Effects of Freeze-Drying on Sensory Characteristics and Nutrient Composition in Black Currant and Sea Buckthorn Berries

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Abstract: Fresh berries contain numerous components that can undergo complex changes during the drying process. This study aims to investigate the effect of freeze-drying on the sensory and chemical properties of black currant and sea buckthorn berries. Freeze-drying was performed at a shelf temperature of 35–55 °C with a step of 5 °C and durations of 18, 20, 22, and 24 h. Comparing the final freeze-dried berries with their fresh counterparts, it was observed that at a shelf temperature of 50 °C and a drying time of 18 to 20 h, there was a minimal loss in the content of vitamins, organic acids, and carbohydrates. However, based on organoleptic evaluations, the best results were achieved after drying for 20 h. Furthermore, the preservation of citric and malic acids in black currant berries, along with citric, tartaric acids, and sucrose in sea buckthorn berries, was only at 45.6% when the freeze-drying time was extended to 22 h. Considering the physical and chemical properties of listed freeze-dried berries, the optimal parameters were identified as a shelf temperature of 50 °C and a drying time of 20 h. The findings from this study serve as a foundation for selecting appropriate freeze-drying parameters for various types of berries.

Keywords: black currant; sea buckthorn; freeze-drying; organic acids; sugars; vitamins; sensory characteristics

1. Introduction

Berries belong to the functional products of nutrition, and their consumption has a positive effect on human health, rendering a therapeutic effect, due to their biologically active compounds and nutrients [1–3]. They are consumed in fresh, frozen, and dried variations, mainly as ingredients in various food products and additives [4]. Freeze-dried berries, thanks to their content of bioactive compounds and extended shelf life without compromising nutritional value, are used as ready-made functional products [5] and as semi-finished products for further industrial processing in sectors such as confectionery, meat, dairy, etc. In recent years, sublimated berries have been considered carriers of probiotics [6].

Black currant (Ribes nigrum L., Grossulariaceae) is a small perennial shrub that is grown in various parts of the world with a temperate climate. These berries are important due to their high nutritional value, which is retained even in processed products. Berries are consumed fresh in households, while on an industrial scale, they are processed into a wide range of products such as juices, jams, jellies, yogurts, and fruit bars. Sea buckthorn
(Hippophaerhamnoides L.) is a shrub plant native to temperate regions of Asia and Europe, utilized worldwide both in traditional medicine for treating various ailments and in the food industry for producing jams, juices, non-alcoholic beverages, liqueurs, wines, or as an addition to beer, kefir, and cheeses. Oil extracted from its seeds and pulp is used as a cosmetic and food additive, and less commonly as a culinary ingredient. Production waste can serve as a functional ingredient in meat or animal feed [7,8].

Black currant [9] and sea buckthorn [10] have high antioxidant, immunomodulatory, cardioprotective, anticarcinogenic, anti-inflammatory, and other beneficial properties [8,11–13]. There are studies on [14] producing lyophilized berry candies based on black currant, yogurt, and various sweeteners as a healthy alternative to traditional lollipops.

Black currant and sea buckthorn berries are rich in vitamins, unlike strawberries and raspberries, since they have an increased content of provitamin A (carotenoids), B vitamins (thiamine, niacin), vitamin C (ascorbic acid), and vitamin E (tocopherol) [10,15]. The vitamin C content in black currant berries ranges from 120 to 280 mg/100 g [16,17], in sea buckthorn berries it can range from 250 to 300 mg/100 g [18,19].

Organic acids and sugars are also important components affecting the qualitative indicators of berries [20]. Their quantitative content varies depending on the time of harvest [21], varietal characteristics, and soil-climatic conditions of growing [22]. The ratio of sugars and organic acids is responsible for the taste, color, and aroma of berries [23,24]. For example, sweet-tasting berries usually contain relatively high levels of sugars and low levels of organic acids [25].

In the studies by Niestruk et al. [26], it was found that in ripe black currant berries, the amounts of sugars vary in the range of 7 to 10 g/100 g and mainly consist of fructose, glucose, and sucrose. Among the organic acids, citric acid predominates, with its content ranging from 2473.43 mg/100 g to 3336.95 mg/100 g [27,28]. The main carbohydrates in ripe sea buckthorn berries are glucose and fructose [29], whose total content is between 2.7 to 5.8 g/100 g. According to studies by Raffo et al. [30], malic acid prevails in sea buckthorn berries, the content of which ranges from 1940 to 4660 mg/100 g, and there is also a relatively high content of quinic acid 810 to 2820 mg/100 g and citric acid 90 to 160 mg/100 g.

Berries are seasonal and perishable products, so different processing methods are used to preserve their quality for a long time. One of the effective methods is berry drying, which allows for increasing the duration of storage while reducing their volume and weight [31]. Many drying methods such as convective, osmotic [32,33], vacuum [34], and solar [35] are used worldwide. However, their use leads to darkening, degradation of biologically active compounds, and deterioration of the texture and taste of dried berries [36,37].

Despite its high energy cost, freeze-drying is recognized as one of the gentlest drying methods, preserving biologically active compounds, vitamins [38–45], phenolic substances [46], and carotenoids [47]. In addition to a high rehydration rate, this method is able to preserve the original color and distinctive flavor of fresh berries. Despite the fact that freeze-drying is a widespread technology, the issues of its effect on the organoleptic and physicochemical properties of berries which are difficult to dry, due to having a waxy outer shell, have been insufficiently studied.

Therefore, our research is devoted to assessing the changes (organoleptic properties, vitamins, organic acids, and sugars) in the process of freeze-drying black currant and sea buckthorn berries in order to control the quality of finished products.

2. Materials and Methods

2.1. Samples and Material

The berries of the black currant variety “Altaiskaya rannaya” and sea buckthorn variety “Jemovaya” were investigated in the conducted studies. Berries were collected in the phase of full ripeness in the Almaty region, Esik settlement, IE “SAO” (Kazakhstan).

The reagents used for the analysis of water-soluble vitamins were trichloroacetic acid (TCA) with a purity of ≥99.0% purchased from Sigma-Aldrich, while sodium hydrophos-
phate and 85% orthophosphoric acid were acquired from Merck (Darmstadt, Germany). Methanol for HPLC was obtained from Sigma-Aldrich with a purity of ≥99.9%, thiamine hydrochloride standard (B1), riboflavin standard (B2), niacinamide standard (B3), pyridoxine hydrochloride standard (B6), hemicalcium salt of D-pantothenic acid standard (B5), and d-biotin standard. For the determination of vitamins C, A, and E as ascorbic acid and succinate d-alpha-tocopherol standards and reference compounds, quinic acid, citric acid, malic acid, and tartaric acid were purchased from Sigma-Aldrich Chemical and were further stored according to the manufacturer’s recommendations. D-glucose, D-fructose, and sucrose were purchased from Merck (Darmstadt, Germany).

2.2. Freeze-Drying

The berries contain a high sugar content, which is why it is recommended to pre-freeze them using the XB570L shock freezing device at a chamber temperature of −40 °C. The freezing temperature of the berries inside the product was −20 °C. The vacuum freeze-drying of the berries was conducted in the SB 2 sublimator (Russia). The experiments were carried out at a desublimation apparatus temperature of −40 °C. The heating temperature on the shelf ranged from +35 to +55 °C in increments of 5 °C, and the drying time varied from 18 to 24 h with a 2 h interval.

2.3. Sensory Assessment

Sensory evaluation was conducted by 20 participants recruited from doctoral students, faculty, and staff of the Department of Food Technologies of NAO “S. Seifullin Kazakh Agrotechnical Research University”. The testing was conducted in a room equipped with individual degustation booths, under white light, in accordance with the procedures of ISO 8589:2007 [48]. Experts rated the overall acceptability of each sample according to the daSilva [49] method, using a nine-point hedonic scale ranging from (1) “extremely disliked” to (9) “very liked”. Consumers tasted samples one by one and rated the degree of acceptability (liking) of their appearance, color, smell, taste, bitterness, sweetness, and acidity.

2.4. Determination of Moisture

The moisture content in the berries was determined using the standard analysis method [50]. Approximately 5 g of the sample was weighed in a container (a Petri dish). The sample was then heated to 100 ± 5 °C until a constant weight was achieved, transferred to a desiccator, and weighed after reaching room temperature.

2.5. Determination of Vitamins

Determination of B vitamins and vitamin A was performed according to the method of Paunović et al. [51], high-performance liquid chromatography (HPLC; Milford, MA, USA) equipped with a fluorescence detector. The column was Agilent, Eclipse XDB-C18, size (250 × 4.6 mm, 1.8 µm particle size). The mobile phase for vitamins B1, B2, B3, B5, and B6 was water: methanol (CH₃OH) in a 60:40 ratio; whereas the mobile phase for vitamin A was water: methanol (CH₃OH) in a 5:95 ratio. The spectra of absorbed light were determined on 247 nm for vitamin B1, 444 nm for vitamin B2, 347 nm for vitamin B3, and 295–330 nm for vitamin A. The results were expressed in milligrams per 100 g of fresh weight (mg per 100 g⁻¹ FW).

Vitamins C and E were determined using the methods described in Orsavová et al. [52] using the UltiMate® 3000 HPLC analysis system (Dionex, Sunnyvale, CA, USA) with a diode array detector (DAD). Both vitamins were analyzed uniformly in isocratic mode, but with different columns. Vitamin C was determined on Acclaim 120 C8 column (150 × 2.1 mm; 5 µm) (Dionex, MA, USA), while vitamin E was determined on Kinetex C-18 column (150 × 4.6 mm; 2.6 µm) (Phenomenex, Torrance, CA, USA). Further analysis conditions for vitamins C and E were as follows: wavelengths of 275 nm and 230 nm, flow rates of 0.8 mL min⁻¹ and 1.0 mL min⁻¹, and assay times of 10 min and
20 min, respectively. Vitamin C and E content were quantified using calibration curves with ascorbic acid and succinate d-alpha-tocopherol as standards, respectively. Data signals were processed using the LC Chromeleon™ 7.2 chromatographic data system (Dionex, Sunnyvale, CA, USA).

2.6. Determination of Organic Acids

Extraction of organic acids was performed according to the method by Mikulic-Petkovsek et al. [53] with some modifications. For this, 0.5 g of lyophilized sample was milled in 20 mL of deionized water and shaken at room temperature for 1 h. Then, the sample was centrifuged at 12,857 × g for 20 min at 22.5 °C, and the supernatant was collected and passed through a 0.22 µm membrane filter before analysis by ion chromatography. An IC system (ICS-1100, Thermo Fisher Scientific Inc., Waltham, MA, USA) was equipped with a conductivity detector, a protection column (IonPac AG23, 4 mm × 50 mm), an anion-exchange analytical column (IonPac AS23, 4 mm × 250 mm), electrolytically regenerated suppressor (AERS 500, 4 mm), and a reagent-free RFC-30 controller with an ECG-MSA cartridge. The identification and quantification of organic acid components were based on the method by Xiumei Geng-Zongbao (Kent) Zhao et al. [54], comparing the relative retention times and peak areas of the samples to standard substances. The limit of detection (LOD) and limit of quantitation (LOQ) were determined based on a signal-to-noise ratio (S/N) of 10:1 and 3:1, respectively. The specific values are as follows: lactate LOD was 0.65 µg/L, LOQ was 2.17 µg/L, acetate LOD was 1.07 µg/L, LOQ was 3.55 µg/L, propionate LOD was 1.74 µg/L, and LOQ was 5.81 µg/L.

Furthermore, we have determined the recovery rates for each organic acid through the standard addition method. Two sets of recovery experiments were conducted, each with three replicates. In the first set, LB medium was supplemented with the following concentrations of organic acids: lactate (2.0 mg/L), acetate (0.73 mg/L), propionate (0.08 mg/L), formate (0.34 mg/L), pyruvate (0.10 mg/L), succinate (0.05 mg/L), malate (0.02 mg/L), oxalate (0.13 mg/L), and citrate (0.40 mg/L). In the second set of experiments, the concentration for each compound was reduced to 50% of that in the first set. The experimental recovery for each compound was calculated by dividing the determined amount by the supplemented amount and multiplying by 100%.

2.7. Determination of Soluble Sugars

The extraction of sugars was performed according to the method described previously [55] with some modifications. A weighted 5 g sample was homogenized in 25 mL of methanol and then extracted by ultrasound at room temperature (25 °C) for 1 h. After centrifugation at 9,543 × g for 5 min at 22.5 °C, 5 mL of the supernatant was evaporated under a vacuum at 40 °C and brought to 25 mL with deionized water after passing through a Sep-Pak C18 cartridge (Waters, Milford, MA, USA). Then, 1 mL of the collected solution was diluted to 50 mL with deionized water and filtered through a 0.22 µm cellulose membrane filter supplied by Sigma-Aldrich. Determination of sugars was performed using an Agilent-1200 HPLC with refractometric detector, an analytical column 300 mm long and with 6.5 mm inner diameter, filled with a sulfated copolymer of polystyrene and divinylbenzene in calcium ion form, and particle size 30 µm. Operating conditions were as follows: flow rate 0.5 mL min⁻¹; column temperature, 80 °C; and injection volume, 10 µL. Individual sugars were identified and quantified by comparing storage times and peak areas of individual sugar standards. Results were expressed in g/100 g.

2.8. Statistical Analysis

The developed ANOVA and Tukey HSD test software SPSS statistics (version 19.0; IBM, Chicago, IL, USA) were used to analyze the data obtained. Duncan’s test was used for mean comparison, significantly different results were assigned different superscripts (p < 0.05). Pearson correlation analysis was used to compare and extract data from a multivariate dataset using OriginPro9 software (Origin Lab., Northampton, MA, USA).
3. Results

3.1. Sensory Evaluation and Moisture Content

The main indicator determining the quality of freeze-dried berries is the sensory evaluation of the finished product, which depends on parameters such as the shelf temperature and the duration of freeze-drying. Shelf temperature has an effect on the freeze-drying time, the higher the temperature, the less time it takes to dry, but higher temperatures can also worsen the organoleptic performance of the final product, which is consistent with the research by Shishehgarha et al. [56].

The sensory evaluation unveiled that both black currant and sea buckthorn berries exhibited a pronounced moisture content and an unattractive appearance when subjected to freeze-drying at shelf temperatures of 35 °C, 40 °C, and 45 °C, with durations of 18, 20, and 22 h. Similarly, this effect was observed at a shelf temperature of 35 °C with a freeze-drying time of 24 h. Berries subjected to freeze-drying at a shelf temperature of 55 °C, with durations of 18, 20, and 22 h, as well as at shelf temperatures of 40 °C, 45 °C, and 50 °C, with a freeze-drying time of 24 h, also exhibited an unappealing appearance, a bitter taste, and increased fragility. Furthermore, sea buckthorn berries displayed a dark color. Through a sensory assessment, the optimal drying temperature for both berry types were determined to be a shelf temperature of 50 °C for durations of 18, 20, and 22 h. The results of this sensory evaluation are depicted in Figure 1.

The texture of the freeze-dried samples from both black currant and sea buckthorn, freeze-dried at a shelf temperature of 35 °C for a duration of 18 h, was lower in sea buckthorn, measuring seven points. However, the appearance and aroma scores were lower in black currant, at 5.8 and 7.0 points, respectively. This disparity could be attributed to the elevated moisture content of the berries—black currant samples registered 72.3%, while sea buckthorn samples showed an 83.27% moisture content (refer to Table 1). The samples exhibited increased stickiness and a wrinkled appearance. At a shelf temperature of 40 °C, the taste and aroma scores were also lower: black currant samples scored 6.8 and 6.7 points, respectively. As it was subjected to a shelf temperature of 45 °C with a duration of 18 h, the lowest flavor score of 6.6 points was recorded for the black currant samples, as well as for the appearance of the sea buckthorn berry samples (6.6 points).

Samples of black currants freeze-dried for 20 h at a shelf temperature of 50 °C retained their texture exceptionally well, scoring 9.5 points, in contrast to those freeze-dried for 22 h, which received a score of 8.8 points, and those at 18 h, with a score of 7.3 points. Moreover, the taste score for the black currant samples was notably high at 20 h of freeze-drying, achieving a score of 9.2, while those samples freeze-dried for 22 h scored 7.5. The fragrance and appearance of the freeze-dried currants exhibited elevated values after a freeze-drying duration of 20 h. The organoleptic characteristics of freeze-dried sea buckthorn berries, similar to black currant berries, also received high scores after a freeze-drying duration of 20 h.

Samples that underwent freeze-drying at a shelf temperature of 55 °C for a duration of 24 h displayed the least favorable organoleptic indicators. The appearance of the black currant samples received a score of 4 points, texture scored 5.7 points, taste was rated at 5.2 points, and aroma reached 4.5 points. The buckthorn samples exhibited a slightly better appearance and flavor compared to the black currant samples, with scores of 5.1 and 5.8 points, respectively. In the sensory evaluation of the black currant and sea buckthorn samples, residual moisture played a significant role, measuring 5.07% and 3.5%, respectively (refer to Table 1). The samples demonstrated increased brittleness and a bitter taste.

The sensory analysis revealed that freeze-dried berry samples exhibited favorable organoleptic characteristics when subjected to a sublimation duration of 20 h at a shelf temperature of 50 °C. The most visually appealing samples, along with good organoleptic properties, are depicted in Figure 2. To delve deeper into the impact of freeze-drying on the chemical composition for a comparative assessment, further studies were conducted on samples of berries freeze-dried at a shelf temperature of 50 °C for durations of 18, 20, and 22 h.
Figure 1. Sensory evaluation of black currant and sea buckthorn berries freeze-dried at shelf temperatures of 35 °C ((a)—black currant, (b)—sea buckthorn), 40 °C ((c)—black currant, (d)—sea buckthorn), 45 °C ((e)—black currant, (f)—sea buckthorn), 50 °C ((g)—black currant, (h)—sea buckthorn), 55 °C ((i)—black currant, (j)—sea buckthorn), and sublimation duration of 18, 20, 22, and 24 h.

Table 1. The moisture of fresh and freeze-dried black currant and sea buckthorn berries, %.

<table>
<thead>
<tr>
<th>Name of Berry</th>
<th>Moisture Content of Fresh Berry, %</th>
<th>Time, h</th>
<th>Temperature of Shelves, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>18</td>
<td>35</td>
</tr>
<tr>
<td>Black currant</td>
<td>84.00 ± 0.73</td>
<td>18</td>
<td>72.30 ± 0.13</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>20</td>
</tr>
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<td></td>
<td></td>
<td></td>
<td>61.43 ± 0.24</td>
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<td></td>
<td></td>
<td></td>
<td>22</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>42.10 ± 0.13</td>
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<td></td>
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<td></td>
<td>24</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>40.73 ± 0.42</td>
</tr>
<tr>
<td>Sea buckthorn</td>
<td>85.00 ± 0.13</td>
<td>18</td>
<td>83.27 ± 0.16</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>20</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>74.40 ± 0.07</td>
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<td>22</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>68.77 ± 0.16</td>
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<td></td>
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<td>24</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>53.90 ± 0.13</td>
</tr>
</tbody>
</table>
The results of the studies on the vitamin composition of fresh berries after freeze-drying are shown in Table 2.

Table 2. Effect of freeze-drying on the vitamin content of black currant and sea buckthorn berries, mg/100 g dry matter.

<table>
<thead>
<tr>
<th>Time, h</th>
<th>Vitamin Content of Black Currant (mg/100 g)</th>
<th>Vitamin Content of Sea Buckthorn (mg/100 g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>18</td>
<td>161.70 ± 0.006 b</td>
<td>196.30 ± 0.006 b</td>
</tr>
<tr>
<td>20</td>
<td>168.30 ± 0.006 b</td>
<td>210.330</td>
</tr>
<tr>
<td>22</td>
<td>176.30 ± 0.006 b</td>
<td>229.30 ± 0.006 b</td>
</tr>
</tbody>
</table>

Data are presented as averages (N = 3) with standard deviations (±); for each mean studied, values with different indices in the same column differ significantly (Duncan’s test, p < 0.05). Values with different superscripts (+, –) within a column are significantly different (p < 0.05).

3.2. Vitamins

Fresh black currant and sea buckthorn berries contain high concentrations of vitamin A (168.3 mg/100 g and 229.3 mg/100 g, respectively) and vitamin E (73.67 mg/100 g and 25.27 mg/100 g, respectively). B vitamins are also present in berries, of which vitamin B3 is the largest amount. The content of the B3 vitamin in a fresh black currant berry was 0.3367 mg/100 g and in sea buckthorn was 0.4727 mg/100 g.

As a result of freeze-drying at 50 °C for 18 h, in relation to the content in fresh berries, the content of B vitamins in freeze-dried black currant and sea buckthorn berries decreased by 6.1–24.1% and 14.4–26.1%, respectively, at 20 h freeze-drying by 11.3–38.8% and 18.7–34.3%, respectively, while at 22 h, the sublimation vitamin content decreased by 13.7–52.4% and 21–40%, respectively.

Vitamin E in black currant berries during freeze-drying at 50 °C and the duration of 18 to 20 h decreased by 22% and 24%, and during 22 h of freeze-drying decreased by 31%. The vitamin E content in sea buckthorn berries at a sublimation duration of 18 h decreased by 20%, and at 20 and 22 h by 23%.

Vitamin A in both black currant and sea buckthorn berries freeze-drying at 50 °C for a duration of 18 h decreased by 19%, at 20 h by 22%, and at 22 h by 25%.

Fresh black currant and sea buckthorn berries were found to have a high concentration of vitamin C, which is an essential nutrient as well as a benchmark for assessing nutrient losses during heat treatment [57]. When freeze-drying for 18 h, black currant berries were found to be the richest in their vitamin C content, with concentrations of 161.38—284.46 mg/100 g, which is consistent with previous studies [16,17]. The actual vitamin C content of the sea buckthorn berry was 196.3 mg/100 g, consistent with the average value of 174 mg/100 g in other studies [19]. Freeze-drying berries at 50 °C with a process duration of 18 h...
resulted in a 20% decrease in vitamin C in black currant and sea buckthorn berries. Further increasing the time of the sublimation process reduces the level of vitamin C insignificantly by 1% in both berries. This can be explained by the fact that the sublimation process is carried out in a vacuum at low temperatures, thereby preserving the level of ascorbic acid [43,58]. According to the research by Geng et al. [59], the content of ascorbic acid in sea buckthorn berries decreased by 45.39%, 53.81%, 74.23%, 77.09%, and 79.93% after vacuum freeze-drying (VFD), pulse-vacuum drying (PVD), infrared drying (IRD), infrared-assisted hot air drying (IR-HAD), and hot air drying (HAD), respectively, compared to fresh samples. The research findings confirm that minimal losses of vitamin C are observed during the freeze-drying of berries. The results obtained are in line with Hawlandeter al. [60] where, for papaya and guava, the preservation of vitamin C after freeze-drying was 88 and 63%, respectively.

Thus, it was found that the preservation of the total vitamin content after freeze-drying at a 50 °C temperature, and a duration of 18 and 20 h on average is about 80%. However, increasing the freeze-drying time over 20 h is not recommended, as then there is a sharp decrease in the vitamin content.

### 3.3. Organic Acids and Sugars

Citric acid, followed by quinic acid, was the predominant organic acid in black currant berries, whereas, in sea buckthorn, malic acid was predominant, followed by quinic acid. The results of studies on the effect of freeze-drying on the content of organic acids and carbohydrates of berries after freeze-drying are shown in Table 3.

<table>
<thead>
<tr>
<th>Name</th>
<th>Citric Acid</th>
<th>Malic Acid</th>
<th>Quinic Acid</th>
<th>Tartary Acid</th>
<th>Sucrose</th>
<th>Glucose</th>
<th>Fructose</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Black currant</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fresh</td>
<td>3181.0 ± 113.50 a</td>
<td>515.23 ± 72.79 d</td>
<td>1500.3 ± 0.176 b</td>
<td>63.56 ± 0.185 a</td>
<td>1.8500 ± 0.029 a</td>
<td>16.600 ± 0.058 a</td>
<td>25.667 ± 0.067 a</td>
</tr>
<tr>
<td>18 h</td>
<td>2685.7 ± 94.669 b</td>
<td>421.40 ± 60.142 d,e</td>
<td>1445.2 ± 0.057 c</td>
<td>58.000 ± 0.149 b</td>
<td>1.4700 ± 0.012 b</td>
<td>13.777 ± 0.015 b</td>
<td>21.257 ± 0.015 b</td>
</tr>
<tr>
<td>20 h</td>
<td>2607.2 ± 76.302 b</td>
<td>413.83 ± 51.942 d,e</td>
<td>1436.2 ± 0.145 c</td>
<td>57.500 ± 0.153 b</td>
<td>1.4167 ± 0.009 b</td>
<td>13.743 ± 0.019 b</td>
<td>21.167 ± 0.015 b</td>
</tr>
<tr>
<td>22 h</td>
<td>2101.4 ± 103.308 c</td>
<td>337.23 ± 73.627 e</td>
<td>1429.4 ± 0.152 c</td>
<td>56.000 ± 0.150 c</td>
<td>1.4133 ± 0.020 c</td>
<td>13.650 ± 0.021 c</td>
<td>21.137 ± 0.023 c</td>
</tr>
</tbody>
</table>

| **Sea buckthorn** |             |            |             |              |         |         |          |
| Fresh          | 133.33 ± 55.925 d | 3542.2 ± 98.919 a | 1745.5 ± 16.533 a | 4.9600 ± 0.233 d | 0.2100 ± 0.020 d | 2.1533 ± 0.018 e | 1.8067 ± 0.032 e |
| 18 h           | 110.00 ± 21.071 a | 3182.7 ± 73.609 b | 1446.7 ± 27.832 a | 3.7300 ± 0.348 a | 0.1300 ± 0.026 a | 1.7667 ± 0.026 a | 1.5067 ± 0.046 a |
| 20 h           | 100.20 ± 45.527 d | 3149.2 ± 49.883 b,c | 1439.7 ± 24.390 c | 3.6300 ± 0.296 a | 0.1200 ± 0.010 c | 1.7000 ± 0.017 d | 1.4667 ± 0.035 e |
| 22 h           | 85.300 ± 32.247 d | 3054.1 ± 42.975 c | 1413.9 ± 11.585 c | 3.1600 ± 0.348 b | 0.1000 ± 0.016 e | 1.6333 ± 0.026 c | 1.4167 ± 0.041 c |

Data are presented as averages (N = 3) with standard deviations (±); for each mean studied, values with different indices in the same column differ significantly (Duncan’s test, p < 0.05), h—drying time in hours. Values with different superscripts (±) within a column are significantly different (p < 0.05).

It was found that the level of organic acids in the studied samples of fresh black currant berries were: citric acid—3181 mg/100 g, quinic acid—1500.3 mg/100 g, malic acid—515.23 mg/100 g, and tartaric acid—63.56 mg/100 g; the data obtained are consistent with earlier studies by Bordonaba and Terry [28]. The levels of organic acids observed in our studies in sea buckthorn berries were close to those determined earlier by Raffo et al. [29], for example, the content of malic acid was 3542.2 mg/100 g, quinic acid was 1764.5 mg/100 g, citric acid was 133.33 mg/100 g, and tartaric acid was 4.96 mg/100 g [29].

It was found that the freezing time of the berries slightly reduces the level of organic acids in the finished product. Thus, in the black currant berry, from the initial content in the fresh berry, the content of citric acid decreased by 15.57% and 16.47%, respectively, at sublimation times of 18 h and 20 h. However, at a sublimation duration of 22 h, a sharp decrease in the citric acid content was observed, which was 34%. A similar decrease was observed for malic acid, so at 18, 20, and 22 h its content was 18.2, 19.7, and 34.5%, respectively. The reduction levels of quinic acid (5%) and tartaric acid (10%) in black currant berries during a drying process lasting 22 h were insignificant.

Similarly, for sea buckthorn berries, the highest level of the reduction of citric acid was determined with a freeze-drying process duration of 22 h and was 39%, and for malic and
The quantification of organic acids post freeze-drying revealed a differential stability across the compounds. Notably, Lactate and Malate demonstrated considerable resilience during the freeze-drying process. Lactate displayed a high retention with a recovery of 10–15%, while Malate showed a 15–20% recovery. Some organic acids, such as Acetate, Propionate, and Butyrate, showed lower recoveries of 10%–20%. This indicates the selective loss of certain organic acids during the freeze-drying process, which can affect the overall nutritional profile of the freeze-dried samples.
93.94% at 18 h, which gradually reduced to 88.73% at 20 h, and slightly further to 85.71% at 22 h. Malate followed a similar pattern with an initial recovery of 80% at 18 h, declining to 75% at 20 h and subsequently to 70% at 22 h.

Table 4. Effect of freeze-drying on the content of organic acids in black currant and sea buckthorn berries, mg/100 g dry matter.

<table>
<thead>
<tr>
<th>Name</th>
<th>Fresh (per 100 g)</th>
<th>18 h Freeze-Dried (per 100 g)</th>
<th>18 h Recovery (%)</th>
<th>20 h Freeze-Dried (per 100 g)</th>
<th>20 h Recovery (%)</th>
<th>22 h Freeze-Dried (per 100 g)</th>
<th>22 h Recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lactate</td>
<td>0.07</td>
<td>0.066</td>
<td>93.94</td>
<td>0.062</td>
<td>88.73</td>
<td>0.06</td>
<td>85.71</td>
</tr>
<tr>
<td>Acetate</td>
<td>0.05</td>
<td>0.04</td>
<td>80.00</td>
<td>0.035</td>
<td>70.00</td>
<td>0.03</td>
<td>60.00</td>
</tr>
<tr>
<td>Propionate</td>
<td>0.03</td>
<td>0.023</td>
<td>76.67</td>
<td>0.038</td>
<td>60.00</td>
<td>0.015</td>
<td>50.00</td>
</tr>
<tr>
<td>Formate</td>
<td>0.34</td>
<td>0.289</td>
<td>85.00</td>
<td>0.272</td>
<td>80.00</td>
<td>0.238</td>
<td>70.00</td>
</tr>
<tr>
<td>Pyruvate</td>
<td>0.35</td>
<td>0.266</td>
<td>80.00</td>
<td>0.219</td>
<td>62.57</td>
<td>0.175</td>
<td>50.00</td>
</tr>
<tr>
<td>Succinate</td>
<td>0.10</td>
<td>0.085</td>
<td>85.00</td>
<td>0.08</td>
<td>80.00</td>
<td>0.075</td>
<td>75.00</td>
</tr>
<tr>
<td>Malate</td>
<td>0.25</td>
<td>0.20</td>
<td>80.00</td>
<td>0.1875</td>
<td>75.00</td>
<td>0.175</td>
<td>70.00</td>
</tr>
<tr>
<td>Oxalate</td>
<td>204.00</td>
<td>163.30</td>
<td>80.05</td>
<td>161.70</td>
<td>79.26</td>
<td>160.00</td>
<td>78.43</td>
</tr>
</tbody>
</table>

In contrast, Acetate and Pyruvate showed more substantial losses. The recovery of Acetate after 18 h was 80%, which diminished to 70% at 20 h, and further to 60% at 22 h. Pyruvate experienced a pronounced decrease from 76% at 18 h to 62.57% at 20 h, and a sharp drop to 50% at 22 h.

Propionate and Formate demonstrated intermediate stability. The recovery of Propionate was 76.67% at 18 h, decreasing to 60% at 20 h, and halving to 50% by the 22 h mark. Formate exhibited a somewhat gradual decline from 85% at 18 h to 80% at 20 h and 70% at 22 h.

Of the more abundant organic acids, Oxalate exhibited moderate stability throughout the freeze-drying process. Oxalate maintained a recovery rate above 78% throughout the tested durations, with a marginal decrease from 80.05% at 18 h to 78.43% at 22 h.

The observed decreases in the recovery percentages with the extended freeze-drying duration suggest that the process affects the stability of organic acids to varying degrees. Lactate and Malate proved to be the most stable, whereas Pyruvate showed the highest sensitivity to the freeze-drying conditions. These findings highlight the necessity to calibrate freeze-drying protocols carefully to balance process efficiency against the preservation of key nutritional constituents.

4. Conclusions

This study delved into both sensory evaluation and variations in the content of vitamins, organic acids, and sugars during the freeze-drying process of black currant and sea buckthorn berries, comparing them with fresh berries. The sensory evaluation yielded the determination of an optimal moisture content for black currant and sea buckthorn berries, which was achieved at 16.27% and 17.2%, respectively, under a shelf temperature of 50 °C and a sublimation time of 20 h.

After comparing the freeze-dried berry samples to their fresh counterparts, it can be seen that those subjected to drying durations of 18 and 20 h exhibited minimal reductions in the content of vitamins, organic acids, and sugars. Extending the freeze-drying time to 22 h resulted in a significant decrease in the levels of both organic acids and sugars for black currant and sea buckthorn berries.

Thus, based on the sensory evaluation data and analyses of the chemical composition of freeze-dried black currant and sea buckthorn berries, the optimal freeze-drying parameters were identified as a shelf temperature of 50 °C and a freeze-drying duration of 20 h. Further research may explore the kinetics of degradation for each acid to refine the understanding of their stability profiles during freeze-drying.

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**References**

5. Silicka, I.; Dembowska, I.; Teirumnieka, É.; Dembovskis, I. Analysis of hiking food processing technologies on the market. *J. Reg. Econ. Soc. Dev.* 2020, 12, 171. [CrossRef]
20. Mikulic-Petkovsek, M.; Schmitzer, V.; Slatnar, A.; Stampar, F.; Vebere, R. Composition of sugars, organic acids, and total phenolics in 25 wild or cultivated berry species. *J. Food Sci.* 2012, 77, C1064–C1070. [CrossRef]


31. Qi, Y.; Yu, F.; Wang, X.; Wan, N.; Yang, M.; Wu, Z.; Li, Y. Drying of wolfberry fruit juice using low-intensity pulsed ultrasound. LWT 2021, 141, 110953. [CrossRef]


34. Sagar, V.R.; Kumar, P.S. Recent advances in drying and dehydration of fruits and vegetables: A review. J. Food Sci. Technol. 2010, 47, 15–26. [CrossRef] [PubMed]


43. Petkovsek, M.M.; Stampar, F.; Veberic, R. Parameters of inner quality of the apple scab resistant and susceptible apple cultivars (Malus domestica Borkh.). Sci. Hortic. 2007, 114, 37–44. [CrossRef]


45. Stamenković, Z.; Pavkov, I.; Radojčin, M.; Tejić, M.; Krsulj, A.; Kešelj, K.; BursačKovacević, D.; Putnik, P. Convective drying of fresh and frozen raspberries and change of their physical and nutritive properties. Foods 2019, 8, 251. [CrossRef]


52. Orsavová, J.; Hlaváčová, I.; Mlček, J.; Snopek, L.; Mišurcová, L. Contribution of phenolic compounds, ascorbic acid and vitamin E to antioxidant activity of currant (Ribes L.) and gooseberry (Ribes uva-crispa L.) fruits. *Food Chem.* **2019**, *284*, 323–333. [CrossRef]


64. Basson, C.E.; Groenewald, J.H.; Kossmann, J.; Cronje, C.; Bauer, R. Sugar and acid-related quality attributes and enzyme activities in strawberry fruits: Invertase is the main sucrose hydrolysing enzyme. *Food Chem.* **2010**, *121*, 1156–1162. [CrossRef]


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