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The effect of refrigeration and room temperature storage conditions on the physico-chemical characteristics of hybrid and freeze-dried blueberries

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A R T I C L E I N F O

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A B S T R A C T

In order to preserve the nutritional properties of fruits after dehydration, it is necessary to use appropriate storage methods. The traditional freeze-dried and combination-dried i. e. vacuum pre- and freeze post-dried, freeze pre- and vacuum post-dried, mid-infrared-freeze-dried, and freeze-mid-infrared dried blueberry samples were stored in vacuum packs for 6 months. The reference drying method, i.e. freeze drying at a pressure of 80–90 Pa and a temperature range between −25 and 20 °C, with a drying time of 22 h, was used. The blueberries were mid-infrared dried at 60 °C for 5 min before and after freeze-drying, and the experimental materials were also vacuum dried at 60 °C for 4 h at 7 kPa vacuum pressure before and after freeze-drying. Drying operation times were ranked from the shortest to the longest as follows: freeze-mid-infrared drying, mid-infrared freeze-drying, freeze pre- and vacuum post-drying, and vacuum pre- and freeze post-drying and conventional lyophilization. The aim of this study was to investigate the effects of different storage conditions, namely refrigeration (5 °C in a refrigerator) and room temperature (23 °C on a shelf in a dark place) on the moisture content, water activity, firmness, rehydration ratio, polyphenol- and flavonoid content, as well as antioxidant capacity of dried blueberries. The dried and vacuum-packaged blueberries stored in the refrigerator and at room temperature can be characterized by the stability of their physical and chemical properties throughout the storage period. It was found that a traditional freeze-drying (FD), freeze-drying and vacuum-drying combination of 4 h at 60 °C (FD-VD4h60 °C) together with vacuum packaging at ambient temperature, is sufficient to ensure a shelf-stable whole blueberry product.

1. Introduction

Blueberries are very popular among consumers, as research has linked their consumption to improved human health [1]. Blueberries (*Vaccinium myrtillus* L.) are often referred to as a “super fruit” and command a high purchase price because they are a rich source of bioactive compounds such as anthocyanins, polyphenols and other flavonoids [2]. Disease prevention effects are attributed to the various antioxidants found in blueberries. Besides vitamin C, phenolic compounds, especially anthocyanins, contribute significantly to the total antioxidant activity [3]. Berries are marketed as fresh or frozen whole fruits, lyophilized berries, puree, juice, pulp and red wine [4].

After blueberries are harvested, the bioactive compounds are prone to oxidative reactions that negatively affect the berry’s phenolic levels and antioxidant capacity. Physical damage occurs due to loss of hardness and microbial decomposition [5].

Various drying techniques are used to prevent the fruit spoilage and extend shelf life. Freeze-drying is one of the best methods for removing water from biological products compared to other dehydration techniques [6]. Freeze-drying (FD) produces the highest quality food product, but it is the most expensive process for producing dehydrated products due to high energy consumption [7].

Therefore, combining a traditional drying technique such as vacuum drying and infrared drying with freeze-drying can reduce operational costs while maintaining the nutritional and quality characteristics of the dehydrated food [8,9].

The results of Nsonzi and Ramaswamy [10] showed that osmotic dehydration minimized color loss during convective air drying. The osmo-convective dried blueberries were not harder than conventional air-dried blueberries. The rehydration rates of osmo-convective dried blueberries were lower than the rehydration rates of freeze-dried and air-dried samples. Blueberries dried under the best osmotic dehydration

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conditions had better color and texture and shorter drying time than the conventional air-dried blueberries.

Freeze-dried and hot-air-microwave-vacuum combination-dried blueberries had a higher retention of total polyphenols and anthocyanins than hot-air-treated blueberries. Freeze-dried blueberries had the highest antioxidant activity, followed by the combination of hot-air and microwave-vacuum, microwave-vacuum and hot-air drying methods [11].

Reyes et al. [12] investigated freeze-drying and infrared drying of blueberries. They found that freeze-drying had a negative effect on ascorbic acid and total polyphenol content, whereas the application of infrared radiation had a positive effect on them.

Zielinska et al. [13] investigated the effect of hot air drying, microwave vacuum drying and the combination thereof on the total amount of polyphenols and anthocyanins as well as the antioxidant capacity of blueberries. The highest anthocyanin content and antioxidant capacity were found in blueberries dried by hot air at 90 °C via microwave vacuum drying.

Liu et al. [14] performed a comparative study of far-infrared radiation heating assisted pulsed vacuum drying and hot-air drying method on blueberry. His method maintained lower color change, better mechanical properties and rehydration capacity, and higher antioxidant activity by shortening operational time, reducing exposure to oxygen, and modifying the microstructure compared to the hot air drying at the same drying temperature.

Wang et al. [15] found that there was no significant difference between microwave freeze-dried and conventional freeze-dried products in terms of color, total monomeric anthocyanins and total phenolic content.

However, while the effects of different drying techniques on the physical and chemical properties of blueberry have been reported, only a few papers have discussed the changes in texture, rehydration, polyphenols- and flavonoids content, and antioxidant capacities during storage of dried blueberries.

Fracassetti et al. [16] investigated the total anthocyanins content and total antioxidant activity of freeze-dried wild blueberry powder samples stored at different temperatures (25 °C, 42 °C, 60 °C and 80 °C) for 49 days.

Calabuig-Jiménez et al. [17] performed hot-air dried and powdered blueberry pomace. The physicochemical properties of the powdered samples were investigated for 20 weeks.

Value addition by drying has proven to be a good option to raise the market potential and availability of many fruits [18]. With proper storage, dried products can be stored without deterioration and the aim is to maintain their market value. The development of modern packaging methods (modified atmosphere packaging and vacuum packaging) can enhance the shelf life of fresh foods and prevent chemical spoilage [19].

In consequence, the goal of this scientific work is to assess the influence of dried blueberry storage conditions on moisture content, water activity, texture, rehydration, bioactive compounds and polyphenol stability.

2. Material and methods

2.1. Preparation of the raw material

The blueberries (the cultivars 'Bluegold') were products of Hungary and were purchased in a local supermarket. Samples were selected from their visual appearance and size. The full ripe berries are an ash blue color. The average diameter of a blueberry was 1.1 cm ± 0,1 cm. The blueberry samples were washed under running tap water and were stored at 5 °C and 70 % relative humidity in a refrigerator (model Lehel HB 160, Lehel Ltd., Jászberény, Hungary) for 1 h before drying tests.

Freeze-drying of blueberries is difficult because the outer layer of the skin of the blueberry is waxy, which prevents moisture moving across the fruit. Therefore, it is necessary to pretreat the raw material [13]. Before freeze-drying and freeze pre-drying the samples were pre-treated with quick-freezing in a laboratory quick freezer (model FT34MKII, Armfield Ltd., Ringwood, UK). Freezing time was 10 min, and freezing temperature was −25 °C. The average initial moisture content of blueberry samples was 87.33 % (wet basis, w. b.) and 6.892 (g water) (g dry matter)$^{-1}$ (dry basis, d.b.) determined by the gravimetric method [20]. For each drying program, 100 g of blueberries were placed in a single layer on a tray.

2.2. Drying methods

Drying experiments were performed by combining three drying methods, i.e. freeze-drying, vacuum drying and infrared drying.

Freeze-drying was performed using an Armfield FT-33 dryer (Armfield Ltd, Ringwood, UK). The temperature of the condenser was set to −48 °C and the pressure of the chamber was 80–90 Pa. The internal temperature of whole blueberries was recorded using T-type thermocouple probes inserted in the center of 4 samples. Sample weight loss was recorded every 1 min during the drying process using a digital balance with precision of ±0.1 g (model PAB-01, Emalog Ltd., Budapest, Hungary). The product reached a final temperature of about 20 °C at the end of the drying process. The drying time to reduce moisture content to equilibrium was 22 h.

A laboratory vacuum oven (model Kambic VS-50C, Kambič Lab. Eq., Semič, Slovenia) combined with a vacuum pump (model Büchi V-501, Büchi AG, Flawil, Switzerland) was used for the vacuum drying. Blueberries were dried at 60 °C for 4 h at 7 kPa (70 mbar) vacuum pressure before and after lyophilization (VD4h-FD and FD-VD4h). T-type thermocouple probes were used to monitor the shelf and product temperature during the drying process. Weight loss of blueberry samples was recorded using a balance (model JKH-500, Jadever Co., New Taipei, Taiwan), with a sensitivity of ±0.1 g. The materials were taken out of the drying apparatus, weighed and then reinserted into the cabinet. The samples were weighed every hour until the equilibrium was reached at FD-VD4h (constant weight).

The infrared drying: A laboratory scale infrared dryer was used for infrared dehydration of the samples (model Precisa HA60, Precisa Instruments AG, Dietikon, Switzerland). The blueberries were dried using mid-infrared radiation (wavelength: 2–3 μm). An infrared heater was installed on the top of the drying chamber. Power output of the infrared heater was 410 W. The average infrared intensity was 4500 W m^{-2}, corresponding to a drying temperature of 60 °C. The temperature was measured by placing T-type thermocouples under the blueberry skin. A digital balance (model Precisa HA60) with the accuracy of ±0.01 g was used for mass determination. The samples were weighed every minute until the equilibrium was reached at FD-MIR5min (constant weight). Blueberries were infrared dried at 60 °C for 5 min before and after lyophilization (MIR5min-FD and FD-MIR5min).

The drying program used in the dehydration experiments is given in Table 1. Table 1 shows, in addition to the drying temperature, the pre- and post-drying times and the total drying time.

The dried samples were packaged under vacuum with an 85 μm thick polyamide/polyethylene (outer/inner) film (model Laica, VT3112, Laica, Barbarano, Italy) immediately after the dehydration procedure (as dried berries are highly hygroscopic).

2.3. Storage conditions

To determine the storage stability in terms of water activity, moisture content, texture, rehydration changes and losses in chemical components, dried whole blueberries were stored under different conditions. Samples were stored in the dark (room temperature) at 23 °C ± 1 °C, 50–55 % RH and a domestic refrigerator (model Lehel HB 160, Lehel Ltd., Jászberény, Hungary) at 5 °C, 70–75 % RH. Packaged samples were stored for 6 months in both storage locations and sampled monthly for

Table 1
The drying parameters of whole blueberries with different drying methods.

Drying methods	FD	Hybrid drying methods			
		VD4h-FD	FD-VD4h	MIR5min-FD	FD-MIR5min
Drying temperature at FD (°C)	−25–20	–	–	–	–
Pre-drying temperature at hybrid drying (°C)	–	60	−25–20	60	−25–20
Post-drying temperature at hybrid drying (°C)	–	−25–20	60	−25–20	60
Pre-drying time at hybrid drying (min)	–	240	600	5	660
Post-drying time at hybrid drying (min)	–	660	240	780	5
Total drying time (min)	1320^{e}	900^{d}	840^{bc}	785^{b}	665^{a}

FD: freeze-drying, VD-FD: vacuum pre- and freeze post-drying, FD-VD: freeze pre- and vacuum post-drying, MIR-FD: mid-infrared-freeze-drying, FD-MIR: freeze-drying and mid-infrared drying. Data are expressed as the average for three replicate. Values in the same column with different superscripts (a,b,c,d,e) are significantly different ($p < 0.05$).

analysis.

2.4. Water activity (a_w) and moisture content (MC)

The water activity (a_w) of dried blueberry (3 g) was determined using a water activity meter (model Novasina Labmaster CH-8853, Novasina AG, Lachen, Switzerland) at 25 °C ± 1 °C. The initial and equilibrium moisture content (MC) of the samples was measured by using the gravimetric method in an oven dryer (LP-302, Kapacitív Ltd., Budapest, Hungary). The samples were dried at 105 °C until a constant weight was obtained. The initial and equilibrium moisture content of the materials was given as g of water per g of dry solids.

2.5. Texture and rehydration

The texture of dehydrated products was assessed by a Brookfield CT-3 texture analyzer (Brookfield Engineering Laboratories Inc., Middleboro, USA). Texture profile analysis were evaluated by a compression test. The applied test parameters were: 4.5 kg force load cell, 2 mm s^{-1} test speed, 20 mm travel distance and 4 mm diameter of cylindrical probe. The maximum depth of penetration was 3 mm and trigger force was 10 g. The penetrometer measurements are reported in Newtons (N).

The rehydration experiment was performed at room temperature. Weighed dried blueberry samples were immersed in distilled water. The rehydration was carried out in 100 mL distilled water at temperature of 20 °C for 30 min. The sample was then removed from the water, spread on paper towels and wiped to remove excess surface water and then reweighed. The rehydration ratio (RR) was calculated using the following equation (1):

$$RR = \frac{W_r}{W_d}, \tag{1}$$

where W_r and W_d were the weights of the rehydrated and the dried samples (in g), respectively.

2.6. Sample preparation for chemical analysis

The dried berries were ground into powder using a laboratory grinder (model QC-124, Kapacitív Ltd, Budapest, Hungary) and 200 mg aliquots were extracted in 20 mL methanol (99.9 %). Then the mixture was incubated in an oven (LP-302, Kapacitív Ltd., Budapest, Hungary) at 30 °C under constant agitation (200 rpm) for 15 min. The solid part was then separated by filtration and the extract was immediately analyzed to determine antioxidant capacity, total phenolic concentration and total flavonoid concentration.

2.7. Determination of total phenolic compounds (TPC)

Phenolic compounds were analyzed using the Folin-Ciocalteu reagent according to the method adopted by Slinkard and Singleton [21]. The absorbance was read at 765 nm using a UV–vis spectrophotometer (model PerkinElmer Lambda 35, PerkinElmer, Waltham, USA) and the results were expressed as mg gallic acid equivalents (GAE) per 100 g of sample dry matter (mg GAE/100 g d m.). All chemicals were analytical grade and were purchased form Sigma-Aldrich Corp. (USA).

2.8. Determination of total flavonoid concentration (TFC)

TFC was measured according to Vuthijumnok et al. [22]. The absorbance was measured at 510 nm using a UV–vis spectrophotometer (model PerkinElmer Lambda 35, PerkinElmer, Waltham, USA). The results were expressed as mg catechin equivalents (CE) per g of sample dry matter (mg CE/g d.m.).

2.9. Determination of antioxidant capacity (AC) by ABTS methodology

The ABTS method (2,2′-azino-bis(3-ethylbenzthiazoline-6-sulphonic acid) is based on the deactivation of the antioxidant radical cation ABTS, which is determined by the reduction in absorbance at 734 nm using a UV–vis spectrophotometer (model PerkinElmer Lambda 35, PerkinElmer, Waltham, USA). The ABTS method was performed as described by Arnao et al. [23] and Apak et al [24]. The results were expressed in μmol as trolox equivalent (TE) per g dry matter (μmol TE/g d.m.).

2.10. Statistical analysis

Experiments were replicated twice (n = 3). Results presented are mean values of each determination ± standard deviation (SD). All data collected were analyzed using one way analysis of variance (ANOVA) and Tukey's test to determine significant differences ($p < 0.05$) among the means. All statistical tests were carried out using the SPSS for Windows Version 22.0 package by SPSS Inc., Chicago, USA.

3. Results and discussion

3.1. Changes of the water activity and moisture content in dried blueberries during storage

In order to better quantify the effect of different storage conditions and duration on the water activity and moisture content of dried blueberries, the percentage deviation in the studied physical characteristics was calculated relative to the baseline data measured immediately after drying was calculated using the following Eq. (2):

$$\text{Percentage deviation } (\%) = \frac{\text{condition measured in a given storage month}}{\text{condition immediately after drying}} \tag{2}$$

The retention of water activity (a_w) and moisture content (MC) in dried blueberries was high with the examined storage methods and period (Table 2).

The moisture content and water activity of dried blueberries by different drying methods were 0.05–0.12 g water (g d.m.$^{-1}$) and 0.11–0.19, respectively. Previous research has reported that the moisture content and water activity of freeze-dried Andean blueberry

Table 2
Variation of moisture content and water activity of dried blueberries as a function of storage time.

Storage conditions	Drying methods	Storage time (month)													
		0/dried		1		2		3		4		5		6	
		MC (g/g, d. m.)	a_w (-)	MC (%)[a]	a_w (%)[a]	MC (%)	a_w (%)	MC (%)	a_w (%)	MC (%)	a_w (%)	MC (%)	a_w (%)	MC (%)	a_w (%)
Room temperature	FD	0.12 ± 0.02	0.19 ± 0.12	100.0^{a}	99.6^{a}	99.5^{a}	99.4^{a}	99.4^{a}	99.2^{a}	99.1^{a}	99.3^{a}	99.2^{a}	99.3^{a}	98.9^{a}	99.2^{a}
	VD4h-FD	0.10 ± 0.02	0.16 ± 0.11	99.4^{a}	99.5^{a}	98.0^{a}	99.5^{a}	98.0^{a}	99.3^{a}	98.1^{a}	99.4^{a}	98.1^{a}	98.9^{a}	98.0^{a}	98.4^{a}
	FD-VD4h	0.08 ± 0.01	0.13 ± 0.09	99.7^{a}	98.9^{a}	99.6^{a}	98.9^{a}	99.6^{a}	99.1^{a}	99.3^{a}	98.8^{a}	99.5^{a}	99.0^{a}	99.3^{a}	99.1^{a}
	MIR5min-FD	0.05 ± 0.01	0.11 ± 0.07	99.8^{a}	99.3^{a}	99.7^{a}	99.4^{a}	99.3^{a}	98.8^{a}	98.8^{a}	98.4^{a}	98.4^{a}	98.3^{a}	97.9^{a}	97.1^{ab}
	FD-MIR5min	0.06 ± 0.01	0.12 ± 0.08	99.5^{a}	99.4^{a}	99.4^{a}	98.8^{a}	98.5^{a}	98.4^{a}	98.5^{a}	98.5^{a}	98.4^{a}	98.5^{a}	98.3^{a}	98.3^{a}
Refrigerator	FD	0.12 ± 0.02	0.19 ± 0.12	99.9^{a}	100.0^{a}	99.6^{a}	99.8^{a}	99.7^{a}	99.8^{a}	99.5^{a}	99.6^{a}	99.4^{a}	99.3^{a}	99.1^{a}	99.0^{a}
	VD4h-FD	0.10 ± 0.02	0.16 ± 0.11	99.6^{a}	98.9^{a}	99.4^{a}	99.0^{a}	99.1^{a}	98.6^{a}	98.7^{a}	98.4^{a}	98.0^{a}	98.4^{a}	97.8^{a}	98.3^{a}
	FD-VD4h	0.08 ± 0.01	0.13 ± 0.09	99.9^{a}	99.8^{a}	99.8^{a}	99.6^{a}	99.8^{a}	99.7^{a}	99.7^{a}	99.6^{a}	99.5^{a}	99.6^{a}	99.5^{a}	99.5^{a}
	MIR5min-FD	0.05 ± 0.01	0.11 ± 0.07	99.9^{a}	99.4^{a}	99.7^{a}	99.4^{a}	99.1^{a}	98.9^{a}	98.7^{a}	98.3^{a}	98.1^{a}	98.2^{a}	97.5^{ab}	97.7^{a}
	FD-MIR5min	0.06 ± 0.01	0.12 ± 0.08	99.9^{a}	99.5^{a}	99.5^{a}	99.2^{a}	99.3^{a}	99.0^{a}	99.2^{a}	98.6^{a}	98.9^{a}	98.4^{a}	98.5^{a}	98.3^{a}

FD: freeze-drying, VD-FD: vacuum pre- and freeze post-drying, FD-VD: freeze pre- and vacuum post-drying, MIR-FD: mid-infrared-freeze-drying, FD-MIR: freeze-drying and mid-infrared drying. Different lower case letters within each row indicate significantly different means among dried samples according to the Tukey test at $p < 0.05$. SD: standard error of mean.

[a] Percentage deviation = value of the condition measured in a given storage month divided by value of the condition immediately after dehydration.

powders ranged from 4.0 % to 9.0 % (in w. b.) and 0.2 to 0.5, respectively [25]. Darniadi et al. [26] found that the water activity value of foam-mat freeze-dried blueberry powder was less than 0.40.

There were no significant differences ($p > 0.05$) in water activity and moisture content between blueberry samples dried by different methods during the investigated period (months 1–6). The same statement is true for the storage methods, i.e. no large differences can be observed for the samples stored at room temperature and in the refrigerator during the six-month storage period. Calabuig-Jiménez et al. [17] found a slight increase in water activity and moisture content during 20 weeks of storage of powdered blueberry pomace in opaque jars. The water activity remained below 0.3.

In the case of blueberries dried using the MIR5min-FD method, it can be observed that the water activity and moisture content decreased continuously during the examined period, for both storage methods. A similar trend is observed for VD4h-FD and FD-MIR5min blueberries when stored in a refrigerator. In contrast, the water activity and moisture content of blueberries dried with the FD-MIR5min process slightly and gradually decreased during storage at room temperature from month 0 to month 3 ($p > 0.05$). The best results, in terms of water activity and moisture content retention, were obtained with FD-VD4h and freeze-dried (FD) blueberries in both storage conditions.

3.2. Changes of the texture and rehydration ratio in dried blueberries during storage

In order to better quantify the effect of different storage conditions and duration on the texture and rehydration of dried blueberries, the percentage deviation in the studied physical characteristics was calculated relative to the baseline data measured immediately after drying (Equation (2)). During the storage period, the dried blueberry showed negligible deterioration in texture and rehydration index (RR) (Table 3).

The hardness of dried blueberries (month 0.) for FD, VD4h-FD, FD-VD4h, MIR5min-FD and FD-MIR5min were 8.44, 8.23, 8.62, 9.07 and 9.58 N, respectively. The rehydration ratio (RR) of dried blueberries (month 0.) for FD, VD4h-FD, FD-VD4h, MIR5min-FD and FD-MIR5min were 3.57, 3.09, 3.25, 2.31 and 2.48, respectively. Yang and Atallah [27] found that freeze-dried blueberries had a higher rehydration ratio than vacuum-dried berries.

Based on the results of the storage test, it can be concluded that there were no significant differences ($p > 0.05$) in the texture and rehydration rate values of blueberries dried by various methods during the 6-month study. Similar to the previous finding, no significant differences in the hardness and RR value of the samples stored at room temperature and in the refrigerator can be detected during the investigated storage period. Skoczeń-Słupska et al. [28] observed that the storage temperature (chilled storage: 2 °C and room storage: 20 °C) had no effect on the rehydration of lyophilized and hot air-dried bilberry products. The blueberries dehydrated by FD, VD4h-FD and FD-VD4h procedures remained stable with respect to the studied parameters both at room temperature and during the storage period in the refrigerator. In other words, these three drying procedures are the best at preserving firmness and rehydration value. The texture and rehydration rate values of FD-VD4h blueberries decrease from month to month in both storage modes, but still remain below significant difference ($p > 0.05$). Regarding the physical parameters of MIR5min-FD and FD-MIR5min blueberries, it can be observed that they drastically decreased during the investigated period when they were stored at room temperature and in the refrigerator. The largest difference in the examined physical properties was observed for FD-MIR5min solution dried blueberries for both storage methods ($p > 0.05$).

3.3. Changes of the total phenolic content, total flavonoid content and antioxidant capacity in dried blueberries during storage

As can be observed in Fig. 1, the total phenolic content (TPC) values of dried blueberries stored at room temperature and in a refrigerator showed a slight decrease during the storage period. Similarly to our results, Calabuig-Jiménez et al. [17] found that the total phenolic content of hot air-dried and powdered blueberry pomace did not change significantly, remained stable over the 20 weeks storage period. The initial values (0) of blueberries preserved by different drying methods

Table 3
Variation of texture and rehydration ratio of dried blueberries as a function of storage time.

Storage conditions	Drying methods	Storage time (month)													
		0/dried		1		2		3		4		5		6	
		Texture (N)	RR (-)	Texture (%)[a]	RR (%)[a]	Texture (%)	RR (%)	Texture (%)	RR (%)	Texture (%)	RR (%)	Texture (%)	RR (%)	Texture (%)	RR (%)
Room temperature	FD	8.44 ± 0.37	3.57 ± 0.16	99.7^{a}	100.0^{a}	99.6^{a}	99.7^{a}	99.6^{a}	99.6^{a}	99.5^{a}	99.6^{a}	99.4^{a}	99.5^{a}	99.4^{a}	99.5^{a}
	VD4h-FD	8.23 ± 0.25	3.09 ± 0.18	99.8^{a}	99.4^{a}	99.7^{a}	99.3^{a}	99.5^{a}	99.3^{a}	99.2^{a}	99.3^{a}	99.0^{a}	99.2^{a}	98.8^{a}	99.1^{a}
	FD-VD4h	8.62 ± 0.42	3.25 ± 0.21	99.5^{a}	99.9^{a}	99.2^{a}	99.7^{a}	99.1^{a}	99.4^{a}	98.7^{a}	99.3^{a}	98.6^{a}	99.1^{a}	98.6^{a}	98.9^{a}
	MIR5min-FD	9.07 ± 0.49	2.31 ± 0.09	99.7^{a}	99.4^{a}	99.1^{a}	99.0^{a}	98.5^{a}	98.5^{a}	97.9^{a}	98.2^{a}	97.7^{a}	98.0^{a}	97.6^{a}	98.1^{a}
	FD-MIR5min	9.58 ± 0.54	2.48 ± 0.12	99.2^{a}	99.6^{a}	98.7^{a}	98.8^{a}	98.1^{a}	97.9^{a}	97.3^{ab}	97.4^{a}	96.9^{ab}	97.1^{ab}	96.6^{ab}	97.0^{ab}
Refrigerator	FD	8.44 ± 0.37	3.57 ± 0.16	99.6^{a}	99.9^{a}	99.6^{a}	99.7^{a}	99.5^{a}	99.5^{a}	99.5^{a}	99.6^{a}	99.4^{a}	99.5^{a}	99.3^{a}	99.4^{a}
	VD4h-FD	8.23 ± 0.25	3.09 ± 0.18	99.6^{a}	99.6^{a}	99.4^{a}	99.4^{a}	99.3^{a}	99.4^{a}	98.7^{a}	99.2^{a}	98.6^{a}	98.9^{a}	98.6^{a}	98.7^{a}
	FD-VD4h	8.62 ± 0.42	3.25 ± 0.21	99.4^{a}	99.1^{a}	99.1^{a}	99.0^{a}	98.8^{a}	98.8^{a}	98.7^{a}	98.3^{a}	98.5^{a}	98.4^{a}	98.1^{a}	98.0^{a}
	MIR5min-FD	9.07 ± 0.49	2.31 ± 0.09	99.8^{a}	99.1^{a}	99.4^{a}	99.0^{a}	98.6^{a}	98.5^{a}	98.0^{a}	98.2^{a}	97.8^{a}	98.0^{a}	97.8^{a}	97.7^{a}
	FD-MIR5min	9.58 ± 0.54	2.48 ± 0.12	99.3^{a}	99.4^{a}	98.6^{a}	99.1^{a}	98.4^{a}	98.2^{a}	97.7^{a}	97.9^{a}	97.0^{ab}	97.4^{a}	96.4^{ab}	97.1^{ab}

FD: freeze-drying, VD-FD: vacuum pre- and freeze post-drying, FD-VD: freeze pre- and vacuum post-drying, MIR-FD: mid-infrared-freeze-drying, FD-MIR: freeze-drying and mid-infrared drying. Different lower case letters within each row indicate significantly different means among dried samples according to the Tukey test at $p < 0.05$. SD: standard error of mean.

[a] Percentage deviation = value of the condition measured in a given storage month divided by value of the condition immediately after dehydration.

correspond to the parameters of the dried material. The FD, VD4h-FD, FD-VD4h, MIR5min-FD and FD-MIR5min dried blueberries had 451, 408, 422, 398 and 359 mg GAE 100 g^{-1} total phenolic content on dry weight basis, respectively. The highest TPC concentration (451 mg GAE 100 g^{-1} d m) was obtained from FD samples. Rodriguez-Mateos et al. [29] identified a total polyphenols content of 637 mg 100 g^{-1} in freeze-dried blueberries. A similar result was obtained by Shivembe and Ojinnaka [30], they identified a TPC concentration of 427.06 mg GAE 100 g^{-1} in freeze-dried blueberries. Zhang et al. [31] validated a twice higher TPC content in lyophilized blueberry pomace than the value reported in this paper. At the end of the storage period, the TPC concentration of FD blueberries (443 mg GAE 100 g^{-1} d m) stored in the refrigerator was the highest, ahead of the TPC value of the FD-VD4h sample (412 mg GAE 100 g^{-1} d m) and the VD4h-FD sample (405 mg GAE 100 g^{-1} d m), with significant differences ($p < 0.05$) only for the refrigerated material. Compared to month 0, the TPC content decreased slightly for FD blueberries (by 1.77 % in the refrigerator and by 5.09 % at room temperature). Similarly to our results, Skoczeń-Słupska et al. [28] found that after 12 months of storage the retention rate of freeze-dried bilberries for polyphenols was 72–74 %, the lower value referring to storage at room temperature and the higher to refrigerated storage. (In this study the products were packed in jars with twist-off caps and stored without exposure to light.) Low degradation of phenolics (10 %) was observed in spray-dried and microencapsulated blueberry extract stored for 4 weeks at 4 °C in the absence of light [32].

During the 6-month storage period, the TPC value of MIR5min-FD and FD-MIR5min samples stored at room temperature decreases continuously. However, the TPC value of FD-MIR5min and VD4h-FD blueberries stored in the refrigerator remained almost constant. In contrast, the TPC value of FD blueberries stored in a refrigerator and at room temperature decreased during the first stage of storage, but remained constant from month 5 onwards. In the case of FD-VD4h and VD4h-FD samples, the values of polyphenols decreased slightly during the 6 months in both storage methods, except for VD4h-FD blueberries stored in the refrigerator. At the end of the storage period, the TPC of blueberry samples stored in the refrigerator was higher than at room temperature, but there was no significant difference ($p > 0.05$) between them.

Fig. 2 shows the variation in the total flavonoid content (TFC) of dried blueberries stored at room temperature and in the refrigerator. The FD, VD4h-FD, FD-VD4h, MIR5min-FD and FD-MIR5min dried blueberries contained 1.55, 1.33, 1.48, 1.26 and 1.19 mg CE g^{-1} total flavonoid content on dry weight basis, respectively. These results correspond to the initial data (month 0). As expected, the TFC value of the sample was highest for the lyophilization (FD). According to a previous research report, the total flavonoid content of freeze-dried blueberries ranged from 1.41 to 2.98 mg CE g^{-1} frozen berries [22].

At the end of the storage period, FD blueberries stored in the refrigerator retained the highest flavonoid content (1.41 mg CE g^{-1} d.m.), followed by FD and FD-VD4h blueberries stored at room temperature (1.35 and 1.34 mg CE g^{-1} d.m.), with no significant difference ($p > 0.05$) between them. Comparing month 6 with month 0, the TFC content decreased slightly for FD blueberries (by 9.03 % in the refrigerator and by 12.9 % at room temperature).

The TFC of blueberries dried by MIR5min-FD and FD-MIR5min methods remained practically constant over the studied storage period, except for the FD-MIR5min sample stored in the refrigerator. In the case of FD-VD4h and VD4h-FD samples, the values of flavonoids decreased slightly during the 6 months in both storage methods. At the end of the storage period, the TFC value of FD and MIR5min-FD blueberry samples stored in the refrigerator was higher than at room temperature, while the opposite was true for the FD-VD4h, VD4h-FD and FD-MIR5min samples, but there was no significant difference ($p > 0.05$) between them.

The antioxidant capacity (AC) of dried blueberries suffered a minimal loss (1.8–4.5 %) during the six-month storage period in both storage

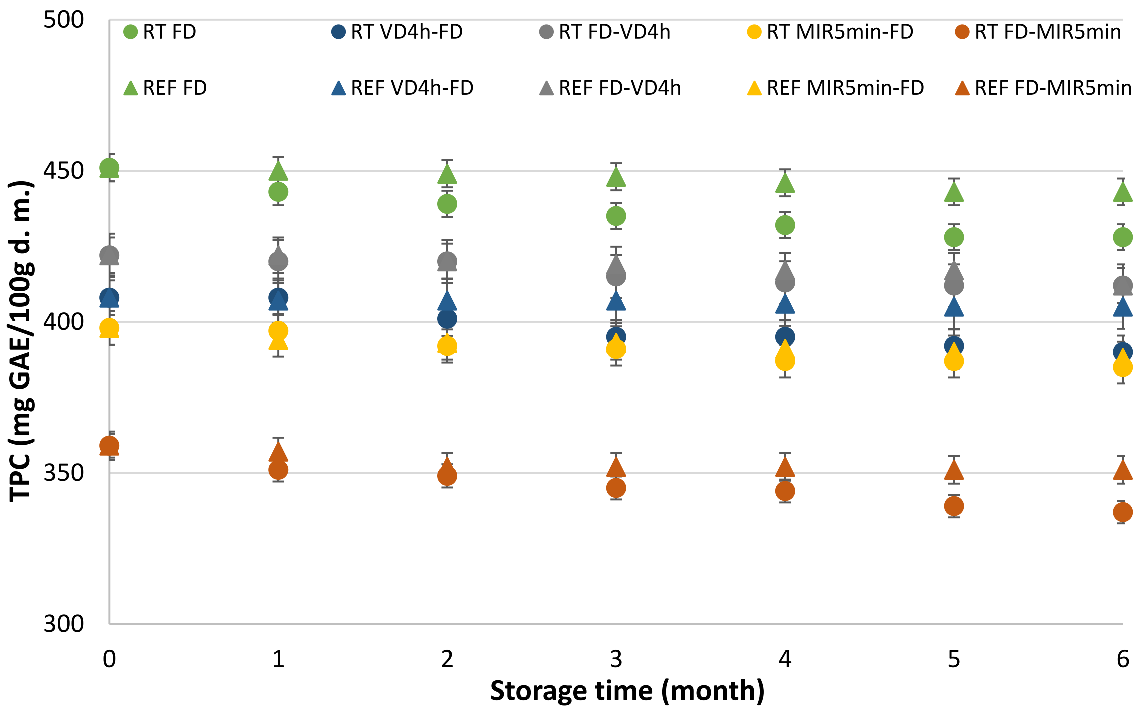

Fig. 1. Evaluation of total phenolic content (TPC) during storage obtained from dried blueberries
FD: freeze-drying, VD-FD: vacuum pre- and freeze post-drying, FD-VD: freeze pre- and vacuum post-drying, MIR-FD: mid-infrared-freeze-drying, FD-MIR: freeze-mid-infrared drying. RT: room temperature, REF: refrigerated. Data are expressed as the average ± standard deviation for three replicate.

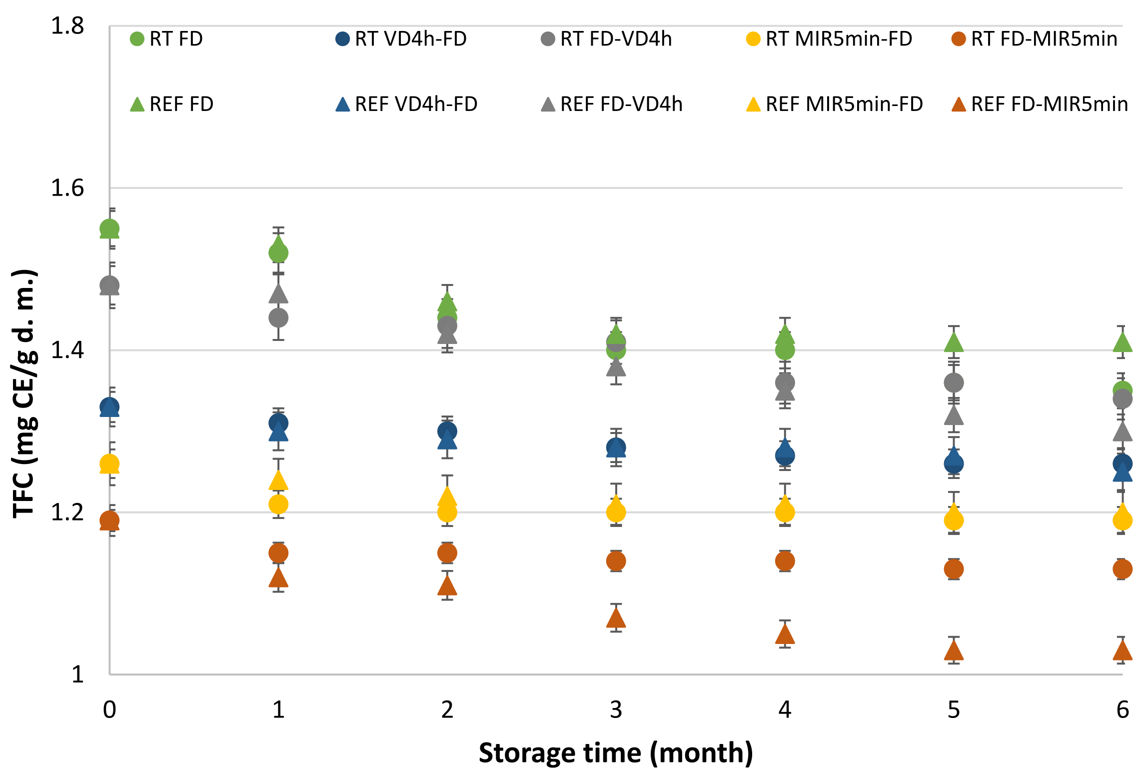

Fig. 2. Changes in total flavonoid content (TFC) of dried blueberries as a function of storage period
FD: freeze-drying, VD-FD: vacuum pre- and freeze post-drying, FD-VD: freeze pre- and vacuum post-drying, MIR-FD: mid-infrared-freeze-drying, FD-MIR: freeze-mid-infrared drying. RT: room temperature, REF: refrigerated. Data are expressed as the average ± standard deviation for three replicate.

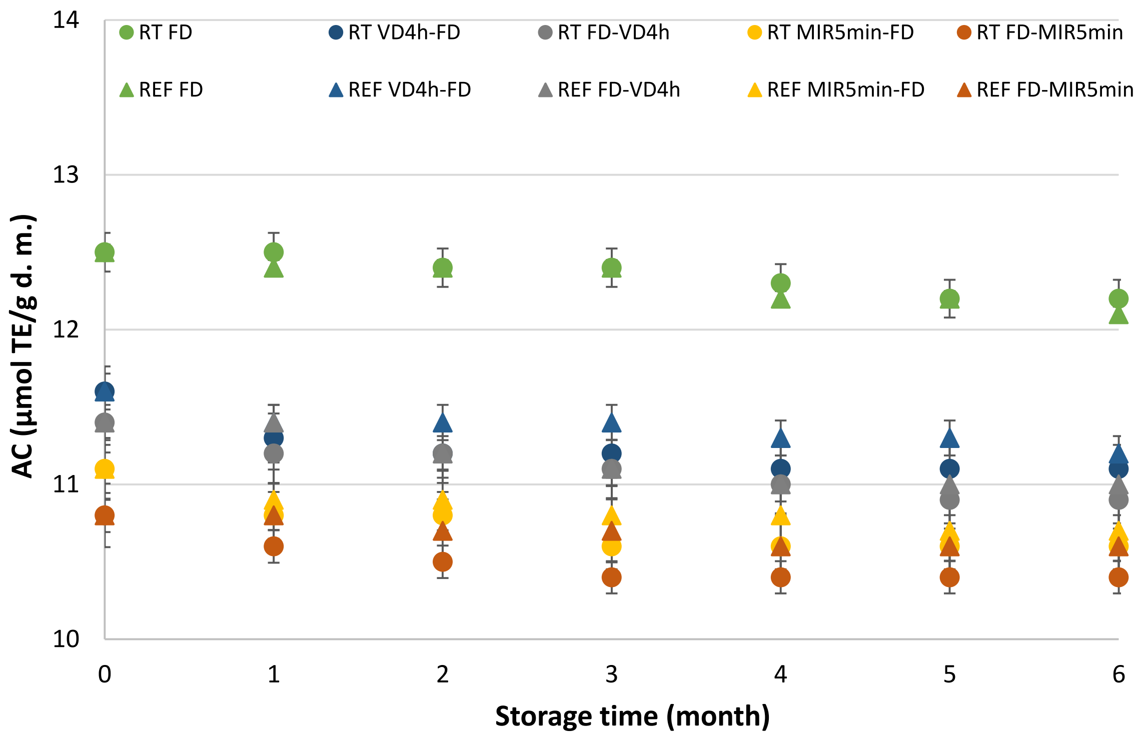

Fig. 3. Change of antioxidant capacity (AC) obtained from dried blueberries over 6 months of storage
FD: freeze-drying, VD-FD: vacuum pre- and freeze post-drying, FD-VD: freeze pre- and vacuum post-drying, MIR-FD: mid-infrared-freeze-drying, FD-MIR: freeze-mid-infrared drying. RT: room temperature, REF: refrigerated. Data are expressed as the average ± standard deviation for three replicate.

methods (Fig. 3). Similarly to our results, Calabuig-Jiménez et al. [17] found that the antioxidant capacity (DPPH, ABTS) of hot air-dried and powdered blueberry pomace did not change significantly, remained stable over the 20 weeks storage period. Immediately after drying, the FD, VD4h-FD, FD-VD4h, MIR5min-FD and FD-MIR5min dried blueberries had a total phenolic content of 12.5, 11.6, 11.4, 11.1 and 10.8 μmol TE g^{-1} on dry weight basis, respectively. At the end of the drying process (month 0.), freeze-dried (FD) blueberries contained the highest AC value (12.5 μmol TE g^{-1} d m.). Mejia-Meza et al. [11] found that freeze-dried blueberries showed higher antioxidant activity than the combination of microwave-vacuum drying method.

If the retention of the antioxidant capacity of dried blueberries at the end of the storage period is investigated, then the following order can be established: FD at room temperature (12.2 μmol TE g^{-1} d m.), FD in refrigerator (12.1 μmol TE g^{-1} d m.), VD4h-FD in refrigerator (11.2 μmol TE g^{-1} d m.), VD4h-FD at room temperature and FD-VD4h in refrigerator (11.1 μmol TE g^{-1} d m.). The AC value of FD samples was found to be significantly higher ($p < 0.05$) compared to the AC value of blueberries dried by combined methods. According to Fracassetti et al. [16], freeze-dried wild blueberry powder placed in vacuum packaging retains its antioxidant activity for 130 days when stored at 25 °C. Skoczeń-Słupska et al [28] observed that after 12 months of storage the retention rate of freeze-dried bilberries for antioxidant activity was 73–77 %, the lower value referring to storage at room temperature and the higher to refrigerated storage.

During the studied storage period, AC decreased slightly in most cases for both storage methods. The AC of blueberries dried by the MIR5min-FD method and storage at room temperature remained practically constant during the studied storage period. In contrast, the AC value of FD blueberries stored at room temperature decreased during the first stage of storage and remained constant from month 5 onwards. The antioxidant capacity of FD-MIR5min blueberries showed constant values throughout the whole storage period. At the end of the storage period, the antioxidant capacity of blueberry samples stored in the refrigerator was higher than at room temperature, except for FD, but there was no significant difference ($p > 0.05$) between them.

Low degradation of antioxidant capacity (15 %) was observed in spray-dried and microencapsulated blueberry extract stored for 4 weeks at 4 °C in the absence of light [32].

In conclusion, the dried blueberries showed a negligible quality loss during the storage period, due to vacuum packaging in the absence of oxygen. Masniyom [33] and Kulcu [34] were stated that vacuum packaging prevents deterioration caused by oxygen and increases the shelf life of the product.

3.4. *Summary of the results*

This section summarizes the results of the quality characteristics of blueberries preserved by different drying methods during storage (room temperature and refrigerator). The results are shown in Fig. 4. The chart is plotted so that the larger the colored area of the spider web graph for a given drying method, the better the method preserved the examined physical and chemical parameters. In the chart, each characteristic is given the equal weighting.

The information in Fig. 4 is of fundamental importance in the decision-making for selecting the appropriate drying method. In both storage methods, lyophilization (FD) has the largest area on the spider web graph. Although the drying time is not shown in Fig. 4, this indicator is worst ($p < 0.05$) for the FD method (Table 1). The FD-VD4h drying method has the second largest area after FD. Therefore, this paper suggests that freeze-drying can be replaced by FD-VD4h combined dehydration method. Since there was no difference in the values of the tested physical and quality parameters between storage at room temperature and storage in a refrigerator, storage on shelf is preferred for economic reasons.

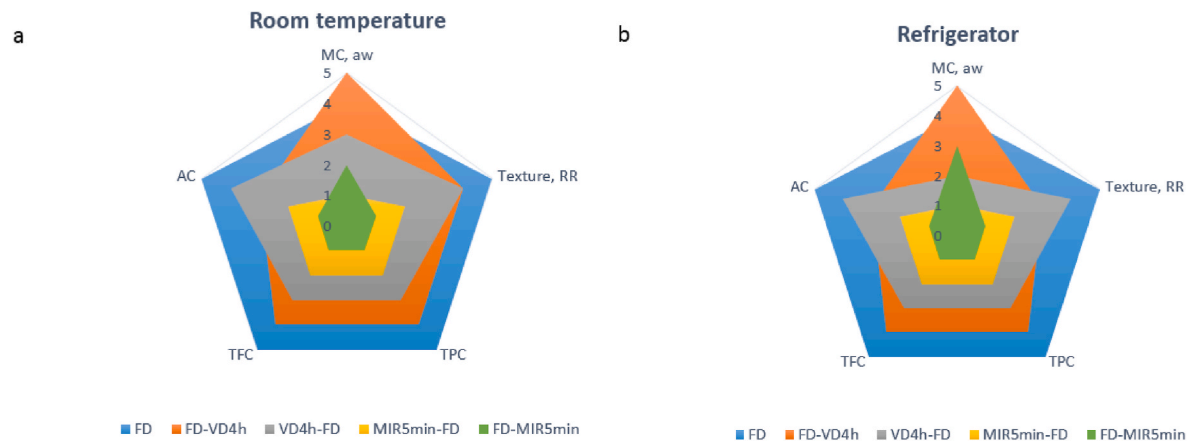

Fig. 4. Comparison of the final product quality of freeze-dried and combined method dried blueberries when stored; a) at room temperature, b) in a refrigerator, with the overall results on a spider web graph.
FD: freeze-drying, VD-FD: vacuum pre- and freeze post-drying, FD-VD: freeze pre- and vacuum post-drying, MIR-FD: mid-infrared-freeze-drying, FD-MIR: freeze-mid-infrared drying. MC: moisture content, a_w: water activity, RR: rehydration ratio, TPC: total phenolic content, TFC: total flavonoid content, AC: antioxidant capacity.

4. Conclusions

This paper has presented the effect of different storage conditions, namely refrigeration (5 °C in a refrigerator) and room temperature (23 °C on a shelf in a dark place), on the physical properties and chemical compounds of dried blueberry products. The length of the storage experiment was 6 months. In terms of physical (moisture content, rehydration, firmness, and water activity) and chemical parameters (polyphenols, flavonoids and antioxidants) tested, drying of blueberries by freeze pre-drying and post-vacuum drying solution of 4 h at 60 °C was the best compared to lyophilization. The drying operation times were ranked from lowest to highest, in the following order: freeze-infrared drying, infrared-freeze-drying, freeze-drying-vacuum drying, and vacuum drying-freeze-drying and conventional lyophilization.

The observed values of polyphenols, flavonoids and antioxidants in the dried and vacuum-packaged sample were slightly reduced during storage, with no significant differences between them. The moisture content, rehydration, hardness, and water activity of dried blueberries placed in vacuum packaging remained unchanged during the 6 months of storage, regardless of the storage method. There was no significant difference between the vacuum-packaged blueberries stored at room temperature and at the refrigerator in terms of the tested parameters. In terms of preservation of chemical compounds, texture, rehydration and water activity, the most stable product was obtained by a combination of freeze-drying and vacuum drying (FD-VD4h60 °C), in addition to conventional lyophilization. It is recommended to store the dried blueberry products in vacuum packaging using a more economical storage method, i.e. at room temperature in a dark place.

CRediT authorship contribution statement

Tamás Antal: Writing – review & editing, Writing – original draft, Validation, Software, Project administration, Methodology, Investigation, Formal analysis, Data curation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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