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SPECIFICITIES OF FRUIT FREEZE DRYING AND PRODUCT PRICES¹

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Summary

Freeze drying, as relatively new process of drying in vacuum at very low temperatures, ensures the preservation of all thermo labile compounds in the initial raw material, and final low content of moisture provides microbiological stability and permanent preservation of products. Red raspberry, because of relatively high content of water (90%), specific structure of aggregate fruit, characteristic bright colour and flavour, was not preserved up to now by conventional drying.

This paper gives an overview on the two different methods of drying fruits: freeze drying and convective drying.

Raspberry sorts Willamette and Meeker were dried by freeze drying, as well as by conventional drying with warm air of low relative humidity. Freeze drying was performed in the device Christ Alpha I/5 under desublimers' temperature of -55°C and processing temperature of raw material of -35°C. Fruits of the sort Willamette were dried to a final moisture content of 18.86%, and Meeker up to 16.15%. Also, some changes in chemical composition, overall aroma, sensory characteristics, density and volume, water activity, loss of vitamin C, the degree of rehydration of the dried fruit in comparison to fresh one were tested. Gained results show that freeze drying can effectively preserve the chemical composition, volume, colour and aroma of raspberry.

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In the paper of authors from foreign countries the influence of different technologies for the products preservation was tested as freezing, freeze drying (or lyophilisation) and conventional drying. Tests were done with the main goal to determine the qualitative changes on strawberry, raspberry, gooseberry, elder, apricot, sour cherry, apple and cornelian cherry. All preservation technologies have reduced the amount of biologically active compounds, such as vitamin C and phenolic compounds. Related to the chemical composition of fresh fruit, decrease of tested parameters was affected by freezing for 15%, freeze drying for 28-32% and conventional drying for 45-48%.

The process of freeze drying is among one of the methods for preservation of raw materials of plant origin (fruits, vegetables, spices and herbs). Final product has a high nutritional value (as fresh fruits and vegetables). Freeze dried products do require special storage conditions (e.g. absence of light, packaging materials with low gas permeability, inert atmosphere, etc.). In this way dried products represent the basis for instant soups, bakery, dairy and confectionery products.

Key words: fruit, raspberry, freeze drying, conventional drying, biologically active compounds, quality.

JEL: *Q16, Q10*

Introduction

Freeze drying is a relatively new technology in the field of preservation of food products. Primarily it was developed for the pharmaceutical industry and drugs drying. For process of freeze drying raspberry first has to be frozen to a temperature usually lower than -30° C (*Janković et al., 2004*). Frozen raspberry is brought into the sublimation chamber where after closing and vacuuming is achieved extremely low pressure, below 10^{-1} bar. Under the influence of high vacuum, ice sublimates in frozen raspberry. Crystals of ice are transferred directly into vapour, thereby avoiding the appearance of liquid phase and migration of dissolved dry matter to the surface. In the pharmaceutical products the moisture content is decreased up to 1 to 5%, depending on the kind of products, while raspberry is dried to the moisture level of 10%. Dried raspberry is packed in gas impervious packaging, vacuum packaging or in package with high level of nitrogen. Freeze drying raspberry can be stored at room temperature in packaging that is light resistant up to 5 years.

In the researches of *Janković et al. (2004, 2006, 2010)*, whole fruits of raspberry sorts' *Willamette* and *Meeker* were freeze drying, with main goal to investigate all changes of volume, density, water activity, loss of vitamin C, the level of rehydration, as well as changes in chemical composition of freeze drying fruit.

Simplified, the freeze drying is a drying process where water is removed (drying of product) by sublimation of ice from previously frozen product (*Vračar et al., 2004*). As the product is dehydrated under vacuum in a frozen state, at temperatures below -30°C, and in the final stage of drying (separation of bound water) temperature under vacuum does not exceed 40°C, product practically preserves its structure and shape, chemical composition,

biologically-physiologically and sensory characteristics (colour, odour, taste). Process of freeze drying is practically done in three main phases:

- Freezing, or sub-cooling of product below its eutectic point (cca. -30°C and lower);
- Dehydration (drying) by ice sublimation under vacuum; and
- Completion of product drying up to moisture content lower than 3% by the normal vacuum drying.

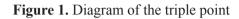
Each of these phases, particularly sublimation, is important for the quality of freeze dried product. Those products are characterized by high porosity and therefore by great surface activity, that results easy absorption of moisture and oxygen. According to many undesirable quality changes caused by adsorption, de-vacuuming of chamber after completion of lyophilisation is performed with an inert gas (nitrogen) and quick packaging into the material that is gas, moisture and light resistant. Freeze dried product could be kept in a nitrogen atmosphere almost indefinitely. Compared to other drying processes, freeze drying has many advantages.

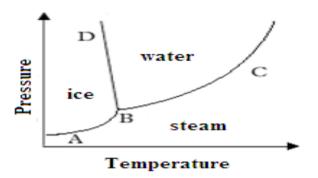
It is well known that fruits and vegetables have many benefits for life and health (*Sipos et al., 2009*). Beside their different and balanced mineral components, there are several organic compounds that are proven from medical aspect. One of the most important effects is their fibber content, which is related to the prevention of coronary arteriosclerosis and other diseases. Nutrients and vitamins dissolve in a product with high moisture content, so they can be easily and effectively used by the human organism. Just through recent decades scientific researches have focused on biologically active compounds, such as microelements and phenolic compounds, that all have antioxidant characteristics too. Although the high content of biologically active compounds was found in tropical and subtropical fruits, traditionally continental fruit also contains significant amounts of these compounds. Unfortunately, vitamins, antioxidants, flavour, aroma and other organic compounds are not resistant to heat stresses, so in several traditional technologies of food processing significant loss of final product occurs.

In Serbia exists favourable climate-edaphic conditions for development and further improvement of fruit growing especially on family agricultural husbandries. Usage of given conditions considers previous establishment of suitable ambient for quick recovery of complete agriculture and economy. There is a need for defining of developmental programs based on marketing in accordance with available ecological conditions and requirements from contemporary national and international market (*Vukoje, Milic, 2009*).

Water, as the matter, could be present in all three aggregate states, depending on the temperature and by the constant atmospheric pressure. With increase in temperature water changes its' aggregate state: solid, liquid or gaseous (*Ivančević et al., 2003*). When the temperature and pressure are decreased in controlled conditions, in one moment water can be brought into condition that at the same time is solid, liquid and gaseous. This point at which all three states of water are in equilibrium is called the triple point. Triple point for water is under

temperature of 0°C and pressure of 0.006 bars. At pressure and temperature below the triple point, water in form of ice goes directly to the gaseous state - steam (Figure 1). Process of direct conversion of solid to gaseous phase is called sublimation, and for evaporation and drying has to be used the latent heat of raw material. During this process temperature of raw materials is constant, despite the heat dissipation.





Man was worried with the problem of preservation at that moment when he wanted to save catch or harvested crops for certain time, because of food security (*Tosić et al., 2003*). Food in its original form contains moisture, which is a good basis for the development of various pathogens. In order to prevent the development of pathogens or to preserve food, various forms of preservation are used: drying, freezing, use of various microbiological and chemical processes, storage in hermetic vessels, etc. Preservation of fruits and vegetables by microbiological, chemical and thermal processes in developed world is becoming much less used, while in expansion is preserving by freezing. There are four basic conditions necessary for the functioning of freeze drying process:

- raw materials must be deeply frozen;
- condensation surface must be at a temperature below -20°C;
- system must be able to provide an absolute pressure of minimum 200 mm Hg;
- existence of controlled heat resources (-40°C and +65°C), which will release the latent heat of sublimation for the transfer of ice into the vapor.

The raw materials are delivered as fresh or frozen. It is stored in the warehouse (cold storage) which is projected to for about 500 t of raw material (it was calculated for frozen peas). It consists of two chambers, one with a constant temperature of -20°C and the second where is around 0°C. In second chamber raw material is prepared for drying (unpacking, spreading on the trays, etc.). The fruits have to be frozen for 24 hours at -20°C. On this way prepared raw material is transported to the dryer, where the drying process by freeze drying lasts from 12 to 24 (48) hours, under conditions of low temperature and high vacuum. Dried product is transported to the department for quality control, where organic and metal impurities are removed. After inspection and sampling, dried product is packed in the final packaging, put on pallets and taken to the warehouse for finished products. Trays

and shelves, where the raw materials were during drying process have to be washed and prepared for the new cycle.

Mitrović and Marković (1996) provide an overview of the development of devices for convective drying of fruits; vegetables and herbs while *Brkić et al. (1998)* give the results of convective drying of sour cherry at batch dryer *Seting*.

The aim of this study was to compare the results of freeze drying fruit with classical convective drying method by several authors.

Material and method

Janković et al. (2010) studied raspberry sorts *Willamette* and *Meeker*, with average diameter of 19.40 mm, average weight of fruit 3.92 g, dark pink, pronounced flavour and aroma characteristic for mentioned varieties. Whole fruits of raspberry were freeze dried.

Raspberries were frozen in classic tunnel under the temperature of -35°C and stored in a chamber at -20°C, until freeze drying. Freeze drying was performed in the device Ehrist Alpha I/5, under the desublimers' temperature of -55°C and processing temperature of material of -35°C, as well as in device Edwards at a temperature of -30°C, pressure of 13Pa and the desorption temperature of 40°C. *Willamette* was dried to the final moisture content to 18.86%, and *Meeker* up to 16.15%. Convective drying is performed in a laboratory apparatus with inlet air temperature of 65°C and relative humidity of 6%.

For the freeze drying process *Vračar et al. (2004)* used the raspberry sort *Willamette* harvest 2003, which was prepared and frozen in cold storage in company Vino-Župa Aleksandrovac. Freeze drying was conducted in company Art-Arom Subotica, in semi-industrial device Usi-Prodi made in France, with a batch of 10 kg under the following conditions: condenser temperature -57°C, temperature at the beginning of sublimation -40°C, final temperature of 30°C, vacuum 10⁻³ mbar, eutectic point was empirically determined, freeze drying lasted for 36 h. Chemical parameters of frozen and freeze-dried raspberry were determined by standard chemical methods (*Vračar, 2001*).

Hungarian researchers have examined the fresh fruit (strawberry, raspberry, gooseberry, elder, apricot, sour cherry, apple and cornelian cherry) purchased on the market in Debrecen (*Sipos et al., 2009*). Identical sample was applied at different food preservation technologies. Apples and apricots were cut into pieces 7-8 mm wide, while other fruit samples were washed and dried by classical method. Freezing was conducted as in households, in commercially available freezers. Fresh fruit was frozen to -18°C and kept 3-7 months. Samples were stored before freeze drying in the refrigerator at the temperature of -18°C. Freeze dryer is produced in company Heto Power Dri PL9000 (Thermo - Fisher Scientific Laboratories, USA). Scale of freeze dryer was from -40 to +42°C at 1 mbar. Convective dryer, ie. laboratory desiccators was made by the company Metefem FTL-2004L (Metefem, Hungary). It worked at 40°C for 1-2 days. Dry samples were stored in closed anti-steam plastic bags. Experiments were repeated for three times.

Results and discusion

Within the chemical analysis of fruit samples dry mater, total acidity, anthocyanins and sugars, pH value and L-ascorbic acid were determined. Chemical analysis of dried fruits was performed after rehydration, *Janković et al. (2010)*. Change of the water content in raspberry during the drying process was measured every hour and it ranged at sort *Willamette* from 82.46 to 18.86% after 48 hours of drying, and at the sort *Meeker* from 81.85 to 16.65%.

Convectively dried fruits had slightly lower content of moisture, 9,9%, while at the same time freeze dried raspberry had 11,59% of moisture. Based on the experience, it was determined that at freeze dried fruit with moisture content lower than 10% comes to coronation, due to the mutual friction of packed fruit. Differences in the content of total acid, glucose and pH values did not differ significantly compared to fresh fruit.

Significant difference was noticed in the content of L-ascorbic acid. Loss of L-ascorbic acid in convectively dried fruit was 63.83%, while in freeze dried was 21.28%, *Janković et al., (2006).* Although the air temperature in convective drying was not to high, change in the L-ascorbic acid content occurred due to activity of oxygen, enzymes, metal ions and transformation into D form. At freeze dried fruit was dried whole fruit in which the loss is proportional to the square of the diameter. As according to *Karel,* drying time is increase with the square of diameter (*Janković et al., 2004*).

The loss of anthocyanins in the fruit is affected by the same factors that lead to the reduction of L-ascorbic acid, such as higher temperature, presence of oxygen, enzymes, metal ions, etc. At convectively dried raspberry loss was 52.21%, while in freeze dried it was only 1,80% (*Janković et al, 2006*). According to literature, the degradation of anthocyanins is proportional to the logarithm of temperature and is the major cause of loss of colour at pH 2–4.

In fresh raspberry 25 peaks with retention times of 3.79 to 53.06 was found (*Janković et al*, 2004). Comparing with similar retention times peaks, at convective dried and freeze dried raspberries the decrease or loss of certain peaks was noticed, as well as the loss of flavour. In convective drying loss was 49.86% and in freeze drying 16.85%. The loss of aroma in freeze drying process is inversely proportional to the content of dry matter in fruit, and depends of the rate of freezing. When the rate of freezing is lower large cavities appear in the dried matrix, which leads to fall of vapour pressure above the dried layer and later to lower temperature of sublimation of ice fronts at the same temperature of heater.

As very important indicator of freeze dried fruit quality is the level of reduction of volume and porosity. Based on *King (Janković et al, 2004)*, change of freeze dried fruit volume in relation to a fresh is low, from 2 to 15%. If due to bad conduction of the freeze drying process comes to afore mentioned collapse of the matrix, it will be get a product which has a large reduction in volume and low porosity. Gained results show that during the process of freeze drying, reduction in volume is very small, only 6%, so in that way dried fruit by shape can be hardly distinguished from the fresh fruit.

Particularly interesting is the extremely high porosity of the freeze dried raspberry. With more than 85% of porosity it remains on the structure like sponge. High porosity on one hand requires very good protection of dried fruits from oxidative changes, as the border area is enormously large. Therefore, the freeze dried products are packed in gas resistant containers, in the atmosphere of gaseous nitrogen instead of air. On the other hand, high porosity has great importance on the speed and level of rehydration.

The reduction of water activity is the essence in food preservation by drying and freeze drying. Activity of any kind of microorganisms could be inhibited when the value of water activity falls below 0.6. From obtained data could be seen that the water activity in freeze dried raspberry was 0.3 and even lower, which promotes it as not favourable environment for the development of osmosis yeasts. Freeze drying process is conducted at lower temperatures so there were no significant changes in chemical composition of raspberry, and the best indicator for the quality of the applied procedure is change in content of thermo-labile vitamin C or L-ascorbic acid. Gained results show that in freeze dried *Willamette* loss of vitamin C was about 17%, and at *Meeker* about 25%, what was significantly lower compared to other drying processes.

The degree of rehydration represents the ratio between fruit weight that was submerged in water for a period of 24 hours at room temperature and weight of same freeze dried fruit before rehydration. Due to the high porosity freeze dried fruits have a relatively high degree of rehydration. Raspberry cultivar *Willamette* had a rehydration degree of 3.27, and *Meeker* 3.36. A slightly higher degree of rehydration was found in the sort *Meeker*, although its porosity was slightly smaller, and that could be explained by the higher content of pectin within the sort *Meeker*.

Results of chemical analysis of frozen raspberry sort *Willamette* (harvest 2003) gained by *Vračar et al. (2004)*, confirmed many results found in literature. They confirm that raspberry is a fruit with low energy value and significant content of the substances that provide to the organism protection organism, as well as medical and dietary impacts. Colour substances in quantities of 0.21% represent anthocyanins which in acidic environment give red colour. Antioxidants are also important, as they protect the human organism from the cardiovascular disease, cancer and other degenerative diseases of ageing. According to the content of vitamins raspberry is not a significant source, particularly in relation to other berry fruit.

According to results of foreign authors researchers about chemical characteristics of fruits, fresh *Cornelian* cherry and strawberries had the highest content of vitamin C, while the lowest values were in apricot and sour cherry (*Sipos et al., 2009*). Phenols have appeared in high concentration within the blue and violet colored fruits. Because of that it is obvious that the sample of elder showed the highest concentration in the fresh material. Similarly, high value of phenolic content was in cornelian cherry, sour cherry and raspberry, while apricots and apples had its lowest level in fruits.

It was noticed that all tested preservation technologies had a significant impact on the content of vitamin C in fruit. The loss of vitamin C by freezing was in average 193%

compared to fresh fruit, by freeze-drying up to 323%, and by conventional drying up to 45.3%. All samples of fruit showed the same tendency.

Cornelian cherry and gooseberry kept their highest relative content of vitamin C, while older apple and raspberry loosed relatively the highest rate, so starting level of vitamin C did not affect the loss rate. Freeze drying and freezing affected almost the same amount of loss in case of sweet cherry and apricot.

Storage technology also affected the total content of phenols in fruits. In average, different types of treatment caused next losses: freezing 15%, lyophilisation 28.1%, desiccation 48.4%, so the tendencies and rates of changes are similar to the content of vitamin C. Differences in this parameter between the results for different types of fruit are less significant. There were only a few percent differences in reducing the total phenolic content between the different treatments.

Authors from the USA mostly worked on quality of freeze dried products, or they tested the impact of freeze drying on quality of dried fruit products. In Table 1 are given the approximate costs for certain raw materials, as well as the costs of energy, and production and sale prices of freeze dried product, determined by the researchers of the *Van Drunen Farms* – USA. Shown data basically support the attractiveness of this technological concept in economic sense.

In Table 2 are presented the data of certain products which are dried in a drum dryer. By comparison of data from these tables next conclusions could be reached: freeze dried products are in average two or more times expensive on the market; for the freeze drying process is necessary to employ more energy; price of freeze dried products cover the cost of drying and potential profit.

No.	Culture	Needed raw material (kg)	Price of raw material (\$/kg)	Cost of energy (\$/kg)	Production cost (\$/kg)	Selling price (\$/kg)
1.	Bilberry, whole fruit	6.8	2.24	1.43	24.64	25.08
2.	Peach, cubes	11.0	1.69	1.21	33.55	36.08
3.	Cranberry	9.5	1.32	1.32	26.33	27.39
4.	Sour cherry	8.0	1.43	1.43	22.17	22.17
5.	Apple cubes	11.0	1.05	1.65	15.62	16.36
6.	Strawberries, whole fruit	10.5	1.49	1.32	31.04	32.25
7.	Apricot	7.0	1.87	1.43	24.86	30.80
8.	Chestnut	9.0	1.54	1.32	27.03	30.14
9.	Blackberries, whole fruit	7.0	3.30	1.32	33.96	35.20
10.	Pear	9.0	1.98	1.32	31.17	31.17
11.	Lemon, chopped	9.0	2.20	1.40	34.07	34.07
12.	Raspberries, whole fruit	6.5	2.20	1.54	25.52	26.40
13.	Orange, pulp	10.0	1.98	1.54	36.96	36.96

 Table 1. Drying of fruits by freeze drying, approximate values (Van Drunen Farms)

No.	Culture	Needed raw material (kg)	Price of raw material (\$/kg)	Cost of energy (\$/kg)	Production cost (\$/kg)	Selling price (\$/kg)
1.	Blueberry	4.75	1.10	5.50	10.72	11.55
2.	Peach	6.0	2.97	5.72	23.54	24.20
3.	Cranberry	4.5	1.32	6.60	12.54	16.50
4.	Cherry	5.0	1.32	5.50	12.10	12.65
5.	Apple	5.0	0.33	4.29	5.94	5.94
6.	Strawberries	8.0	0.99	6.60	14.52	15.18
7.	Pear	8.6	1.21	6.51	16.91	17.60
8.	Blackberry	5.0	1.38	6.60	13.53	15.40
9.	Raspberries	4.0	2.86	5.28	16.72	17.27
10.	Plum	5.0	1.36	4.40	11.22	11.88

Table 2. Drying of fruits by drum dryer	, approximate values ((Van Drunen Farms)
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Conclusion

According to the research results about the quality of freeze dried raspberries it could be concluded that freeze drying is a very suitable method for drying of sensitive fruits such as raspberry. The results of all analyzes indicate that the quality of convectively dried fruits in comparation to freeze dried is much worse. Advantage of freeze drying is reflected in better preservation of L-ascorbic acid, for about 54%, anthocyanins, for around 51%, lower loss of total flavour, for about 66%, lower volume reduction, for about 92%, higher porosity, for about 49% and better organoleptic mark, for about 51%. All these advantages of freeze drying are realized due to the drying by sublimation from frozen state under the relatively low temperature within the desorption process.

Freeze dried raspberry has a low water content and low water activity, and could be considered as microbiologically safe and permanently preserved if it is stored in appropriate gas-impermeable containers. The porosity of the freeze dried fruit is over 80%, so at first glance they are very little inconsistent to the fresh fruits. Rehydration of freeze dried fruits is fast and good. Changes in chemical composition are minimal, so higher level of preserved vitamin C, anthocyanins and colored matter is very important. Anthocyanins belong to a group of polyphenols, and they are of great importance in human nutrition (they are included to a group of *free radical catchers* with anti-cancer characteristics).

In freeze dried raspberry of sorts *Willamette* and *Meeker* were not found major differences in physical, as well as, in chemical composition and organoleptic characteristics. *Willamette* has larger and more intensely colored fruits than sort *Meeker*, while *Meeker* has higher content of pectin, so all observed differences can be explained by the differences of the characteristics in initial raw material.

In a survey of foreign authors it was determined the reduced amount of biologically active compounds at eight fruit species, at all processing treatments: freezing, lyophilisation and conventional drying. Level of change in different preservation technologies is similar to content of vitamin C and total phenols. In relation to the chemical composition of fresh fruit, freezing resulted in reduction of investigated parameters for 15%, freeze dried for 28-32%

and conventionally dried up to 45-48%. Therefore, freezing is the most favoured procedure for obtaining of all healthy components, while freeze drying keeps the most natural look of preserved fruit, so after rehydration it was received the original taste and shape of fruit.

In some researchers have come to the conclusion that freeze dried products are in average more expensive on the market (for two or more times), since freeze drying process necessary consume more energy. Despite mentioned price of these products covers the costs of drying and gained profits.

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SPECIFIČNOSTI SUŠENJA VOĆA LIOFILIZACIJOM

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Rezime

Liofilizacija, kao relativno nov postupak sušenja u vakuumu pri vrlo niskim temperaturama, obezbeđuje očuvanje svih termolabilnih jedinjenja u početnoj sirovini, dok krajnji nizak sadržaj vlage obezbeđuje mikrobiološku stabilnost i trajno konzervisanje proizvoda. Crvena malina zbog ralativno velikog sadržaja vode (oko 90%), specifične strukture zbirnog ploda, karakteristične svetle boje i arome, nije do sada konzervisana klasičnim sušenjem.

Malina, sorte "vilamet"i "miker" sušena je postupkom liofilizacije i klasično, toplim vazduhom niske relativne vlažnosti. Liofilizacija je vršena u uređaju "Ehrist alpha I/5", pri temperaturi desublimatora –55°C i radnoj temperaturi materijala –35°C. "Vilamet" je sušen do krajnjeg sadržaja vode do 18,86%, a "miker" do 16,15%. Ispitivane su promene u hemijskom sastavu, ukupnoj aromi, oganoleptičkim svojstvima, gustini i zapremini, aktivnost vode, gubitak vitamina C, stepen rehidracije kod osušenih plodova u odnosu na sveže. Dobijeni rezultati pokazuju da se liofilizacijom može uspešno očuvati hemijski sastav, zapremina, aroma i boja maline.

U radu mađarskih stručnjaka ispitivan je uticaj različitih tehnologija za konzervisanje proizvoda: zamrzavanje, sušenje i sušenje zamrzavanjem (frezze-drying ili liofilizacija). Ispitivanja su obavljena u cilju ustanovljavanja kvalitetnih promena na jagodama, malini, ogrozdu, zovi, kajsiji, višnji, jabuci i drenu. Sve tehnologije konzerviranja smanjile su količinu biološki aktivnih jedinjenja, kao što su vitamin C i ukupni fenoli. U odnosu na hemijski sastav svežeg voća, smanjenje ispitivanih parametara rezultiralo je: 15% zamrzavanjem, 28-32% liofilizacijom i 45-48% sušenjem.

Proces liofilizacije u SAD je jedan od postupaka konzervisanja sirovina biljnog porekla (voća, povrća, začinskog i lekovitog bilja). Finalni proizvod ima visoku prehrambenu vrednost, kao i sveže voće i povrće uz prednost da se lako čuva i da ne zahteva nikakve posebne uslove skladištenja. Ovako sušeni proizvodi su osnova za instant supe, pekarske i mlečne proizvode, kao i za slatkiše.

Ključne reči: Voće, malina, liofilizacija, sušenje, biološki aktivna jedinjenja, kvalitet.

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