, St. Paul, MN, USA) Batch # 6CC	Minifilled hybrid	82	60	Zirconia/silica	0.6 µm	Bis-EMA, UDMA, Bis-GMA	A2
Filtek Supreme (3M ESPE, St. Paul, MN, USA) Batch # 7CB	Nanofilled	78.5	59.5	ZrO ₂ /SiO ₂ nanocluster, SiO ₂ nanofiller	5–20 nm with 20 nm silica filler	Bis-GMA, Bis-EMA, UDMA, TEGDMA	A2B
Premise (Kerr Hawe, Orange, CA, USA) Batch # 3249984	Nanohybrid	84	69	Barium alumino borosilicate glass, silica nanofiller, PPF [†] , barium glass, discrete nanofiller	Glass: 0.4 µm, silica: 0.02 µm	Bis-GMA, Bis-EMA, TEGDMA	A2

Bis-GMA: bisphenol A glycol dimethacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; CDCDMA: cycloaliphatic dicarbonic acid dimethacrylate; Bis-EMA: ethoxylated bisphenol A glycol dimethacrylate; PPF: pre-polymerized filler particles.

Microhardness test

With each restorative material, the 42 specimens were randomly divided into six groups ($n=7$ per group), according to the six immersion solutions to be investigated in this study. Microhardness testing was carried out using a microhardness tester (Micromet 5114, Buehler Ltd., Lake Bluff, IL, USA) with a pyramid-shaped diamond indenter.

Each specimen was placed on the platform with the test surface facing the indenter. The indenter made three indentations at different areas on the specimen using a 200-g load and with 15-s dwell time. Each measurement was taken automatically at a distance of 1 mm from each other. The average value of the three measurements was converted into a Vickers hardness number (VHN) expressed in kg/mm^2 .

Immersion in storage solutions

The six immersions solutions used were: distilled water (control solution), 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).

Following baseline microhardness evaluation, seven specimens of each restorative material were immersed in individual vials containing 5 mL of an immersion solution for 2 min at room temperature ($23\pm1^\circ\text{C}$). After rinsing with distilled water, the specimens were immersed in fresh vials of distilled water at 37°C for

24 h. This cycle (2 min in immersion solution, distilled water rinse, 24 h in distilled water) was repeated daily for three immersion periods: 1 week, 1 month, and 6 months. Control specimens were immersed in individual vials containing 5 mL of distilled water (pH 6.58) at 37°C , and the water was changed daily.

All vials were sealed to prevent immersion solutions from evaporating. The solutions were changed daily, and their pH levels were measured with a pH meter (HI 221, Hanna Instruments Inc., Woonsocket, RI, USA) prior to immersing the specimens. After 1 week, 1 month, and 6 months of immersion, specimens were removed for the microhardness test.

Statistical analysis

Normality of data distribution was first determined using the Kolmogorov-Smirnov and Shapiro-Wilk tests. As data in some groups were not normally distributed, repeated measures ANOVA was applied for parametric data and the Friedman test for nonparametric data. This was followed by *post hoc* Bonferroni test for pairwise multiple comparisons. Values were expressed as mean $\pm$ SD. Significance was designated as $p<0.05$. Statistical analysis was performed using the Statistical Package for Social Sciences (SPSS) software, version 17.0 (SPSS Inc., Chicago, IL, USA).

RESULTS

Figures 1–6 show the mean surface hardness values, standard deviations, and significant differences of each restorative material before and after the 1-week, 1-month,

Fig. 1 Mean Vickers hardness values and standard deviations of restorative materials before and after immersion in distilled water.

Within each material group, the same small letters indicate that data are not statistically significant, whereas different letters indicate statistical significance according to the Bonferroni test at $p<0.05$.

Fig. 2 Mean Vickers hardness values and standard deviations of restorative materials before and after immersion in Powerade.

Within each material group, the same small letters indicate that data are not statistically significant, whereas different letters indicate statistical significance according to the Bonferroni test at $p<0.05$.

Fig. 3 Mean Vickers hardness values and standard deviations of restorative materials before and after immersion in Gatorade.

Within each material group, the same small letters indicate that data are not statistically significant, whereas different letters indicate statistical significance according to the Bonferroni test at $p < 0.05$.

Fig. 5 Mean Vickers hardness values and standard deviations of restorative materials before and after immersion in Burn.

Within each material group, the same small letters indicate that data are not statistically significant, whereas different letters indicate statistical significance according to the Bonferroni test at $p < 0.05$.

Fig. 4 Mean Vickers hardness values and standard deviations of restorative materials before and after immersion in X-IR.

Within each material group, the same small letters indicate that data are not statistically significant, whereas different letters indicate statistical significance according to the Bonferroni test at $p < 0.05$.

Fig. 6 Mean Vickers hardness values and standard deviations of restorative materials before and after immersion in Red Bull.

Within each material group, the same small letters indicate that data are not statistically significant, whereas different letters indicate statistical significance according to the Bonferroni test at $p < 0.05$.

and 6-month immersion periods. Repeated measures ANOVA revealed that the surface hardness values of the four restorative materials were significantly affected by both the immersion solution ($p < 0.001$) and immersion period ($p < 0.001$). Interaction of the immersion solution and immersion period factors was significantly different for all restorative materials ($p < 0.001$), except Filtek Z250 ($p = 0.117$).

Surface hardness changes of the specimens in each immersion solution for each immersion period were

determined by percentage difference (%). The softening characteristics of the restorative materials differed at each immersion period according to the type of immersion solution. 1-week immersion in distilled water increased the mean surface hardness values of all the four restorative materials, whereas 1-week immersion in any of the sports and energy drinks decreased their mean surface hardness when compared to their baseline measurements. Significant differences were detected for all restorative materials ($p < 0.001$) when comparing the

1-week and 1-month immersion periods. After 6 months' immersion, all restorative materials had significantly lower surface hardness than after 1 week or 1 month ($p < 0.001$).

Gradual decreases in mean surface hardness were observed for all restorative materials at each immersion period for all sports and energy drinks. Significant differences in hardness change were observed between Compoglass F and other restorative materials regardless of immersion solution ($p < 0.01$). After 6 months, Compoglass F demonstrated significantly decreased surface hardness when compared to Filtek Z250 and Filtek Supreme ($p < 0.05$). On the other hand, no significant differences in hardness change were observed among the other three restorative materials at each immersion period for all sports and energy drinks ($p > 0.05$). At 6 months, no significant differences ($p > 0.05$) were observed between Compoglass F and Premise, Filtek Supreme and Premise, and Filtek Z250 and Filtek Supreme. Regardless of immersion period, the mean surface hardness values of all restorative materials immersed in distilled water were significantly different from those immersed in sports and energy drinks ($p < 0.001$).

For Compoglass, there were no significant differences in surface hardness at each immersion period for immersion in Powerade, Gatorade, X-IR, and Red Bull solutions ($p > 0.05$). However, significant differences were observed between Powerade and Burn ($p < 0.01$) and between Burn and Red Bull ($p < 0.05$). For Filtek Z250, there were no significant differences in surface hardness among any of the sports and energy drinks ($p > 0.05$). For Filtek Supreme, there were no significant differences among Powerade, Gatorade, X-IR, and Red Bull solutions ($p > 0.05$). However, significant differences were observed between Powerade and Burn ($p < 0.01$), Gatorade and Burn, and Burn and Red Bull ($p < 0.05$). For Premise, there were no significant differences in surface hardness among the sports and energy drinks, except between Burn and Red Bull ($p < 0.05$).

For all the immersion solutions used in this study, the surface hardness of all restorative materials decreased significantly after 1 month and 6 months of immersion ($p < 0.001$). According to Bonferroni test for pairwise multiple comparisons, surface hardness was least reduced by immersion in distilled water ($p < 0.001$); on the contrary, Burn energy drink caused the greatest reduction in surface hardness ($p < 0.05$) after 6 months of immersion.

DISCUSSION

In the oral cavity, restorative materials are exposed to changes in temperature and acid-base balance due to food and drink consumption²³. To ensure clinical longevity, dental restorative materials should be adequately resistant to such fluctuating conditions or be only minimally affected by them²³. However, food and drinks are generally in contact with the tooth or restoration surfaces for a short duration only before

being washed away by saliva. In previous studies on dental erosion, experimental substrates were immersed in acidic solutions for prolonged periods with no room for the cleansing role of saliva^{21,22,24}.

In the present study, a different strategy was used to test the erosive potential of sports and energy drinks. Restorative materials were immersed in these beverages for only 2 min a day, then rinsed and immersed in distilled water for the rest of the day. The transient exposure to acidic solutions followed by the washing effect of saliva were thus simulated and repeated over a longer period of up to 6 months.

In a study by Turssi *et al.*²⁵ to evaluate the influence of storage media on the surface morphology of resin-based materials, they found that distilled water and artificial saliva yielded similar effects. Similarly, Yap *et al.*¹⁵ reported equivalent degradation of composite materials after exposure to water or artificial saliva. Therefore, distilled water instead of artificial saliva was used in the present study to simulate the washing effect of saliva.

In recent years, consumption of sports and energy drinks has increased among young adults for the purpose of boosting performance and endurance^{1,2}. Thus, the five most commonly consumed sports and energy drinks were investigated for their effects on the surface hardness of four composite resins materials routinely used for restoring teeth. Results showed that all the sports and energy drinks pronouncedly reduced the surface hardness of all the restorative materials tested. The compomer, Compoglass F, was most adversely affected by the acidic beverages. Therefore, the first null hypothesis—which stated that the immersion solution would have no effect on the surface hardness of composite resin restorative materials—was rejected.

Interestingly, the surface hardness of all restorative materials increased after 1 week of immersion in distilled water. This phenomenon could be attributed to an effect of the chemical postcuring process, whereby monomer conversion increased and chemical bonds continued to be made^{21,26}. The sports and energy drinks tested in this study had pH levels ranging between 2.67 and 3.79. Immersion therein for 1 week resulted in lower surface hardness of all the restorative materials than at baseline, suggesting a susceptibility of the composite resins to erode under acidic conditions^{27,28}. In particular, Compoglass F showed significantly lower surface hardness than the other restorative materials after 1 week of immersion, and this was true for all sports and energy drinks. The structures of compomers disintegrate at low pH²⁹. Therefore, acidic conditions posed by the sports and energy drinks caused Compoglass F to release more fluoride than did the neutral-pH distilled water, leading to its decreased surface hardness. In a study by Abu-Bakr *et al.*³⁰ which evaluated the effect of low-pH soft drinks on surface hardness, solubility, and surface texture of tooth-colored restorative materials, it was also found that a compomer immersed in low-pH drinks for 7 days exhibited significantly lower surface hardness than when immersed in deionized water.

The surface hardness of all restorative materials decreased between 1 week and 1 month of immersion in all the tested solutions. Deterioration of resin materials probably occurred because of water absorption, such that the presence of water softened the resin by swelling the polymer network and reducing the frictional forces between the polymer chains^{31,32}. Moreover, composite resins have high solubility in low-pH solutions, leading to surface erosion and dissolution, matrix softening, and loss of structural ions^{27,28,33,34}.

After 6 months of immersion, further softening of all the restorative materials was observed. This was probably a result of increased interaction —by virtue of increased contact time— between the immersion solutions and resin materials, leading to greater water uptake and higher erosive impact of the acidic conditions on the materials. As reported by Moraes *et al.*³⁵, storage for extended time periods in aqueous media are detrimental to the composite surface. Therefore, progressive degradation over a 6-month storage period altered the microstructures of the restorative materials, such that the inorganic particles were no longer provided with a stable structure, predisposing them to filler dislodgment and elution³⁵. The tested restorative materials contained barium glass, silica, and zirconia/silica fillers. It has been shown that restorative materials containing inorganic fillers such as zinc, barium glass, and zirconia/silica were more susceptible to aqueous attack than those containing quartz fillers^{15,16,36}. Therefore, the dislodgement and elution of these inorganic filler particles might have contributed to the reduction in surface hardness of the tested restorative materials over a longer immersion time.

Our second null hypothesis was that the immersion period would have a significant effect on the surface hardness of resin composite materials. Our results indicated that this was true, and thus the second hypothesis was accepted. All the restorative materials showed significantly reduced surface hardness after 6 months of immersion in sports and energy drinks, but Compoglass F exhibited the largest reduction at each immersion period for all the tested beverages. Yap *et al.*³⁶ showed that food-simulating liquids affected the surface roughness and hardness of compomers and resin composites in different degrees, which agreed with the results of this study. Differences in their susceptibility to softening and degradation could be attributed to the interaction between the acidic beverages and the chemical components of resin composites^{15,36,37}. Compoglass F contained Bis-GMA and UDMA, and Bis-GMA- and UDMA-based polymers are more susceptible to softening after exposure to chemical agents^{15,36}. The other three restorative materials contained Bis-EMA and TEGDMA, which provided better resistance against chemical attack³⁸. Another contributing factor was filler type and content. Compared to Compoglass F, the other three restorative materials had a higher content of filler particles with a narrow particle size distribution, hence upping their resistance against chemical degradation.

The nature of acid present in test solutions also

influenced the degree of surface degradation of resin composites. The sports and energy drinks used in this study contained citric acid. Citric acid has been shown to be the most aggressive storage medium for dental hard tissues and resin-based restorative materials^{28,30,39}. Compared to compomers, pure composite resins were found to be less affected by low-pH beverages¹⁷ or acidic solutions²⁸. Therefore, Filtek Z250, Filtek Supreme, and Premise exhibited less surface hardness change with no significant differences among them than did Compoglass F at each immersion period. This was because the citric acid found in the sports and energy drinks affected the solubility of compomer Compoglass F more than the other resin composites^{17,30}.

Among the examined sports and energy drinks, Burn —with the lowest pH at 2.67— had the greatest surface hardness reduction effect on the restorative materials tested. Its high acidity might have a greater softening effect on the resin matrix, thus promoting the dislodgement and leaching out of filler particles^{17,18} and reducing the load resistance of the restorative materials. In a recent study¹⁷ which investigated the effects of various beverages on microhardness, surface roughness, and solubility of restorative materials, it was found that low-pH beverages increased the solubility and surface roughness and decreased the surface hardness of the restorative materials tested. However, the effect of pH value of acidic beverages on the surface hardness of restorative materials was not examined in this study. Further research is needed to evaluate the impact of pH levels.

Temperature could be an important factor in the erosive effect of sports and energy drinks on the surface hardness of restorative materials. These drinks are usually consumed at a low temperature (*e.g.*, 4°C). In the present study, however, the 2-min immersion was performed at room temperature (23±1°C). West *et al.*⁴⁰ reported that acidic drinks were less erosive on enamel when consumed at low rather than high temperatures. Therefore, it could be safely assumed that the detrimental changes observed in this study were due to the acidic nature of the solutions used, since solution temperature was maintained at room temperature.

The complex environment of the oral cavity could not be exactly and entirely replicated by *in vitro* experimental conditions. Although the present study confirmed that acidic sports and energy drinks had a detrimental effect on the surface hardness —and hence lifespan— of composite resin restorative materials, it remained to be known if the durability of restorative materials would be thus adversely affected in the oral environment³⁵. Saliva is known to have a protective effect in moderating the extents of wear, abrasion, and fatigue of restorative materials by interfering with the hydrolytic degradation process. Therefore, the long-term clinical and *in vitro* performance of restorative materials needs further evaluation.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions were drawn:

1. All the solutions used in this study significantly reduced the surface hardness of the restorative materials, particularly after a 6-month immersion period.
2. Polyacid-modified resin composite (compomer) showed significantly greater reduction in surface hardness than did the other pure resin composites tested.
3. Immersion in distilled water caused less surface hardness reduction over time than did the low-pH sports and energy drinks.
4. Effect of sports and energy drinks on the surface hardness of a restorative material also depended on exposure/contact time and chemical composition of the restorative material.
5. The benefits of sports and energy drinks for most exercising individuals are negligible when compared to water. However, these drinks wield definite detrimental effects on the surface integrity of dental restorations and tooth structure.

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