# Effect of Energy Drinks on Discoloration of Silorane- and Dimethacrylate-Based Composite Resins

Ghazaleh Ahmadizenouz<sup>1</sup>, Behnaz Esmaeili<sup>1</sup>, Zohreh Ahangari<sup>2</sup>, Soraya Khafri<sup>3</sup>, Aghil Rahmani<sup>2</sup>

<sup>1</sup>Assistant Professor, Dental Materials Research Center, Department of Restorative and Esthetic Dentistry, Faculty of Dentistry, Babol University of Medical Sciences, Babol, Iran

<sup>2</sup> Dental Student, Student Research Committee, Babol University of Medical Sciences, Babol, Iran

<sup>3</sup>Assistant Professor, Department of Social Medicine and Health, Faculty of Medicine, Babol University of Medical Sciences, Babol, Iran

#### Abstract

both time points.

Spectrophotometry

**Objectives:** This study aimed to assess the effects of two energy drinks on color change  $(\Delta E)$  of two methacrylate-based and a silorane-based composite resin after one week and one month. Materials and Methods: Thirty cubic samples were fabricated from Filtek P90, Filtek Z250 and Filtek Z350XT composite resins. All the specimens were stored in distilled water at 37°C for 24 hours. Baseline color values (L\*a\*b\*) of each specimen were measured using a spectrophotometer according to the CIEL\*a\*b\* color system. Ten randomly selected specimens from each composite were then immersed in the two energy drinks (Hype, Red Bull) and artificial saliva (control) for one week and one month. Color was re-assessed after each storage period and  $\Delta E$  values were calculated. The data were analyzed using the Kruskal Wallis and Mann-Whitney U tests. **Results:** Filtek Z250 composite showed the highest  $\Delta E$  irrespective of the solutions at both time points. After seven days and one month, the lowest  $\Delta E$  values were observed in Filtek Z350XT and Filtek P90 composites immersed in artificial saliva, respectively. The  $\Delta E$ values of Filtek Z250 and Z350XT composites induced by Red Bull and Hype energy drinks were not significantly different. Discoloration of Filtek P90 was higher in Red Bull energy drink at both time points. **Conclusions:** Prolonged immersion time in all three solutions increased  $\Delta E$  values of all

composites. However, the  $\Delta E$  values were within the clinically acceptable range (<3.3) at

Keywords: Color; Composite Resins; Energy Drinks; Silorane Composite Resin;

Journal of Dentistry, Tehran University of Medical Sciences, Tehran, Iran (2016; Vol. 13, No. 4)

<sup>ed</sup>Corresponding author: Z. Ahangari, Dental Student, Student Research Committee, Babol University of Medical Sciences, Babol, Iran

s.ahangari69@gmail.com

Received: 24 January 2016 Accepted: 7 June 2016

# **INTRODUCTION**

Direct restorations are widely used in restorative dentistry. Composite resins are the material of choice for many of these restorations [1]. The popularity of composite resins is mainly because of their esthetic properties, ability to bond to tooth structure and lower cost compared to indirect restorations [2]. A successful composite resin restoration should mimic the tooth color and retain its color over a long period of time in the oral cavity [3]. Color change is one drawback of composites, which necessitates their replacement over time [4].

The color change of composites can be caused by intrinsic or extrinsic factors. Intrinsic discoloration is related to changes in resin material itself [5], which is associated with changes in resin matrix and fillers, size and distribution of fillers, type of photoinitiator (especially camphorquinone) and incomplete polymerization [2,6,7]. Extrinsic discoloration can be caused by adsorption or absorption of stains with exogenous origin i.e. diet, poor oral hygiene and habits such as smoking [7,8]. The degree of discoloration depends on factors such as type of dye, surface roughness, type of

Product	Manufacturer	Туре	Content					T -4
(code)			Organic matrix	Fillers	Particle size	Filler weight	Filler volume	Lot number
Filtek (P90)	3M ESPE, St. Paul, MN, USA	Silorane	Silorane	Quartz, yttrium fluoride	0.47 μm	76%	55%	N468933
Filtek (Z250)	3M ESPE, St. Paul, MN, USA	Microhybrid	Bis-GMA, UDMA, Bis- EMA	Zirconia/silica	0.01-3.5 μm	84.5%	60%	N528844
Filtek (Z350)	3M ESPE, St. Paul, MN, USA	Nanofilled	Bis-GMA, UDMA, TEGDMA, Bis- EMA	Zirconia/nano- silica	5-20 nm	82%	59.5%	N495372

Table 1: Composition of restorative materials (shade: A3) used in this study

Bis-GMA: Bisphenol A glycol dimethacrylate; UDMA: Urethane dimethacrylate; Bis-EMA: Ethoxylated bisphenol A glycol dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; Bis-EMA: Ethoxylated bisphenol A glycidyl methacrylate

composite material, temperature, UV radiation and duration of exposure to coloring agents [4,9,10]. Extrinsic discoloration may be fixed by scaling and polishing, but if deeper layers are involved, color change is usually irreversible [9, 10]. Previous studies have reported composite discoloration due to consumption of beverages such as tea, coffee, cola and red wine. The color change potential is different depending on the composition and properties of materials [4].

In the recent years, consumption of energy drinks has become very popular especially among adults (aged 18 to 35 years) and people interested in sport activities [11,12]. Energy drinks are essentially non-alcoholic drinks, which contain taurine, caffeine, glucuronolactone, inositol and vitamin B complex. Some of them contain minerals and carnitine, others contain sugar at a concentration of 12-14% and the rest are formulated without sugar and have been marketed to enhance physical resistance, increase focus and stimulate metabolism [11]. These drinks have high erosion potential due to acidic pH and high sugar content [13]. Few studies have been carried out on the effect of energy drinks on composite resin restorations and some of them have indicated the role of these drinks in staining nanofilled, micro-hybrid and methyl methacrylate composite resins [2,14,15]. Resin matrix structure and fillers have direct impacts on staining susceptibility of composite resins [2]. Various composite resin materials have been marketed, with improved physical properties and modified resin matrix, size, type and amount of filler particles [2]. Recently introduced low-shrinkage composite resins contain silorane monomers, with different resin formulation from that of methacrylate-based composite resins [16]. Silorane-based composites show mechanical properties similar to methacrylate-based composite resins, but with lower marginal microleakage, higher flexural strength, less solubility in water and fluids and lower water sorption [9,17].

Silorane contains cationic ring-opening hybrid monomer and is formed by the reaction of siloxane and oxirane molecules [7]. Siloxane forms the hydrophobic backbone and results in long-term sustainability of the material. Oxirane is responsible for cationic polymerization reaction and reduces polymerization shrinkage [16,17]. Although several studies have been conducted on mechanical properties of silorane, the color stability of these composites still requires additional research [5]. The aim of the present study was to assess the color change of micro-hybrid, nanofilled and silorane-based composite resins exposed to two types of energy drinks and artificial saliva for one week and one month.

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Staining solutions	Composition	Manufacturer	
Red Bull	Water, sucrose, glucose, acidity regulators (sodium citrates, magnesium carbonate), carbon dioxide, acidifier citric acid, 0.4% taurine, 0.03% caffeine, inositol, vitamins (niacin, pantothenic acid, B6, B12), flavoring, colors (caramel, riboflavin)	Red Bull GmbH, Am Brunnen, Austria; pH=3.54	
Нуре	Carbonated water, sugar, acidifier citric acid E330, acidity regulator sodium citrate E331, taurine, caramel sugar syrup, 0.032% caffeine, flavoring, 0.024% glucuronolactone, vitamins (niacin, pantothenic acid, B6, B2, B12)	51 857	
Artificial saliva	Sodium chloride 86.550 mg/100mL, 16.625 mg/100mL calcium chloride, 32.600 mg/100mL dipotassium phosphate, 62.450 mg/100mL potassium chloride, 5.875 mg/100mL magnesium chloride, sorbitol, sodium carboxymethyl-cellulose, purified water preservative: methyl parahydroxybenzoate (E218)	Hypozalix, Biocodex, France	

Table 2 : The com	position and r	nanufacturers of	f the staining	solutions
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The null hypotheses of this study were:

1. Type of composite resin would not affect its color stability, 2. Energy drinks would not change the staining susceptibility of composite resins.

# MATERIALS AND METHODS

In this experimental study, A3 shade of three types of composite resins was selected: Filtek P90 silorane-based composite resin, Filtek Z250 microhybrid composite and Filtek Z350 XT nanofilled composite (Table 1).

A total of 30 cubic samples, measuring 5 mm in length, 5 mm in width and 2 mm in thickness, were fabricated of each composite, adding up to a total of 90 samples, using a customized polyvinyl chloride mold. Two composite resin layers, 1 mm thick, were placed in the mold incrementally with a plastic instrument and pressed by a piece of transparent polyester matrix tape (Mylar Strip, SS White Co., Philadelphia, PA, USA) and a glass slide to prevent air retention and create a smooth surface. Each layer was cured for 20 seconds according to the manufacturer's instructions using a LED lightcuring unit (Valo, Ultradent Product Inc. South Jordan, USA) with a light intensity not less than 800 mW/cm2. Emitting light was measured with a radiometer (Demetron LED Radiometer, Kerr,

Orange, CA, USA) before polymerization. In order to standardize the distance between the device and the sample surface, the device head was placed vertically in contact with 1mm thick glass slide. The lower surface of each sample was marked by a scalpel. Afterward, all the specimens were stored in distilled water at 37°C for 24 hours to ensure complete polymerization. In order to simulate the clinical conditions, the top surfaces of all specimens were sequentially polished with 600 to 1200-grit silicone carbide abrasive papers under wet conditions for 30 seconds. After each polishing step, the specimens were thoroughly rinsed with water for 10 seconds to remove debris and air-dried.

Samples from each composite material were then randomly divided into three subgroups of 10, based on the solutions (artificial saliva, Red Bull energy drink and MPF Hype energy drink). Artificial saliva served as a control solution (Table 2). Ten specimens of each composite resin were immersed in 30 mL of MPF Hype energy drink (pH=3.42; Hype Energy, Warsaw, Poland) or Red Bull energy drink (pH=3.54; Red Bull GmbH, Am Brunnen, Austria) for five minutes a day at room temperature in a sealed container to avoid solution evaporation. After daily fiveminute immersion in the energy drinks, the samples were washed with distilled water and

Composite resin	Immersion		P-value		
Composite resin	period	Нуре	Red Bull	Artificial saliva	I-value
	1 week	0.69±0.21	0.99±0.26	0.77±0.20	0.041
Filtek P90	1 month	1.51±0.12	1.84±0.22	1.01±0.12	< 0.001
Filtek Z250	1 week	1.18±0.26	1.07±0.19	0.81±0.11	0.003
Fillek Z250	1 month	2.58±0.33	2.60±0.22	1.51±0.25	< 0.001
E-14-1- 7250X/F	1 week	0.69±0.13	0.69±0.18	0.52±0.09	0.011
Filtek Z350XT	1 month	1.93±0.55	2.00±0.41	1.15±0.16	0.001

**Table 3:** The mean and standard deviation values of color change ( $\Delta E$ ) of composite resins after immersion in different solutions (P-values are for the Kruskal-Wallis test at  $\alpha$ =0.05 level of significance)

then stored in artificial saliva at 37°C for the rest of the day. For the control subgroups, 10 samples from each composite group were immersed in 30 mL of artificial saliva (Hypozalix, Biocodex, France) in sealed containers at 37°C for 24 hours a day. At the end of each storage period, the specimens were rinsed under running tap water and then the excess water on the surfaces was removed by gentle drying with a tissue paper. The solutions were refreshed daily for each subgroup.

# Assessment of color change:

Baseline color measurements of all the specimens were performed according to the CIE  $L^*a^*b^*$  color space before immersion in solutions using a spectrophotometer (Vita Easyshade, Vident, Brea, CA, USA) and against a white background by a single operator.

each measurement. Before the spectrophotometer was calibrated according to the manufacturer's recommendations. The instrument was placed in the calibration block holder so that the probe tip was flush with and perpendicular to the calibration block and depressed the block. The device was then automatically calibrated after hearing three audible beeps. After successful calibration, the measurement menu was displayed and the "Single Tooth" measurement mode was selected. The probe tip was positioned directly on the sample covering its entire surface and the shade was instantly measured. This device automatically calculates 'a', 'L' and ʻb' parameters for each sample based on the CIE Lab system. CIE Lab system is a three-dimensional colorimetric system. 'L' represents lightness, ranging from 0 (for perfect black) to 100 (for perfect white); 'a' and 'b' are chromaticity coordinates in which 'a' indicates red-green axis (red = +a; green = -a) and 'b' indicates bluevellow axis (vellow = +b; blue = -b). The 'a' and 'b' are considered 0 for neutral colors and their values increase for saturated or intense colors. Color measurement was conducted three times for each sample and average values of 'L', 'a' and 'b' were recorded.

In addition to baseline color assessment, measurements were performed after 7 days and 30 days with the same technique. Before color assessment, samples were washed for five seconds using distilled water and dried by absorbent paper towels. The mean values of  $\Delta a$ ,  $\Delta b$  and  $\Delta L$  after three measurements were calculated and recorded where  $\Delta a$ ,  $\Delta b$  and  $\Delta L$ indicated change in L, a, and b parameters, respectively, before and after immersion for each time period (seven days, one month). The color change ( $\Delta E$ ) of the samples for each time period (baseline and seven days, baseline and one month) was calculated and recorded according to

Composite resin	Immersion period —	Staining solutions			
Composite resin	Inimersion period	Нуре	Red Bull	Artificial saliva	
Filtek P90	1 week	0.2	0.2	0.2	
r niek r 90	1 month	0.2	0.2	0.2	
Filtek Z250	1 week	0.2	0.2	0.038	
FILLER Z250	1 month	0.2	0.2	0.2	
F'14 - L 7250X/T	1 week	0.2	0.2	0.2	
Filtek Z350XT	1 month	0.2	0.082	0.2	

Table 4: P-values of one-sample Kolmogorov-Smirnov test

the following formula:

 $\Delta E = \sqrt{(\Delta a)^2 + (\Delta b)^2 + (\Delta l)^2}$ 

#### Statistical analysis:

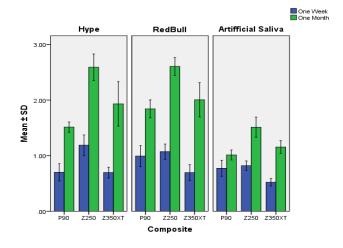
All the statistical analyses were performed with SPSS version 22 for Windows (SPSS Inc., IL, USA). The results were primarily analyzed using Kolmogorov–Smirnov test to evaluate normal distribution of data. When Kolmogorov–Smirnov test results did not show normal distribution of data, non-parametric Kruskal–Wallis test was used. Mann–Whitney U test was used to compare the color change among the composite resin samples in three different solutions, and Wilcoxon signed rank test was used for comparisons between the evaluation periods for each experimental group. Statistical significance level was set at P<0.05.

#### RESULTS

The mean and standard deviation of  $\Delta E$  values for the three composites after immersion in different solutions for seven days and one month are presented in Table 3 and Figure 1.

Color change was considered to be clinically unacceptable when the  $\Delta E$  values were equal or greater than 3.3. Although our results indicated that prolonged immersion time in all three solutions increased  $\Delta E$  values of all composite groups, the color change values were within the clinically acceptable range ( $\Delta E$ <3.3) after one week and one month of immersion.

Considering the small amounts of P-values presented in Table 4 and small sample size of 10 in each group as well as the incompetency of parametric tests for analyzing these data, we analyzed the results by non-parametric tests. Based on the results of the Kruskal–Wallis test, there were significant differences in color change of each composite resin among the solutions at both time points. Regarding stainability of the materials, among all the composite materials tested, the highest  $\Delta E$  values were observed in the Filtek Z250 specimens irrespective of the solutions, at both time points.



**Fig. 1**: The mean and standard deviation of color change of composite resins after immersion in different solutions for one week and one month

Immersion period	Composite resin	Нуре	Red Bull	Artificial saliva
	Filtek P90- Filtek Z250	< 0.001	0.496	0.705
1 week	Filtek P90- Filtek Z350XT	0.880	0.028	0.007
	Filtek Z250- Filtek Z350XT	< 0.001	0.002	< 0.001
	Filtek P90- Filtek Z250	< 0.001	< 0.001	< 0.001
1 month	Filtek P90- Filtek Z350XT	0.034	0.462	0.041
	Filtek Z250- Filtek Z350XT	0.013	0.003	0.004

Table 5: P-values of the Mann Whitney U test for pairwise comparisons of composite resins in terms of  $\Delta E$ 

On the other hand, the lowest  $\Delta E$  values were observed in Filtek Z350XT and Filtek P90 composites after seven days and one month of immersion in artificial saliva, respectively. Tables 5 and 6 list multiple comparisons of  $\Delta E$ values in terms of composite resins and staining solutions. Comparison of  $\Delta E$  between composite groups after seven days of immersion in the solutions revealed that:

-In Hype energy drink, color change of Filtek Z250 composite was significantly higher than that of Filtek P90 and Filtek Z350XT composite resins. There were no significant differences between Filtek P90 and Filtek Z350XT.

- In Red Bull energy drink, the highest color change was observed in Filtek Z250 composite resin, which was not significantly different from that in Filtek P90 composite resin. The color change of Filtek Z350XT composite resin was significantly lower than the other two groups.

- In artificial saliva, the color change of Filtek Z350XT was significantly lower than the other two groups and there were no significant differences between Filtek Z250 and Filtek P90 groups. Comparison of  $\Delta E$  between composite groups after one month in different solutions demonstrated that:

-In Hype energy drink, there were significant differences among the three types of composite resins so that Filtek P90 and Filtek Z250 showed the highest and lowest color change, respectively.

- In Red Bull energy drink, the highest color change was observed in Filtek Z250 composite resin, which was significantly different from that of Filtek P90 and Filtek Z350XT composite resins.

- In artificial saliva, there were significant differences in color change of all three types of composite resins, with Filtek Z250 showing the highest and Filtek P90 the lowest values, respectively.

- In comparison of the two energy drinks, significant differences were observed between the Hype and Red Bull energy drinks only in Filtek P90 specimens in both evaluation periods and chromatic changes induced by the Red Bull drink were more than Hype energy drink. The exact P-values for all the above-mentioned comparisons are presented in Tables 3-6.

# DISCUSSION

In this study, the effects of two energy drinks on color change of three commercially available composite resins from the same manufacturer were evaluated. Color change evaluation results showed that after one month, the least staining was observed in silorane-based composite resin in all the solutions tested, which was consistent with the findings of previous studies [8,18].

This might be justified by the lower water sorption of silorane-based compared to

Immersion period	Staining solutions	Filtek P90	Filtek Z250	Filtek Z350XT
	Hype-Red Bull	0.021	0.406	0.910
1 week	Hype-Artificial saliva	0.450	0.002	0.007
	Red Bull- Artificial saliva	0.059	0.005	0.016
	Hype-Red Bull	0.003	0.705	0.870
1 month	Hype-Artificial saliva	< 0.001	< 0.001	0.004
	Red Bull- Artificial saliva	<0.001	< 0.001	<0.001

**Table 6:** P-values of pairwise comparisons between staining solutions by Mann Whitney U test in terms of  $\Delta E$ 

methacrylate-based composite resins. Lower water sorption of the silorane molecule is attributed to the presence of the hydrophobic siloxane radicals in the formulation of this resin and good synergy between the organic matrix and the filler content [18,19]. Reis et al, [20] showed that hydrophilic materials have higher water sorption and undergo greater discoloration than hydrophobic materials. Another explanation for higher color change in methacrylate-based composite resins can be the occurrence of microcracks due to higher polymerization shrinkage and secondary infiltration of fluids into the organic matrix of these composite resins [21]. The staining susceptibility of a material may also be attributed

to its filler type. Composite resins containing quartz fillers are more resistant to aqueous attack [21]. Yap et al, [22] reported that zirconia glass fillers are also susceptible to aqueous attack. In the present study, Filtek Z250 and Filtek Z350XT contained zirconia/silica fillers, while Filtek P90 contained quartz and yttrium fluoride as inorganic fillers. Therefore, differences in filler composition could be a possible reason for the higher color change in methacrylate-based composite resins in this study.

Based on our results, after one week, discoloration of Filtek Z350XT composite resin was lower than that of the silorane-based composite resin. Many factors can affect the discoloration of a restorative material, including the nature of the monomer, degree of conversion (DC), type, morphology and percentage of the filler particles and type of the staining agent. Boaro et al, [23] reported that DC of a siloranebased composite resin was significantly lower than that of a methacrylate-based composite resin after polymerization. 72 hours However, polymerization of P90 composite continues on the rate of ring-opening depending polymerization and presence of active cations, and DC increases after 24-48 hours. Therefore, higher color change of Filtek P90 during the first week may be due to higher water sorption and loss of the covalent bonds between fillers and matrix during the first 72 hours as a result of lower DC. On the other hand, in longer immersion periods its hydrophobic property reduces the extrinsic color change [23].

results indicated that Filtek Our Z250 microhybrid composite resin was most prone to color change. Contrary to our findings, some previous studies reported a lower color change in Filtek Z250 compared to Filtek Z350XT nanofilled composite resin [5,8]. Filtek Z350XT has almost the same matrix formulation as Filtek Z250 except for the presence of TEGDMA in Filtek Z350XT. Kalachandra and Turner [24] showed that increasing the amount of TEGDMA from 0% to 1% will increase the water sorption of Bis-GMA-based composite resins from 3% to 6%. On the other hand, our results were consistent with the findings of Erdemir et al, [2]

who showed higher color change in Z250 than Filtek Z350XT immersed in different energy drinks. The staining susceptibility of a composite resin might be attributed to its filler type. Inorganic fillers might debond from the matrix, leaving a void, and thus increasing the surface roughness and making the surface susceptible to extrinsic discoloration. The nanofilled composite resin used in our study contained silica fillers with a particle size of 20nm and clusters of zirconia/silica particles with sizes ranging from 0.6 to 1.4µm. It seems that the smaller filler size of Filtek Z350XT nanofilled composite resin might have allowed a smoother surface after polishing, which was less susceptible to exterior staining. Another reason for higher staining of Filtek Z250 could be a high proportion of silane present in the structure of this composite resin. It has been suggested that silanization of filler particles plays an important role in discoloration due to the fact that silane has high water sorption [25,26]. In this study, immersion in artificial saliva induced the lowest discoloration in all tested composite resins, which is consistent with other investigations. Artificial saliva has no pigments; thus the color change might be due to water sorption of the matrix, which results in swelling and plasticizing of the polymer along with interfacial gaps created between the filler and resin matrix that allow stain penetration and discoloration [4,11]. Discoloration of materials also depends on duration of exposure to coloring agents in the oral cavity [27]. As observed in the present study, in all the study groups there was a significant increase in  $\Delta E$  of composite resins with an increase in the immersion time. Increased interaction between the chemical agent and resin, in addition to enhanced penetration of staining substances into resin, might be possible reasons for this finding [2].

According to previous studies,  $\Delta E$  values below 1 are not noticeable by the human eye;  $\Delta E$  values between 1 and 3.3 are considered appreciable by skilled operators, but clinically acceptable;

whereas,  $\Delta E$  values higher than 3.3 are perceptible by non-skilled considered individuals. and are hence not clinically acceptable. All the materials tested in the present study revealed clinically acceptable color change (<3.3) after 30 days of immersion in the solutions. Similar to our findings, Erdemir et al, [12] reported  $\Delta E$  values lower than 3.3 for both Z250 and Filtek Z350XT composite resins immersed in Red Bull and water for one month. In another study, Al-Dharrab [14] reported that  $\Delta E$  values of Filtek Z350XT composite resin were also lower than 3.3 after seven and 30 days of immersion in Red Bull energy drink. Other authors have also observed an acceptable color change in silorane-based composite resin when immersed in water for up to 30 days [8,9,28]. In this study, color change induced by two types of energy drinks was similar for Filtek Z250 and Filtek Z350XT composite resins; however, in Filtek P90 specimens, Red Bull energy drink induced greater discoloration. Both energy drinks used in this study contain caramel as a coloring agent, which is the chemical agent responsible for the pale yellow color of these drinks. Vitamin B2 (riboflavin) is also another ingredient, which is a yellow-orange substance and imparts the yellow color to vitamin supplements. Having the same coloring agents in both drinks may be a reason for similar discoloration seen in Filtek Z250 and Filtek Z350XT samples. The higher color change seen in the Filtek P90 samples immersed in Red Bull might be attributed to compatibility of the matrix phase of this composite resin with the substances present in this drink. It has been reported that a low pH can negatively affect the wear resistance of dental materials and increase the erosion of polymers [8,29,30]. Both energy drinks used in this study contain citric acid and are acidic in nature. The pH values of Hype and Red Bull energy drinks used in this study were roughly the same (3.42 and 3.54, respectively). The organic fillers of a composite resin may degrade upon contact with citric acid and this effect is pHdependent [21]. Benetti et al, [1] on the other hand reported that storage in citric acid did not influence the color change of Filtek Z250, Z350XT and Filtek P90 composite resins. The effect of pH on surface microstructure and roughness of composite resins was not evaluated in the present study; therefore, further studies are recommended to investigate the effect of prolonged consumption of energy drinks with different pH values on staining susceptibility and roughness of these materials.

### CONCLUSION

Within the limitations of this in vitro study, it can be concluded that all solutions used in this study affected the color stability of tested composites. With prolonged immersion periods,  $\Delta E$  values increased for each material showing that stainability of composite resins is time dependent. However, the color change values were within clinically acceptable range for both immersion times and both energy drinks. Color change induced by energy drinks was roughly the same except for Filtek P90 composite resin in Red Bull showing higher color change values. Further studies are recommended to assess the long-term discoloration effects of energy drinks with different compositions on restorative materials.

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