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Research article

Energy drinks alter the surface morphology and roughness of composites, fissure sealants and titanium: An *in vitro study*

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HIGHLIGHTS

- Consumption of energy drinks is increasing amongst children and young individuals.
- Preventive, restorative, and orthodontic materials used in young individuals are affected by energy drinks.
- Roughness and morphological change of dental materials is observed in case of Hell and Burn impact.
- Burn damages more titanium, fissure sealant and composite materials.

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ABSTRACT

Objectives: The influence of energy drinks on dental materials are relatively under addressed. Our aim was to investigate the effect of energy drinks on dental materials used intraorally in young individuals. Commonly used preventive, restorative, and orthodontic materials were tested *in vitro*.

Methods: The effect of two commercially available energy drinks (HELL, BURN) was investigated on different dental materials: machined, anodized Titanium (grade 5: Ti6Al4V) and composites (Grandio Seal, VOCO; Filtek Z250, 3M ESPE; Estelite SQ, TOKUYAMA). The roughness (Ra) and morphological changes were examined by atomic force microscopy (AFM) and scanning electron microscopy (SEM).

Results: AFM and SEM revealed significant differences in the Ra and morphology of the samples. AFM results for the machined and anodized titanium samples showed that the two energy drinks modified the surface roughness differently; BURN changed the roughness of machined samples significantly, while anodized discs were not altered significantly by the two energy drinks. In case of composite samples there was no significant difference for the Estelite SQ, relative low differences for the Filtek Z250 and significant changes in the morphology and surface roughness of Grandio Seal.

Significance: On all tested materials, changes in the surface roughness and morphology were more or less detected, proving energy drinks do in fact have a harmful effect. It can be concluded that material erosion depends on the material composition and particle arrangement. Where the surface is characterized by a regular, uniform particle arrangement, energy drinks are less able to influence the roughness, while for samples where the surface is rich in aggregates, the material erodes the surface much more easily.

1. Introduction

The consumption of energy drinks has become increasingly popular among children, adolescence, and young individuals worldwide, as they are advertised to provide higher athletic performance, concentration, attention, wakefulness, to increase energy and reduce physical and mental stress [1]. Excessive consumption is further exaggerated by the fact that they are easily accessible as they are sold in numerous places [2, 3].

Many potential health problems have been proved to be related to the heavy and chronic consumption of these popular products including

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migraines, anxiety, agitation, gastrointestinal irritation, insomnia, arrhythmias and other cardiovascular complications [4, 5, 6]. Their effects on dental materials are relatively under addressed, although these are important aspects to consider as well. In young individuals, preventive, restorative, and orthodontic materials are used primarily to maintain or improve their oral health.

Energy drinks usually contain several substances such as caffeine, guarana, taurine, ginseng, vitamins, herbal supplements, and sugar that are responsible for these features [1]. Due to their acid content, energy drinks can also cause erosion of teeth [7, 8, 9, 10, 11]. Dental erosion is a process in which tooth loss occurs due to acids that are not derived from microorganisms. If the acidic effect persists, the superficial tooth loss spreads deeper and deeper, and over time a visible defect develops [12, 13].

Previous studies showed that the consumption of Coca-Cola had an erosive effect on the surface of different restorative CAD/CAM materials (composite, resin and ceramics) as the surface presented different microhardness values before and after acid exposure [14, 15].

In addition to fissure sealing, proper nutrition is the other determinant factor of primary prevention. The development of caries is highly correlated with the amount of carbohydrates ingested in one's diet, the main sources of which are sugary beverages [16]. Sugar consumption is one of the major etiological factors in the development of caries. The relationship between tooth decay and sugar consumption is influenced by many factors, such as the availability of sugar to bacteria, or the presence of acid-producing bacteria in the dental plaque [17, 18, 19]. Fluoride and saliva are also important features in the fight against bacteria and acids [20].

Many teenagers consume alcoholic beverages along with energy drinks. Several studies have shown that consuming both drinks together reduce the perception of the presence of intoxication and increases the desire to drink [21]. Reissig et al. have revealed that some genetic factors also play a role in whether an individual consuming an energy drink develops caffeine intoxication or dependence [1].

Ruiz et al. have shown that teenagers who regularly consume energy drinks are more prone to smoking, alcohol consumption, and illegal drug use. This suggests that children and teenagers are not recommended to consume energy drinks at all [22].

Dental professionals should be familiar with energy drinks and their potential consequences on dental materials [23]. Therefore, our aim was to *in vitro* investigate the effect of two commercially available energy drinks (HELL and BURN) on the surface properties of different commonly used dental materials in young individuals (Table 1).

2. Materials and methods

2.1. Specimen preparation

Discs were fabricated from five different materials. 1. Machined (non-treated, or turned) titanium, 2. Anodized titanium. The Ti samples (discs of 1.5 mm thickness and 9 mm in diameter) were cut from ASTM F136 grade 5 Titanium alloy rods (Denti System®, Hungary). The cutting method produced a surface roughness matching the criteria of an abutment, according to Bollen et al, proven by the AFM measurements [24]. The anodized samples were cut from the same type of rod and an anodizing procedure was applied according to the protocol of Wieland Edelmetalle Gmbh. (Germany). The following composite materials were used: 3. Grandio Seal (VOCO, Germany) light-curing nano-hybrid fissure sealer, 4. Filtek Z250 (3M Espe, India, Karnataka; material composition: Bis-GMA, UDMA, Bis-EMA matrix, 60 m% silica/zirconia, average particle size 0.6 μm, particle size range: 0.01–3.5 μm), and 5. Estelite Sigma Quick (Tokuyama Dental Corp., Japan; material composition: 82 m% silica-zirconia and composite filler, average particle size: 0.2 μm, particle size range: 0.1–0.3 μm, monomer matrix Bis -GMA and TEGDMA). Ten discs of each material were prepared.

Table 1. Compositions of the experimental beverages as listed on their respective packaging. Nanofil (Estelite Sigma Quick, Tokuyama) and microfil (Filtek Z250, 3M ESPE) composites, a fissure sealant (Grandio Seal, VOCO) on Table 2 and CP Grade 5 Ti materials were tested as restorative, preventive and orthodontic materials, respectively.

HELL	water, sugar, citric acid, carbon dioxide, taurine, sodium citrate, caffeine, flavours, color, vitamins
BURN	water, sugar, citric acid, carbon dioxide, taurine, sodium citrate, caffeine, flavors, color, vitamins, guarana, ginseng, arginin, ascorbic acid, maltodextrin, glucunolactone, E150d, E202

2.2. Surface treatment with energy drinks

Surface of the discs were treated by two different types of commercially available energy drinks BURN (Monster Beverage Corp., Corona, California) and HELL (Hell Energy Hungary Ltd., Szikszó, Hungary) using the following protocol: first, the discs were cleaned with 70% ethanol and after soaking for 15 min, they were placed in distilled water and allowed to stand for 10 min. Following the cleaning protocol, the discs were immersed into energy drinks for 30 min [13]. After that, all the discs were rinsed with distilled water, dried and placed into a sterile screw-type plastic cup until surface examinations were performed. As a control, the discs were just treated with the cleaning protocol. All specimens were immersed at 37 °C degree, as it is important to reproduce the temperature of the oral cavity [11].

2.3. Surface roughness analysis using atomic force microscopy (AFM)

The surface roughness (Ra) and morphology of the samples was studied with AFM (PSIA XE-100 instrument, PSIA Inc., South Korea). The damping of the cantilever's oscillation amplitude due to the intermittent tip-sample contact is used for surface profiling. The tips were single-crystal silicon cantilevers (type: N, NSG30 series with Au reflective coating, resonant frequency 240–440 kHz, force constant 22–100 N/m) purchased from NT-MDT (Russia). At least six independent measurements were performed for each sample in tapping mode, and the height, deflection, and 3D images with areas of 20 μm × 20 μm and 5 μm × 5 μm were captured. Ra was determined using the AFM software program as the arithmetic average of the surface height relative to the mean height [25].

2.4. Surface profile analysis using scanning electron microscopy (SEM)

Treated and non-treated samples were investigated with a scanning electron microscope (SEM) equipped with a built-in energy dispersive X-ray spectrometer (EDS) (Jeol JSM-IT500HR, Jeol, Tokyo, Japan). Fast and high accuracy elemental analysis was provided using the dry silicon-drift (SDD) EDS detector [26, 27].

Images were captured using secondary electron imaging mode and 5, 10 and 15 kV accelerating voltage. Samples were coated with gold (Jeol JFC-1300 auto fine coater, Jeol, Tokyo, Japan). The morphological characteristics of the treated and non-treated discs were recorded at 500, ×2000, ×5000, ×10000 and ×20000 magnifications and were tilted at 45° for a better surface visualization.

Table 2. Ingredients of the experimental dental materials as listed on their respective packaging.

Estelite SQ	(1- Metil-Ethilidene) -Bis [4,1-Fenilenoxi (2-Hidroxi-3,1-Propane-diil)] Bismetacrilate, 2,2′-Ethilendioxidiethil Dimetacrilate, 2,6-di-Tert-Butil-P-Cresole, DL-Bornane-2,3-dione, Mequinole
Filtek Z250	Silane Treated Ceramic, Bisphenol A Diglycidyl Ether Dimethacrylate (BISGMA), Bisphenol A Polyethylene Glycol Diether Dimethacrylate (BISEMA6), Diurethane Dimethacrylate (UDMA), Triethylene Glycol, Dimethacrylate (TEGDMA), Aluminum Oxide, N,N-Dimethylbenzocaine
Grandio Seal	Triethylene Glycol Dimethacrylate, Fumed silica, Bisphenol A Diglycidyl Ether Dimethacrylate (BISGMA)

2.5. Statistical analyses

For the Ra (nm) values of the surfaces measured by AFM the arithmetic means ± the standard error of the mean (SEM) was calculated and plotted. The data were compared via one-way analysis of variance (ANOVA), after normality testing. These were followed by Tukey's HSD, LSD and Scheffé post hoc tests to determine statistical differences after multiple comparisons (SPSS 21, SPSS, Chicago, Illinois, USA). The level of significance was established at $p = 0.05$.

3. Results

3.1. AFM examinations of Ti samples

The surfaces of the control and treated Ti discs were examined using AFM in the 5 μm × 5 μm and 20 μm × 20 μm areas.

In Figure 1a, a typical machined Ti surface can be seen, with grooves running in parallel on the surface, and small burrs are also visible. The grooves arise from the cutting tool used for machining the sample. On the AFM images the color is becoming brighter from the depth of the fissure upwards. AFM measurements for the samples with a turned surface (control group), in the range of 5 μm × 5 μm gave a surface roughness Ra = 15 ± 1 nm. HELL-treated specimens had a roughness of 8 ± 1 nm (Figures 1b and 3), significantly not different from the non-treated samples (p = 0.301). BURN-treated specimens had a roughness of 124 ± 11 nm (Figures 1c and 3), significantly different from the control samples (p < 0.0001).

AFM measurements in the 20 μm × 20 μm range gave a surface roughness of Ra = 63 ± 6 nm in the control (turned surface) group (Figures 1d and 3). The HELL-treated specimens had a roughness of 50 ± 2 nm (Figures 1e and 3), significantly not different from the control surfaces (p = 0.071). BURN-treated specimens had a roughness of 100 ± 6 nm (Figures 1f and 3), significantly different from the control group (p = 0.016).

For the anodized samples AFM measurements gave a surface roughness Ra = 101 ± 7 nm in the 5 μm × 5 μm range (Figures 2a and 3). The HELL-treated specimens had a roughness of 103 ± 0.4 nm (Figures 2b and 3), significantly not different from the non-treated group (p = 0.792). The BURN-treated specimens had a roughness of 77 ± 5 nm (Figures 2c and 3), significantly not different from the non-treated group (p = 0.369).

AFM measurements for the anodized samples in the 20 μm x 20 μm range gave a surface roughness Ra = 96 ± 5 nm (Figures 2d and 3). The HELL-treated specimens had a roughness of 118 ± 11 nm (Figures 2e and 3), significantly not different from the non-treated group (p = 0.073). The BURN-treated specimens had a roughness of 114 ± 10 nm (Figures 2f and 3), significantly not different from the non-treated group (p = 0.147).

Figure 3 shows the overall bar graph of Ti samples, measured at different sample sizes. In the 5 μm × 5 μm range, HELL smoothed the turned, roughened the anodized (not significantly), while BURN roughened significantly the turned and slightly flattened the anodized. In the 20 μm × 20 μm range, HELL non significantly flattened the turned and roughened the anodized, while BURN roughened significantly the turned and not-significantly the anodized samples (see Figure 3).

Comparing the values measured at different samples sizes, the higher the resolution, the greater the roughness difference between the turned and anodized samples. The reason for the significantly lower Ra values is that in the 5 μm × 5 μm range the field of view is smaller and the grooves are completely undetectable. In case of anodized samples, in the small field of view the granular texture will be predominant and not the parallel grooves typical for the turned surfaces. The granules have a diameter between 100–300 nm and height between 1-2 μm. The major factor of the roughness is, produced by the anodization process.

The two energy drinks modified the roughness of the surfaces differently: BURN energy drink had a more significant effect on both surfaces than HELL; the turned surfaces were significantly modified by BURN and the anodized samples were not altered by the energy drinks.

3.2. AFM studies of composite samples

The surfaces of the control and treated composite discs were examined using AFM in the same manner as for titanium discs, in the same areas of 5 μm × 5 μm and 20 μm × 20 μm.

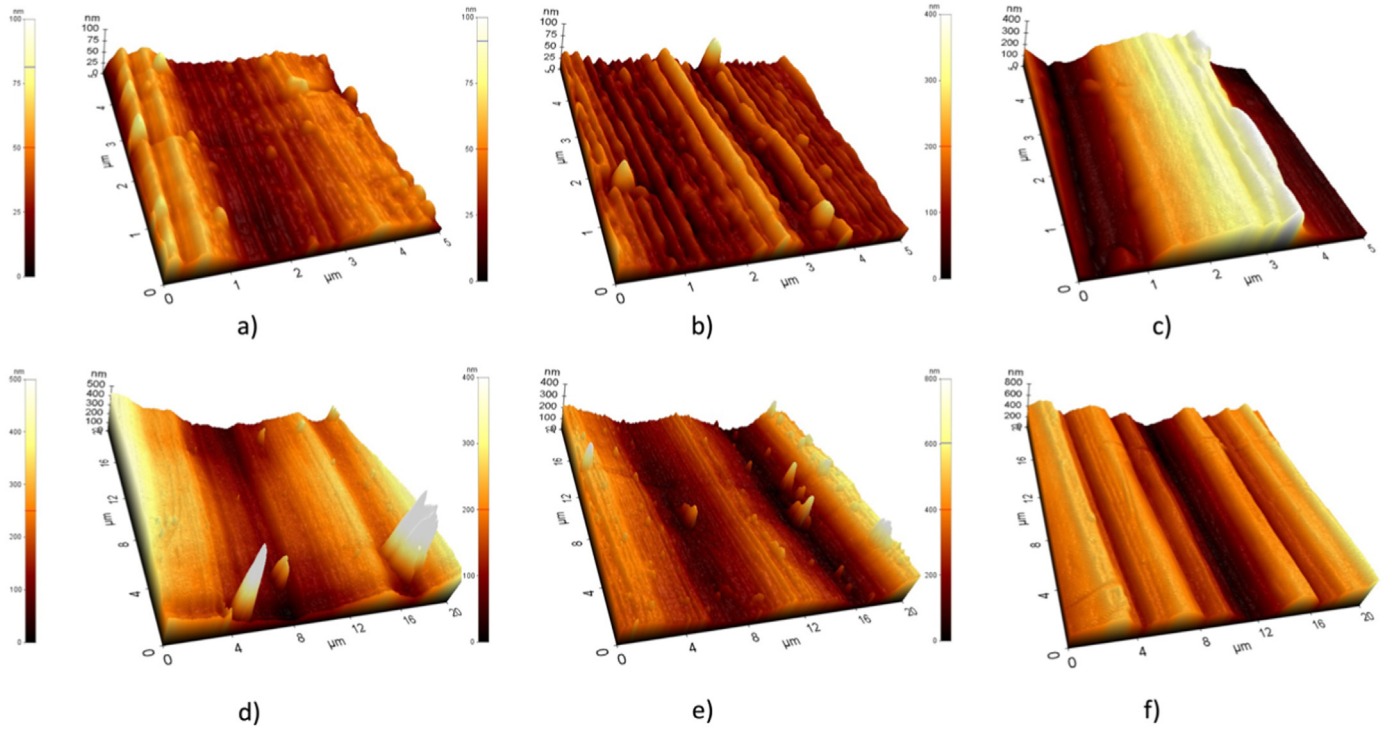

Figure 1. AFM measurements of turned titanium discs in 5 μm × 5 μm range a) control, b) HELL-treated, c) BURN-treated and in 20 μm × 20 μm range d) control, e) HELL-treated, f) BURN-treated (3D images).

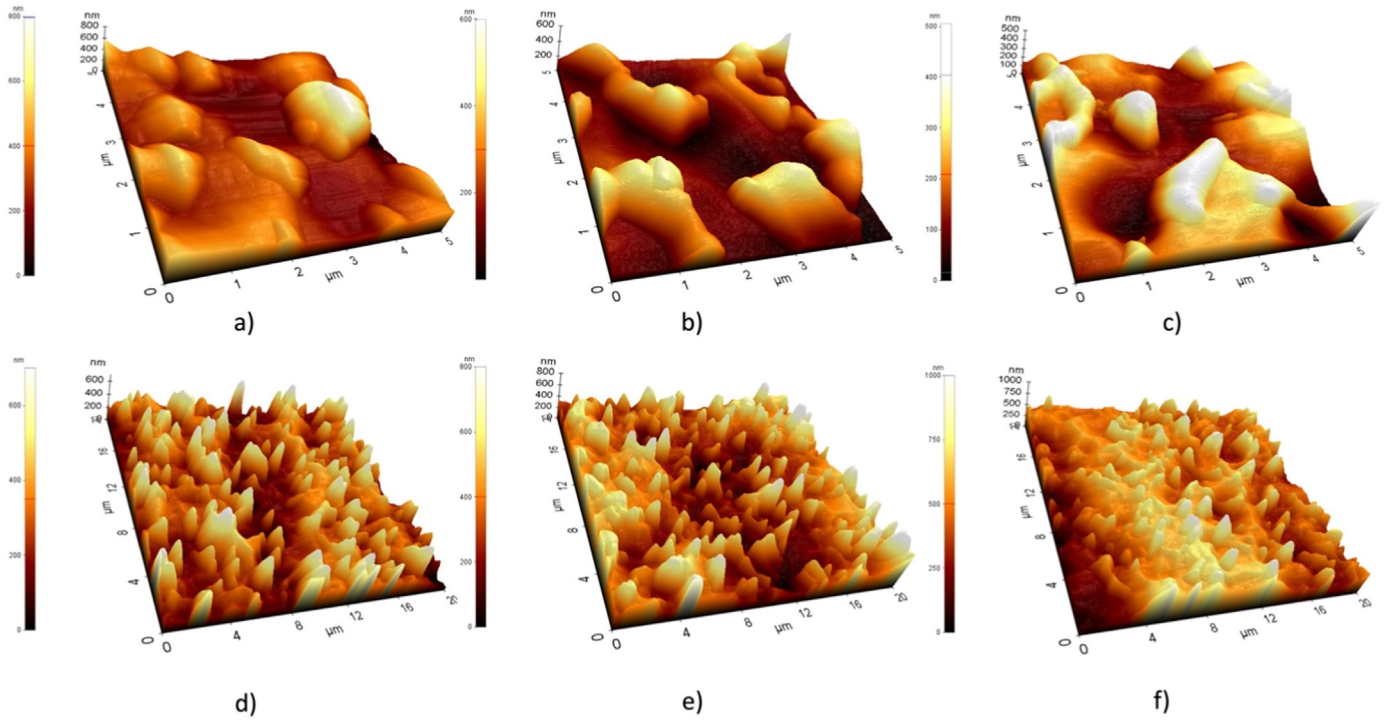

Figure 2. AFM measurements of anodized titanium discs in 5 μm × 5 μm range a) control, b) HELL-treated, c) BURN-treated and in 20 μm × 20 μm range d) control, e) HELL-treated, f) BURN-treated (3D images).

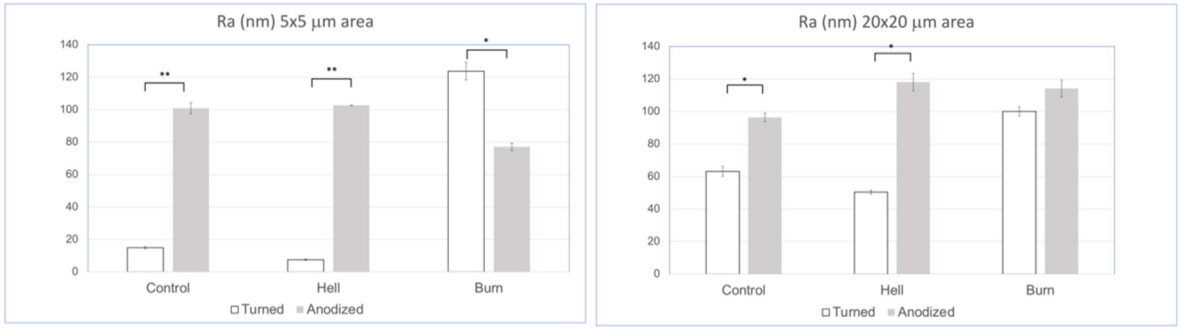

Figure 3. Comparative bar graph of Ra (nm) of titanium samples in the 5 μm × 5 μm and in the 20 μm × 20 μm area. Asterisks denote significant differences (*$p < 0.05$ and **$p < 0.0001$).

3.2.1. Grandio seal

AFM measurements in the 5 μm × 5 μm range for the non-treated (control) Grandio Seal samples gave a surface roughness Ra = 124 ± 11 nm (Figures 4a and 7). Roughness was 105 ± 8 nm for HELL-treated samples (Figures 4b and 7), significantly not different from the control group ($p = 0.156$). For BURN-treated specimen's Ra was 152 ± 11 nm (Figures 4c and 7), significantly not different from the control group ($p = 0.108$).

AFM measurements in the range of 20 μm × 20 μm gave a surface roughness Ra = 460 ± 36 nm in the control group for the Grandio Seal samples (Figures 4d and 7). The HELL-treated specimens had a roughness of 237 ± 19 nm (Figures 4e and 7), significantly different from the control group ($p < 0.0001$). The BURN-treated specimens had a roughness of 251 ± 18 nm (Figures 4f and 7), also significantly different from the control group ($p < 0.0001$).

3.2.2. Filtek Z250 composite

AFM measurements at 5 μm × 5 μm sample size gave a surface roughness of Ra = 47 ± 6 nm in the control group of Filtek Z250 composite (Figures 5a and 7). Roughness was 78 ± 6 nm for HELL-treated samples (Figures 5b and 7), significantly different from the control group ($p = 0.0014$). For BURN-treated specimen's Ra was 93 ± 6 nm (Figures 5c and 7), significantly different from the control group ($p < 0.0001$).

AFM measurements in the 20 μm × 20 μm range gave a surface roughness Ra = 70 ± 8 nm for the control Filtek Z250 samples (Figures 5d and 7). Ra was 126 ± 11 nm for the HELL-treated samples (Figures 5e and 7), significantly different from the control group ($p = 0.0003$). The BURN-treated specimens had a roughness of 228 ± 13 nm (Figures 5f and 7), significantly different from the control group ($p < 0.0001$).

3.2.3. Estelite sigma quick composite

AFM measurements for control Estelite S Q samples, gave a surface roughness Ra = 41 ± 4 nm in the 5 μm × 5 μm range (Figures 6a and 7). Roughness was 54 ± 4 nm for HELL-treated samples (Figures 6b and 7), significantly not different from the control group ($p = 0.067$). For BURN-treated specimen's Ra was 48 ± 3 nm (Figures 6c and 7), significantly not different from the control group ($p = 0.229$).

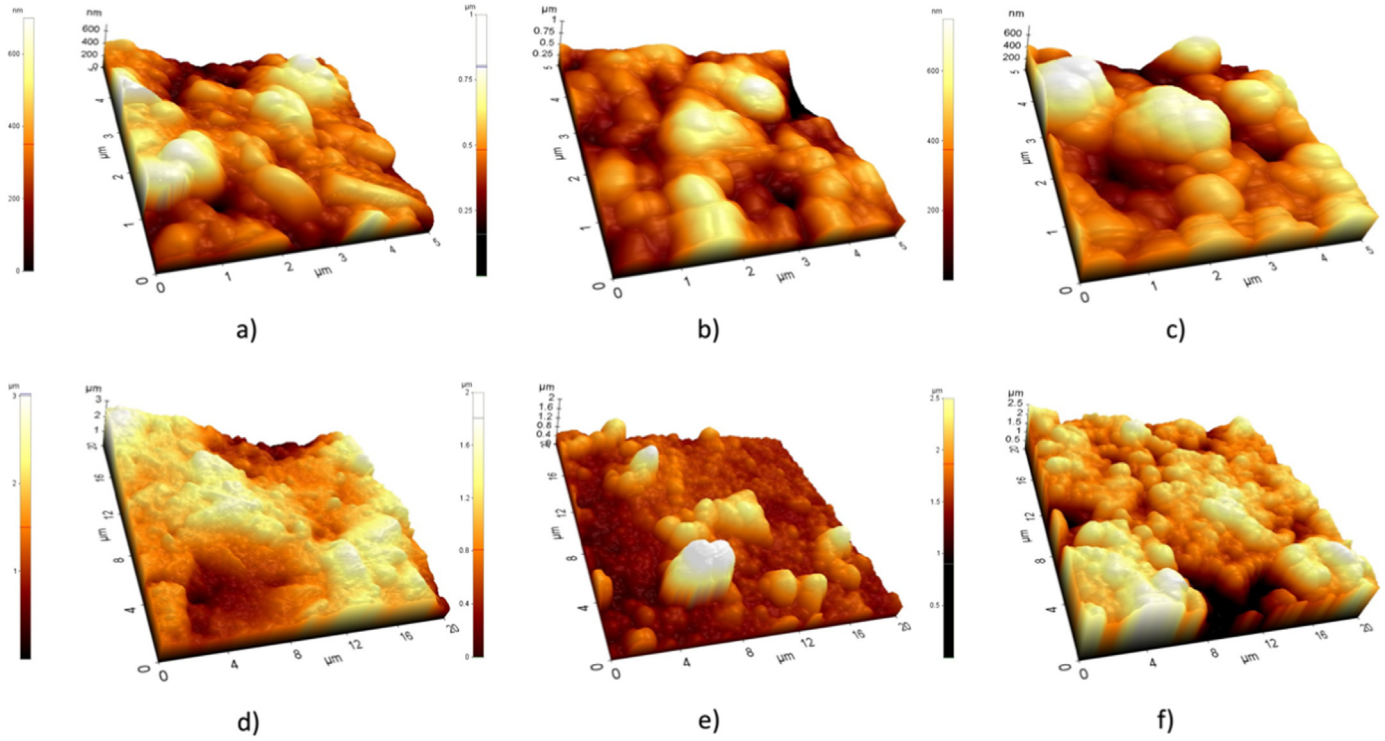

Figure 4. AFM measurements of Grandio Seal discs in 5 μm × 5 μm range a) control, b) HELL-treated, c) BURN-treated and in 20 μm × 20 μm range d) control, e) HELL-treated, f) BURN-treated (3D images).

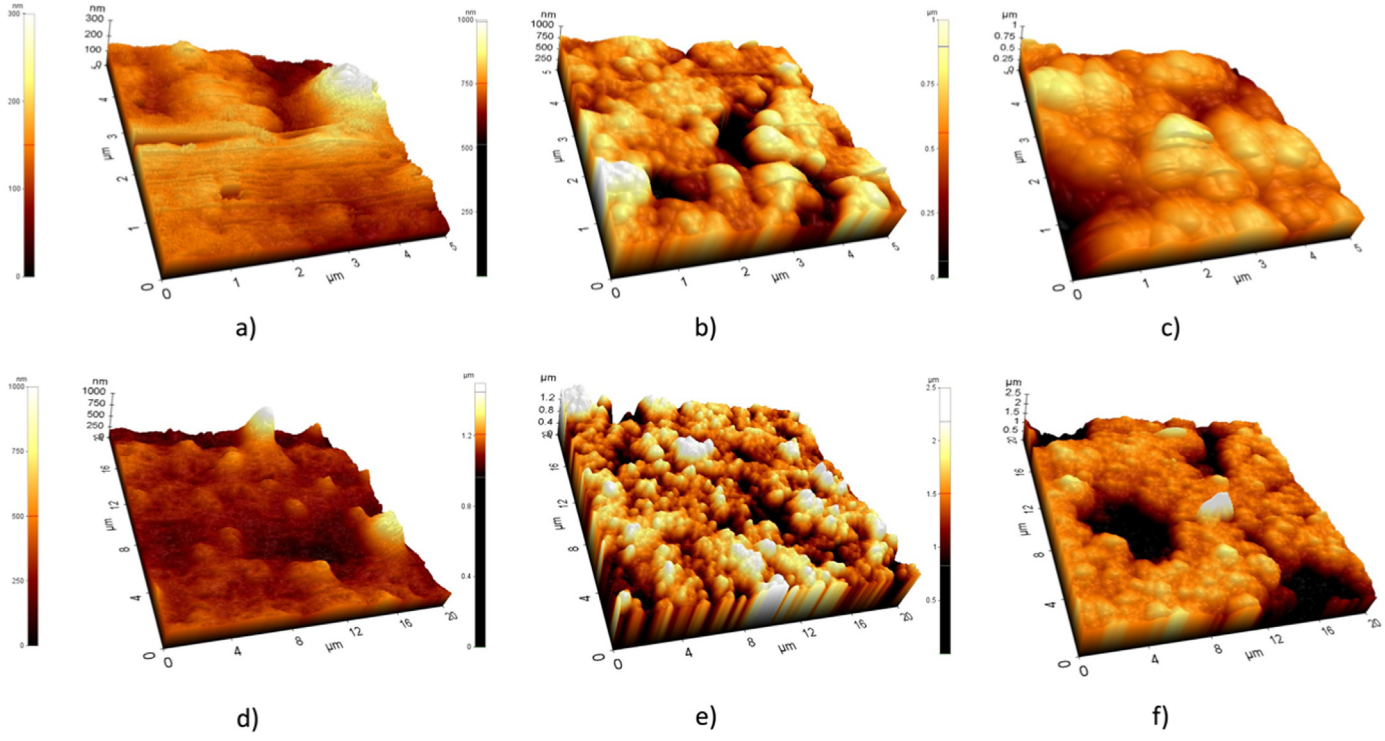

Figure 5. AFM measurements of Filtek Z250 discs in 5 μm × 5 μm range a) control, b) HELL-treated, c) BURN-treated and in 20 μm × 20 μm range d) control, e) HELL-treated, f) BURN-treated (3D images).

AFM measurements for the control Estelite S Q samples in the range of 20 μm × 20 μm gave a surface roughness Ra = 112 ± 11 nm (Figures 6d and 7), while on the HELL-treated samples 127 ± 11 nm (Figures 6e and 7), significantly not different from the control group ($p = 0.389$). Ra was 95 ± 7 nm (Figures 6f and 7) on the BURN-treated samples, significantly not different from the control group ($p = 0.189$).

Figure 7 displays the overall bar graph of composite samples, measured at different sample sizes. In the 5 μm × 5 μm range, HELL

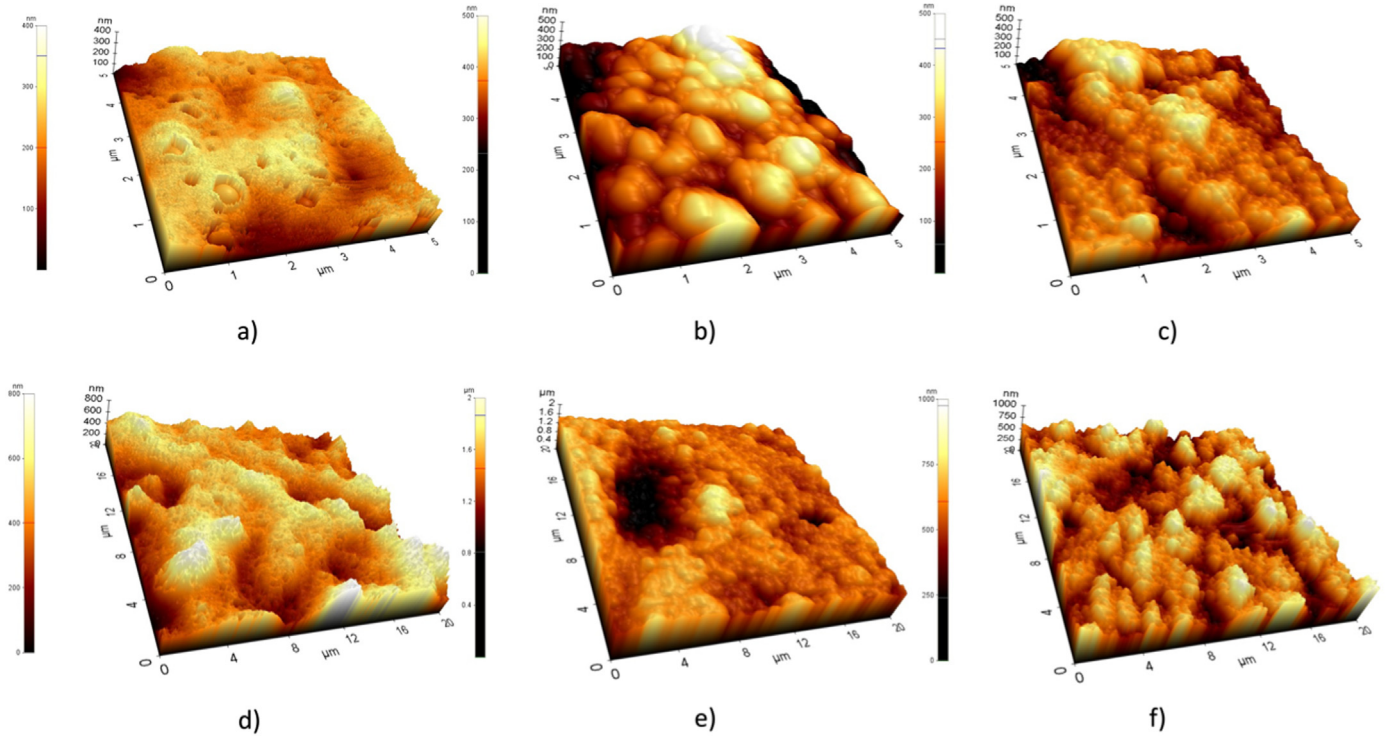

Figure 6. AFM measurements of Estelite S Q discs in 5 μm × 5 μm range a) control, b) HELL-treated, c) BURN-treated and in 20 μm × 20 μm range d) control, e) HELL-treated, f) BURN-treated (3D images).

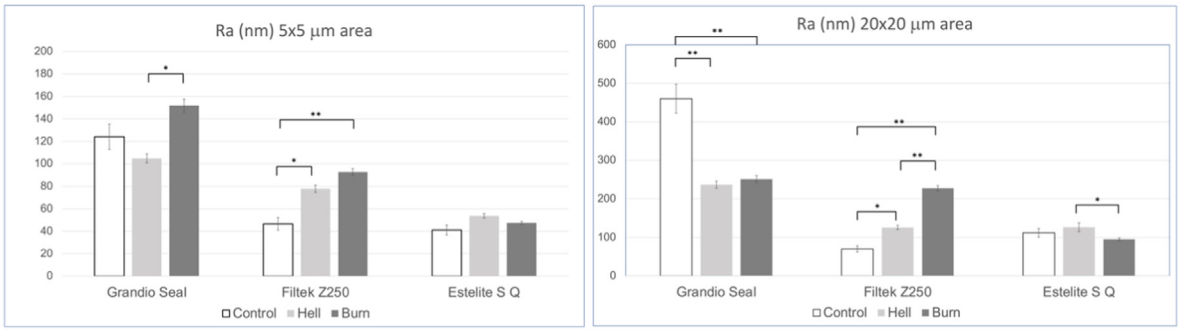

Figure 7. Comparative bar graph of Ra (nm) values of composite samples in the 5 μm × 5 μm and in the 20 μm × 20 μm area. Asterisks denote significant differences (*$p < 0.05$ and **$p < 0.0001$).

significantly roughened Filtek Z250, not significantly roughened Estelite S Q samples and not significantly smoothed Grandio Seal. BURN significantly roughened Filtek Z250, slightly Grandio Seal and Estelite S Q. In the 20 μm × 20 μm range, HELL significantly flattened Grandio Seal and significantly roughened Filtek Z250, while not significantly roughened Estelite S Q. BURN significantly smoothed Grandio Seal and slightly Estelite S Q. BURN significantly roughened Filtek Z250 (see Figure 7).

Comparing the values measured at different samples sizes, the higher the measured size area, the greater the roughness of the samples. The reason for that are the different fields of view. Furthermore, the two energy drinks modified the roughness of the surfaces differently: BURN energy drink had a more significant effect on all three surfaces than HELL. Beside that the different composites behave differently to the impact of the energy drinks. Grandio Seal suffered the biggest change regarding roughness and morphology, followed by Filtek Z250. The least damage underwent Estelite S Q samples. This effect is due to the different composition of the materials and to the inhomogeneity of the size of the granules of the samples. Estelite S Q samples present the most homogeneous granules size between the samples (particle size range: 0.1–0.3 μm), and therefore it is less effected by the energy drinks.

3.3. SEM examination of Ti samples

SEM images (Figure 8) revealed considerable differences in the surface morphology and structure of the investigated samples. Turned (control) discs had circular grooves originated from the cutting tool used for cutting the samples (Figure 8(a, b, c)). Parts of circular grooves made by the cutting tool are visible, with small burrs on the machined (turned) surface.

Small granules developed on the surface of anodized Ti6Al4V samples (Figure 8(d, e, f)). These discs exhibited typical, irregular surface characteristics with small grains of 0.1–3 μm size.

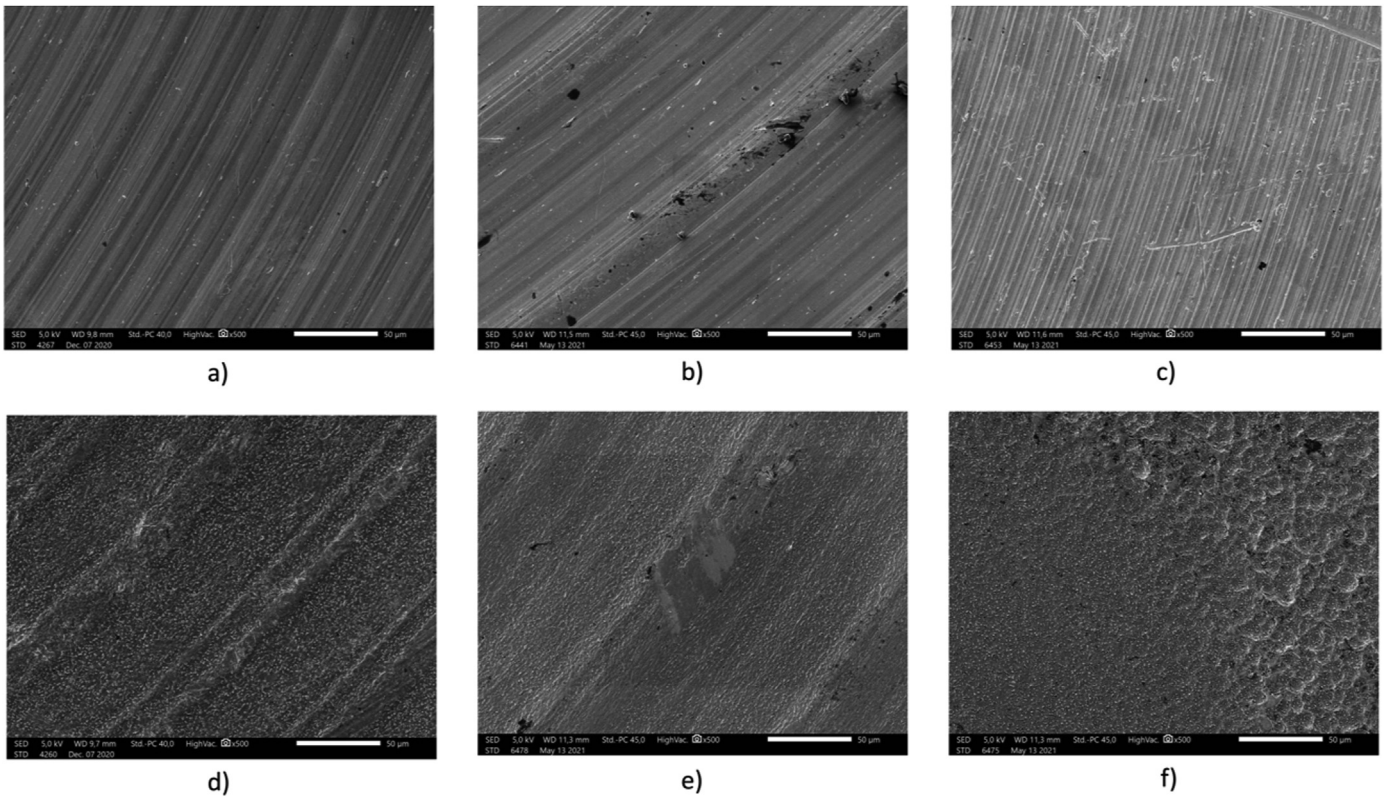

Figure 8. SEM images of titanium discs at ×500 magnification of a) non-treated turned (control) b) HELL-treated turned c) BURN-treated turned d) non-treated anodized e) HELL-treated anodized f) BURN-treated anodized.

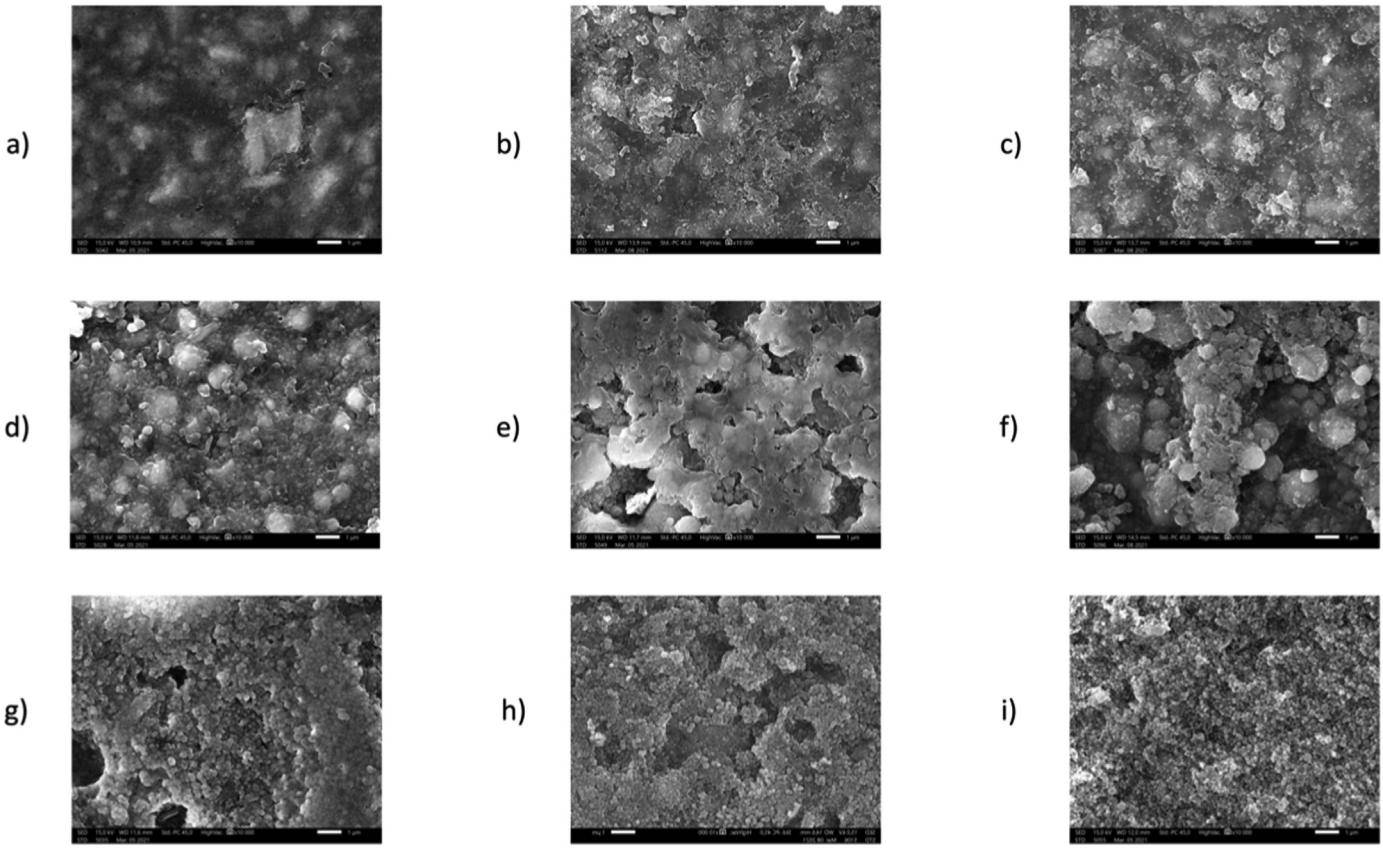

Figure 9. SEM examination at ×10.000 magnification 1; Grandio Seal a) control b) HELL-treated c) BURN-treated. 2; Filtek composite d) control e) HELL-treated f) BURN-treated. 3, Estelite S Q composite g) control h) HELL-treated i) of BURN-treated.

3.4. SEM measurements of composite samples

3.4.1. Grandio seal composite

SEM images (Figure 9(a, b, c)) revealed an inhomogeneous granular structure for Grandio Seal fissure sealer and due to the energy drink treatments changes are visible in the morphology and structure of this material.

3.4.2. Filtek Z250 composite

Filtek Z250 composite material SEM images are presented in Figure 9(d, e, f). The images reveal the granular composition of this material, with particle size range: 0.01–3.5 μm, in accordance with the AFM measurements. HELL and BURN energy drinks caused material removal and roughness increase.

3.4.3. Estelite S Q composite

Figure 9(g, h, i) shows typical SEM images of Estelite S Q composite. For this material also a granular structure is characteristic, with a more homogeneous particle size: 0.1–0.3 μm. Energy drink treatment did not change significantly the structure of the material, as the same particle size and structure can be seen.

4. Discussion

Consumption of energy drinks has begun increasingly popular. However, their effects on dental materials are relatively under addressed. Due to their acid content and several substances energy drinks can also cause erosion of teeth [1]. Sajadi et al. proved that Coca-Cola decreased the shear bond strength of orthodontic brackets [28].

Our aim was to *in vitro* investigate the effect of the energy drinks on dental materials used intraorally in young individuals. Discs were fabricated of 5 different widely used dental materials (machined and anodized titanium, three different types of composites). They were treated with popular energy drinks.

The results of AFM and SEM studies on titanium, the two types of composites, and the fissure sealant discs under the limitations of this study confirmed that energy drinks cause a significant change in the surface roughness and morphology of the various dental materials. The effects observed in this *in vitro* model can be different in the oral cavity, as this is an environment protected by saliva, electrolytes, and proteins. The pH and the buffering capacity of these drinks are of upmost importance [10, 13].

The surface roughness determined by AFM depends on the field of measurement due to the different macroscopic features of the surfaces. The turned (machined) samples presented typical grooves due to the concentric circles created by the cutting tool which are visible in the best way on the 20 μm × 20 μm size images. For the anodized samples the anodization process created a granular structure overlapping on the original turned surface. AFM images measured at 20 μm × 20 μm and 5 μm × 5 μm sample size revealed these features and showed significant difference compared to the turned samples. In general anodization gave a rougher surface, but this surface is still in the range of the smooth surfaces (0.01–0.13 μm).

The AFM results for the turned and anodized titanium samples showed that the two energy drinks modified the surface roughness differently, as BURN energy drink had a more significant effect on both surfaces than HELL. The effect of the BURN energy drink was most significant in the 5 μm × 5 μm range, as it significantly roughened the turned sample and just slightly smoothed the anodized one. The reason for that can be the thicker TiO_2 layer of the anodized sample. This protective layer prevents the erosive effect of the energy drink [29, 30].

In the case of the composite samples, neither BURN nor HELL energy drink caused a significant difference in surface roughness for the Estelite SQ composite, which is due to the composition and uniform grain size structure. Based on the SEM and AFM measurements of the Filtek Z250 composite surface, it can be stated that the surface layer is also characterized by a relatively uniform grain size, compared to the Grandio Seal discs. Both control and treated discs underwent much less surface roughness change than Grandio Seal samples. The surface structure of the fissure sealant discs is characterized by disordered location of the grains, different particle sizes and aggregate formation.

5. Conclusion

Under the limitations of this study it can be concluded that machined Ti surfaces are more vulnerable to energy drinks than anodized Ti surfaces. Furthermore, it was established that for composite materials with different composition and different particle arrangement, the impact of BURN and HELL energy drinks is altered. Where the surface is characterized by a regular, uniform particle arrangement, energy drinks are much less able to affect the roughness, while for samples where the surface is rich in aggregates, the material erodes much more easily the surface. Besides this we must consider the different compositions of BURN and HELL energy drinks, as these may play an important role also.

Declarations

Author contribution statement

Bela Kolarovszki: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Alíz Sándor: Conceived and designed the experiments; Performed the experiments.

Péter Szabó: Performed the experiments; Analyzed and interpreted the data.

Judit Kopniczky: Performed the experiments; Contributed reagents, materials, analysis tools or data.

Dorottya Frank: Contributed reagents, materials, analysis tools or data; Wrote the paper.

Ákos Nagy: Analyzed and interpreted the data.

Kinga Kinga Turzó: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

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Data availability statement

Data will be made available on request.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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