Development of sublimation drying modes of rose hip fruits Freezing drying of rose hip

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Abstract. The aim of the study was the selection of effective modes of rosehip sublimation dehydration. An Iney-6M unit was used for freeze drying. The selection of the duration of the sublimation stage and the final drying temperature was carried out. At the same time, such indicators as the duration of drying, organoleptic assessment and the preservation of vitamin C were analyzed. The duration of freeze drying of rose hips with a period of sublimation of 4, 5, 6 and 7 hours was 6, 7, 8 and 8.5 hours, respectively. It was found that with a decrease in the duration of the sublimation stage, a decrease in the quality characteristics of dry rose hips is observed. It was found that when the drying temperature changes from 70 to 40 °C, the drying time increases from 6.5 to 9.5 hours. At the same time, the loss of vitamin C also decreases. Based on the research, the most favorable modes of sublimation dehydration of rose hips have been determined - the final drying temperature is 50 °C and the duration of the sublimation stage is 5 hours. Under these modes, the duration of lyophilization is 8 hours, the total organoleptic assessment is 18.25 points out of 20, and the preservation of vitamin C is 27%. Lyophilized rose hips can be used as an independent product, or ground into powder for adding to other products - confectionery, dairy, bakery products, as well as biologically active additives and functional food.

1 Introduction

Rosehips are a rich source of vitamins, minerals and essential amino acids [20, p. 108; 19, p. 1; 22, p. 1]. High content of vitamin C is especially noted in rose hips - its concentration ranges from 400 to 1500 mg/100g [9, p. 310; 16, p. 116]. This is several times more than in currants and tens of times more than in apples. The highest content of vitamin C is observed in mature, but firm rose hips. In addition to ascorbic acid, the rose hips also contain vitamins A, E and B1.

In addition, rose hips contain citric and malic acids, pectin substances, as well as micro - and macronutrients such as potassium, calcium, iron, manganese, etc. The chemical and mineral composition of rose hips is presented in table. 1 [2, p. 112].

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Table 1. Chemical and mineral composition of rose hips [2, p. 112].

Chemical composition		Mineral composition	
Water, %	64,89-67,89	Potassium, mg/g	19,60-20,60
Dry matter, %	32,11-35,11	Calcium, mg/g	4,36-4,96
Ascorbic acid, mg%	521,49-528,51	Magnesium, mg/g	4,76-5,36
Flavonoids, mg %	150,4-154,4	Copper, mg/g	0,10-0,20
Pectin substances, mg %	0,019-0,021	Iron, mg/g	0,03-0,15

The substances that make up the rose hips have a beneficial effect in inflammatory processes, accelerate tissue regeneration, better carbohydrate metabolism and improve vascular permeability [18, p. 3; 24, p. 564]. Rosehip oil helps to reduce gastric secretion and gastric acidity. In addition, the pectin substances that make up the rose hips remove heavy metals from the body [6, p. 120]. This is the reason for the use of rose hips not only in the production of confectionery products, juices, tea, etc., but also biologically active additives and functional food products [23, p. 45].

From rose hips, liquid concentrates and dry powders are produced. In the latter case, it is necessary to use drying plants.

2 Literature review

Drying of rose hips is carried out for technological reasons, or to extend its shelf life: fresh rose hips are stored for no more than 2-3 weeks. Drying in the open air is practically not used, since the temperature does not allow the rose hips to dry properly.

The most common method is convective drying [11, p. 7]. The disadvantage of this method is the relatively long duration of the process. An increase in the temperature of the drying agent is possible only up to a certain value - at too high temperatures, denaturation of thermolabile components of the product is observed. In the literature, there is evidence that the drying of rose hips should be carried out at a temperature of the order of 60-70 °C [9, p. 311; 8; 15, p. 672].

To intensify the drying of rose hips, ultrasonic radiation is used. So, in one of the works [1], the drying of rose hips was carried out in a combi steamer with an ultrasonic apparatus. Ultrasonic waves allow warm air to quickly destroy the boundary layer and penetrate to the center of the product, which causes uniform heating and uniform moisture removal. In the course of research it was found that the use of ultrasound can reduce the drying time by 2 times, as well as reduce the loss of vitamins by 17% [1, p. 83].

Microwave treatment is also used to destroy the cell structure of berries. The invention [4] proposes the processing of rose hips in a microwave field for 1.0-1.5 minutes, after which the rose hips are sent for drying at a temperature of 60-65 °C for 9-10 hours. This method allows you to speed up the process of removing moisture from the fruit and reduce the loss of vitamin C.

It is possible to intensify the process of drying fruits and berries by lowering the pressure - using the so-called vacuum drying [21, p. 3765; 10; 17, p. 42]. Vacuum drying is widely used not only for drying fruits and berries, but also for many other products [3, p. 121; 4, p. eighteen; 5, p. fourteen; 12; 13; 14]. Vacuum drying can be carried out in several stages. For example, in the invention [7] it is proposed to dry berries in 2 stages: in the first stage, the residual pressure is 8-12 kPa, and the drying temperature is 60-65 °C, in the second stage, the residual pressure is 3-4 kPa, and the temperature is 70-80 °C.

Lyophilization - freeze drying is a promising method of dehydration. It is carried out at a pressure below the triple point of water. The product self-freezes at this pressure and moisture sublimates from a solid state to a gaseous state. The advantage of freeze drying is the ability to carry out the process at a relatively low heating temperature, which allows you

to preserve the valuable components of the product.

Thus, the purpose of this work was to select the modes of freeze drying of rose hips.

3 Materials and methods

The object of research was rose hips (harvest 2020).

For freeze drying, there was used an installation *Iney-6M*, the diagram of which is shown in Fig. 1.

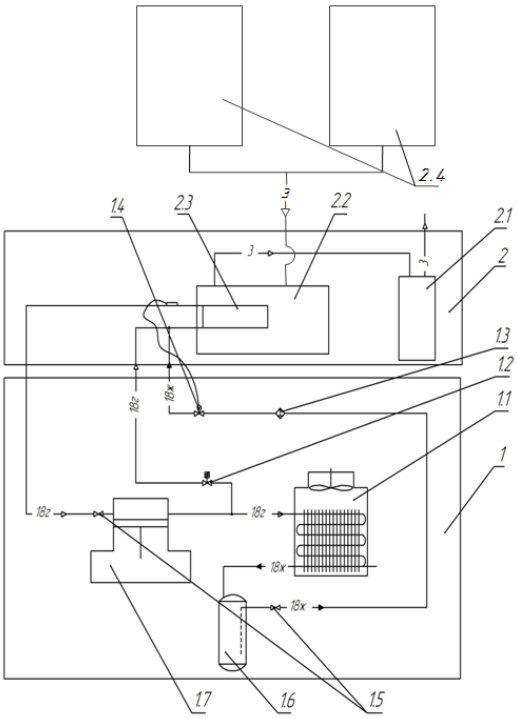

Fig. 1. Schematic diagram of a freeze drying plant:
1 - refrigerating machine; 1.1 - condenser; 1.2 - solenoid valve; 1.3 - filter drier; 1.4 - thermostatic valve; 1.5 - shut-off valve; 1.6 - receiver; 1.7 - compressor; 2 - vacuum unit; 2.1 - vacuum pump; 2.2 - desublimator; 2.3 - evaporator of desublimator; 2.4 - drying chambers

Operating this installation (Fig. 1), the product is put on pallets, which are placed in drying chambers. Then the chambers are closed and a vacuum pump is turned on, reducing the pressure to 50 Pa. To supply heat, heating lamps are used, placed vertically in the drying chambers. The desublimator freezes out the moisture contained in the removed air. Heat removal from the heat exchange surface of the desublimator is carried out due to the operation of the refrigerating machine, which consists of a compressor, condenser, filter-dryer, evaporator, receiver, thermostatic, solenoid and shut-off valves.

Drying was carried out in 2 stages: a sublimation stage and a final drying stage. During the sublimation stage, the heating lamps were turned off, and the moisture contained in the product was sublimated into the environment. At the second stage, the heating lamps were switched on and the product was dried up. Drying was carried out until a moisture content of about 5% was reached.

The moisture content was determined by an accelerated method using a device *Chizhov*, by drying a sample of the product in accordance with GOST 3626-73 and GOST R 51464-99.

The content of vitamin C was determined by the method of capillary zone electrophoresis using a capillary electrophoresis system "Kapel' 105".

Organoleptic evaluation of dry rose hips was carried out according to such indicators as taste, color, smell and texture. The maximum score for each of the indicators was 5 points. The evaluation was carried out by a tasting committee consisting of 4 people. The arithmetic mean of all 4 values was taken as the final value.

4 Results

According to the results of the analysis, the initial moisture content of rose hips was 67.13%, and the concentration of vitamin C was 4.83%.

As part of the research, the duration of the sublimation period and the drying temperature were selected.

Fig. 2 shows the graphs of changes in the relative mass of rose hips during freeze-drying at different durations of the sublimation period. The final drying temperature was 60 °C.

Table 2 shows the results of organoleptic evaluation of dried rose hips depending on the period of sublimation.

Table 3 shows the indicators of the effectiveness of freeze drying of rose hips depending on the drying temperature. In this case, the duration of the sublimation period was 5 h.

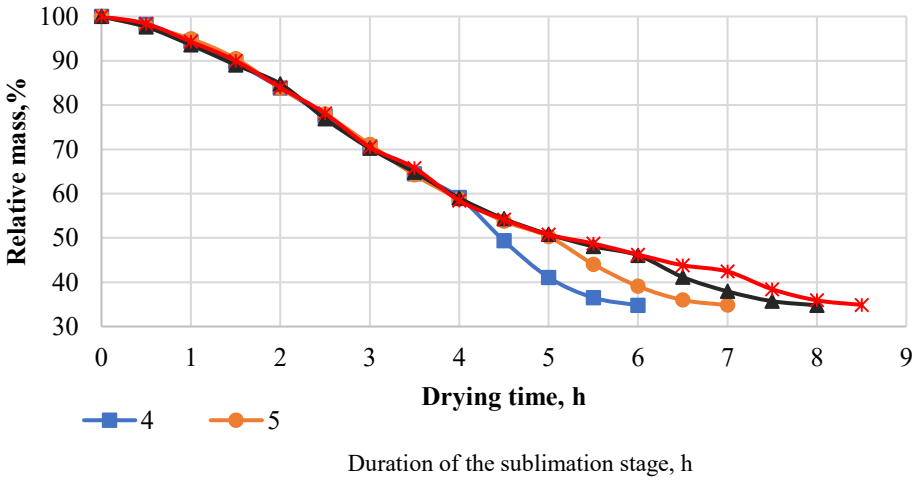

Fig. 2. Graphs of changes in the relative mass of rose hips during freeze drying with different duration of the sublimation period

Table 2. Organoleptic evaluation of dry rose hips depending on the period of sublimation.

Indicator	Duration of the sublimation period, h			
	4	5	6	7
Taste	3,75	4,25	4,5	5
Color	4,25	4,75	4,75	4,75
Smell	4	4,5	4,5	4,75
Consistency	4	4,25	5	5
Total	16	17,75	18,75	19,5

Table 3. Indicators of the rose hips drying effectiveness when selecting the final drying temperature.

Drying indicator	Final-drying temperature, °C			
	40	50	60	70
Drying time, h	9,5	8	7	6,5
Total organoleptic assessment, points	18,75	18,25	17,75	15,25
Moisture content, %	5,2	4,9	4,6	4,5
Vitamin C content, %	10,94	10,25	8,73	7,20

5 Discussion

The first stage of drying is the sublimation stage. During the first 15-30 minutes. the installation enters the operating mode: the vacuum pump then lowers the residual pressure in the drying chambers to 50 Pa. In this case, there is an intensive removal of heat from the product and it self-freezes. The relative mass at this stage changes insignificantly - by 1.5-2% (Fig. 2).

After the installation reaches the mode, the removal of most of the moisture contained in the product begins. The rate of change in the relative mass is about 10-14%/hour. 4 hours after the start of the drying process, the relative weight of the rose hips decreased to 60%.

At the third stage, the heating lamps turn on and the second stage of drying begins - final drying. After turning on the heating lamps, there is a noticeable jump in the rate of moisture removal. Moreover, the earlier the heating lamps turn on, the more dramatically the drying speed changes.

It is worth noting that the duration of the sublimation stage was determined as the total duration of freeze drying (including the setting of the installation to the operating mode) until the heating lamps were turned on.

The duration of freeze drying of rose hips with a period of sublimation of 4, 5, 6 and 7 hours was 6, 7, 8 and 8.5 hours, respectively.

The analysis of the organoleptic properties of dry rose hips made it possible to establish that with a decrease in the duration of the sublimation stage, a slight decrease in quality characteristics is observed. So, with the reduction of the sublimation stage from 7 to 6 hours, the overall organoleptic assessment decreases by 0.75 points. When the duration of the sublimation period is reduced from 6 to 5 hours, the organoleptic assessment is reduced by another 1 point. The greatest decrease in the quality of rose hips (by 1.75 points) occurs when the sublimation period is reduced from 5 to 4 hours. The change in the quality of the dry product is due to the fact that with a decrease in the sublimation period, the duration of the heating lamps increases.

Based on the foregoing information, it is recommended to lyophilize the rose hips with a sublimation period of at least 5 hours. In further experiments, the sublimation period was 5 hours.

To investigate the effect of the final drying temperature, corresponding experiments were carried out. According to the results of the experiments, it was found that a change in the final drying temperature entails a nonlinear change in the duration of moisture removal (Table 3). With a change in the final drying temperature from 70 to 40 °C, the drying time increases from 6.5 to 9.5 hours. The total organoleptic assessment also increases - from 15.25 to 18.75 points out of 20.

Within the framework of the research, the effect of the final drying temperature on the preservation of vitamin C in rose hips was also analyzed. Vitamin C is one of the most unstable vitamins during storage. Significant losses of this vitamin are observed when the pulp of the product comes into contact with oxygen. Therefore, in this case, the fruits were not crushed before freeze-drying.

The negative factors contributing to a decrease in the vitamin C content are freezing and

exposure to heating lamps. According to known data, freezing has a relatively weak effect on the concentration of vitamin C. The storage of the product itself in a frozen or native state has a greater effect. With regard to the effects of heating lamps, high temperatures have a much more harmful effect than freezing. In this case, an important factor is not only the temperature, and the duration of its exposure. An increase in the final drying temperature, on the one hand, entails a reduction in the duration of dehydration, but on the other hand, an intensification of the temperature effect.

It was found that after drying, the concentration of dry matter increases, which also affects the content of vitamin C. Taking into account the increase in the concentration of dry matter, the loss of vitamin C at a final drying temperature of 40 °C was 21%. An increase in the final drying temperature to 50 °C entails an increase in vitamin C losses up to 27%. With a further increase in the drying temperature to 60 and 70 °C, the preservation of vitamin C decreases to 37 and 48%, respectively.

Based on the research results, it is possible to recommend a final drying temperature of 50°C.

6 Conclusion

In the course of the study carried out, the most favorable modes of sublimation dehydration of rose hips were determined - the temperature of additional drying is 50 °C and the duration of the sublimation stage is 5 hours. Under these modes, the duration of lyophilization is 8 hours, the total organoleptic assessment is 18.25 points out of 20, and the preservation of the vitamin C - 27%. Research results can be useful for food industry workers, technologists and researchers. A possible direction for further research may be the selection of freezing modes - self-freezing or preliminary freezing, as well as the selection of the drying layer thickness. Lyophilized rose hips can be used as an independent product, or ground into a powder for addition to other products - confectionery, dairy, bakery products, as well as biologically active additives and functional food products.

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