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TECHNOLOGY IMPROVEMENT OBTAINING POWDERED DRIED HONEY

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The object of the study is the production of powdered dried honey. The limited use of honey in the food industry is due to its physicochemical properties. Viscosity and stickiness create problems during its dosing, mixing, storage and transportation.

Honey in powder form has a high commercial potential. The great advantages of using dry honey are the reduction of storage space, ease of processing and dosing.

Honey is characterized by organoleptic and physico-chemical parameters affecting the drying process and the quality of the final product.

The resulting powdered dry honey, in compliance with the drying modes recommended by us, has retained all its useful biological properties. The water content, as well as the dry matter content in fresh and dried honey met the criteria for the composition of honey.

Consequently, reducing the water content during the drying process significantly contributes to increasing the stability of honey during storage.

The developed technology provides for reducing the drying temperature to 50 °C, which also has a positive effect on the nutritional value of the final product.

It is established that the maximum proportion of frozen moisture in the sublimation process is observed at temperatures from minus 30 °C to minus 40 °C, depending on the types of honey, and an increase in the drying temperature above 40 °C shortens the duration of the drying process, but may affect the quality of the product.

Powdered honey is in demand in the food, pharmaceutical and cosmetic industries, due to increased dosing accuracy due to the flowability of dried honey. However, it should be borne in mind that dried honey is very hygroscopic due to the presence of sugars and the amorphous state after drying

Keywords: natural honey, powdered dry honey, freeze drying, crystallization process, storage

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1. Introduction

Honey is a valuable food product with useful therapeutic and prophylactic properties. The main advantage of honey is an extremely rich chemical composition containing antioxidants, vitamins, enzymes, as well as macro- and microelements [1].

Despite the beneficial qualities of honey, its use on an industrial scale is very insignificant due to its natural properties [2]. At the same time, honey is in demand in the pharmaceutical industry [3], in the production of cosmetics [4], as well as in the food industry [5].

The limited use of honey in the food industry is due to its physicochemical properties: stickiness, high viscosity and plasticity. The sticky consistency of honey causes problems with dosing – honey sticks to the walls of the equipment, which leads to large losses and difficulty in cleaning devices [6]. An additional limitation of the use of honey is its natural crystallization process. Crystallized honey complicates further processing and processing, requires re-liquefaction, which entails additional costs [7]. Thus, the natural properties of honey create problems during its dosing, storage and transportation.

However, the production of powdered honey is still a problem for both industry and researchers. Powdered honey has a high commercial potential in the food, pharmaceutical and cosmetic industries, by expanding the possibility of use in the technological process.

Powdered dry honey obtained in compliance with technological drying regimes retains all its useful biological properties. At the same time, the influence of the parameters of different varieties of honey on drying remains unexplored. Therefore, research devoted to improving the technology of obtaining powdered dried honey are scientific relevant.

2. Analysis of literature data and problem statement

The paper [8] presents the results of studies on the production of dry honey. It is shown that various drying methods are used to obtain dry honey: spray, vacuum, drum and freeze drying. But there were unresolved issues related to the production of powdered dried honey and the influence of its physico-chemical properties on the regularity of the drying process.

Spray drying is a fairly common method [9]. Such a method, in addition to removing water (its main purpose), microcapsulates substances sensitive to sharply changing environmental conditions, as a result of which the resulting product can retain valuable properties. This is very important in the case of honey, which contains biologically active compounds. A comparative study of vacuum drying and freeze drying of different varieties of honey shows that the dried product has similar characteristics in quality and composition, regardless of the drying method [10].

However, according to the conducted research, some preference should be given to dry honey obtained as a result of freeze-drying. Compared with honey dried in vacuum, freeze-dried honey, i.e. with pre-freezing, had a well-preserved and less damaged structure [11]. To obtain dry honey, the authors used [12] a microwave vacuum dryer. The drying temperature varied from 30 °C to 50 °C until the moisture content was below 2.5 %. The disadvantage of this method is changes in the content of basic sugars (fructose, glucose, maltose and sucrose), as well as alcohols and esters. Therefore, the choice of the drying method and temperature has a significant impact on the drying kinetics, drying speed, effective moisture diffusion and quality parameters.

To assess the quality of dry honey powder its organoleptic and physico-chemical parameters were determined.

It has been established that honey powder has the following organoleptic characteristics: color – from white to creamy yellow; aroma – pleasant, natural, but quite strong, without odors unusual for honey; taste – pleasant, sweet, without unusual flavors; consistency – non-hygroscopic, after crystallization it is dense, but remains in the form of powder; crystallization – from fine-grained to coarse-grained; mechanical impurities – natural (presence of pollen particles); there should be no signs of fermentation.

When determining the physico-chemical parameters of dry honey, the following values were obtained: diastase number – 6 units. Goth, the total acidity is 2.4 cm^3. The authors studied the effects of drying parameters on the determination of the content of minerals (calcium, potassium, phosphorus, sodium, iron, copper, zinc) and toxic elements (cadmium, cobalt, lead, arsenic). It has been established that the obtained honey powder complies with regulatory and hygienic safety requirements [13].

Studies of the kinetics of drying and the effectiveness of removing moisture from natural honey have shown that vacuum drying with pre-sublimation of honey reduces the activity of water, which increases microbiological stability during storage and reduces the possibility of fermentation of honey. The water activity in dried honey ranged from 0.405 to 0.427 [14]. However, in this work, the drying temperature reached over 50 °C, which negatively affected the quality of honey.

Despite the existing difficulties of obtaining, dried honey is in demand in the confectionery industry for the production of certain types of sweets, as well as in the baking industry to replace sucrose [15–17].

Consequently, the conversion of raw honey into powder makes it possible to increase its use in the food industry. However, the issues of the influence of rheological parameters of different varieties of honey on the drying process remained unresolved. Despite numerous studies conducted in the field of honey drying, the exact determination of the parameters of the drying process of biological material remains difficult due to the large spread of physico-chemical parameters depending on the variety, geographical and climatic conditions of collection [12].

All this suggests that it is advisable to conduct research on improving technologies for obtaining powdered dried honey.

3. Aim and objectives of the study

The aim of this study is to improve technologies of obtaining powdered dried honey. This will increase the stability of dried honey during storage while maintaining rheological.

To achieve this aim, the following objectives were solved:

– to conduct a comparative analysis of the physico-chemical properties of honey varieties: sweet clover, mountain and sunflower;

– to establish rational parameters of technologies for obtaining powdered dried honey.

4. Materials and methods of research

Varieties of honey were selected as objects of research: sweet clover, mountain and sunflower from apiaries of the East Kazakhstan region.

Sunflower honey was gently crystallized, with a characteristic intense yellow color. Honey of the “Donnik” variety was a gently crystallized state of light yellow color. Honey of the “Mountain” variety was in a liquid state with a dark brown tint.

The main hypothesis of the study is the possibility of establish rational parameters of honey drying. The honey varieties in question have approximately the same humidity, but differ significantly in viscosity. To simplify the drying process, all samples had the same layer thickness during drying and pre-freezing.

The physico-chemical parameters of honey were determined in accordance with regulatory documents [18].

The mass fraction of water in honey was measured by a refractometric method using a digital refractometer model SNEL-104.

The mass fraction of reducing sugars and sucrose is determined by the colorimetric method, calculating the optical density of a solution of potassium ferrocyanide after it reacts with reducing sugars of honey.

The diastase number on FEK-3 was measured by the method of colorimetric determination of the amount of substrate cleaved under the conditions of an enzymatic reaction, and its subsequent calculation.

Total acidity was determined by titrometric method.

To assess the content of hydroxymethylfurfural (GMF), the Selivanov-Fig method was used, based on the formation in an acidic medium of the product of the interaction of GMF with resorcinol colored cherry red, with a positive reaction and a content of GMF of at least 25.0 mg/kg.

Studies to determine the technological modes of freeze drying of honey were carried out on the ALPHA 1-2 LDplus laboratory unit. Freezing of the studied honey samples was carried out at temperatures: –20 °C, –30 °C, –40 °C, –50 °C. The freezing temperature was not lowered below –50 °C, due to the fact that honey loses its healing properties when exposed to temperatures below minus 50 °C.

To optimize the sublimation temperature, changes in the relative mass of honey and the drying rate at a certain temperature and layer thickness of 2 mm were studied. The freeze-drying temperature was varied from – 5 to –45 °C in increments of 5 degrees.

5. The results of the study of obtaining powdered dried honey

5. 1. Comparative analysis of physico-chemical properties of honey varieties: sweet clover, mountain and sunflower

The main hypothesis of the study was to obtain dry honey without reducing the biological value. The main assumptions in the work are that the surface and internal temperatures are the same during vacuum and drying.

To determine the quality and naturalness of honey, physicochemical parameters were studied: the mass fraction of water, reducing sugars and sucrose, diastase number, total acidity and the content of oxymethylfurfural. The results of the studies are presented in Table 1.

Table 1

Physico-chemical parameters of honey: sunflower, sweet clover and mountain

The name of the indicator	Honey variety			Requirements according to gost
	Sunflower	Donnik	Mountain	
Mass fraction of water, %, no more	15.06	15.26	16.04	20
Mass fraction of reducing sugars, %, not less	98.49	98.91	97.12	65
Mass fraction of sucrose, %, no more	1.38	0.10	0.20	5
Diastase number of Gote units, at least	22.41	8.17	13.36	8
Free acidity, meq/kg, no more	27	26	25	40
Mass fraction of GMF, million-1 (mg/kg), no more	12.57	22.91	24.34	25

It is established that all 3 varieties of honey under consideration comply with regulatory requirements.

One of the main indicators of the physico-chemical state of honey are viscosity, humidity and temperature. The changes in viscosity indices from temperature and humidity are studied. The results of the study of the effect of temperature on viscosity are presented in Table 2.

Table 2

Influence of temperature on viscosity

Honey variety	Viscosity at 20 °C, MPa x s	Viscosity at 35 °C, MPa x s
Donnik	184.5	27.9–>(liquid)
Mountain	114.3	25.6–>(liquid)
Sunflower	359.8	32.5–>(liquid)

The results of the study of the effect of humidity on the viscosity of honey are presented in Table 3.

Table 3

Effect of humidity on viscosity

Honey variety	Honey Humidity, %	Viscosity, MPa x s
Donnik, sample No. 1	15.1	184.5
Sample No. 2	19.3	74.7
Sample No. 3	21.3	37.3
Mountain, sample No. 1	16.3	114.5
Sample No. 2	17.9	59.2
Sample No. 3	19.9	31.8
Sunflower, sample No. 1	15.3	359.8
Sample No. 2	19.9	129.5
Sample No. 3	21.7	67.2

5. 2. To establish rational parameters of technologies for obtaining powdered dried honey

As is known, freeze drying technology includes two main stages: pre-freezing of dried materials and subsequent removal of the frozen part of moisture in vacuum by ice-steam phase transition. At the same time, the level of preservation of the native properties of the preserved objects crucially depends on the methods and modes of pre-freezing, on the temperature level, the removal of moisture by sublimation, as well as the drying temperature of the drying object.

Preliminary samples of frozen honey samples under study at temperatures: –20 °C, –30 °C and –40 °C are shown in Fig. 1.

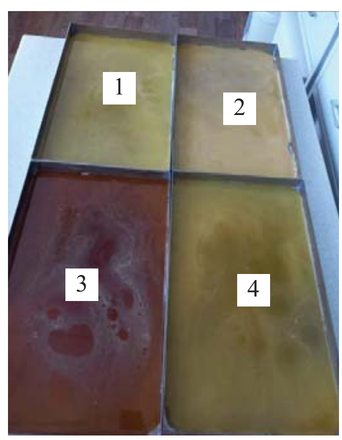

Fig. 1. Frozen honey samples: 1, 4 – sunflower; 2 – sweet clover; 3 – mountain

The frozen samples were then freeze-dried. The freeze drying process was carried out in two stages: at the first stage, under conditions of temperature and pressure below the cryohydrate point, the crystallized moisture is sublimated into the environment, while most of the moisture is removed from the product – 80–90 %. At the second stage, heat is applied to the product (up to a temperature of 40–50 °C), thereby removing the remaining moisture.

The results of experimental studies of changes in the relative mass of honey from time to time under different temperature conditions of freeze drying, for example, the sunflower variety are shown in Fig. 2.

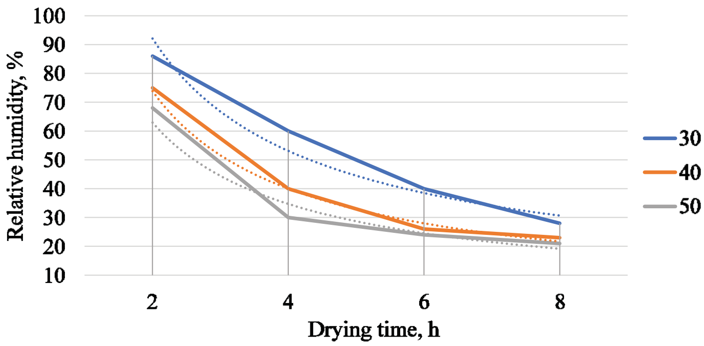

Fig. 2 The change in the relative mass of honey from time to time under different temperature conditions of freeze drying

Other varieties of honey had a similar character of the relative weight of honey from time to time under different temperature conditions of freeze drying.

The obtained results showed that the freeze-drying process for all types of bee honey with an initial sample temperature at –40 °C has a minimum drying duration (from 7 to 9 hours), despite a decrease in the intensity of the drying process in the initial period.

The curves of changes in the relative mass of honey from time to time under different temperature regimes of freeze drying are described by the equations:

$$y1=92.103x^{-0.794},\ R^2=0.9608, \tag{1}$$

$$y2=73.844x^{-0.884},\ R^2=0.9965, \tag{2}$$

$$y3=62.907x^{-0.857},\ R^2=0.9729, \tag{3}$$

where $y1$ is the change in relative mass at a freeze-drying temperature of 30 °C;

$y2$ is the change in relative mass at a freeze-drying temperature of 40 °C;

$y3$ is the change in relative mass at a freeze-drying temperature of 50 °C.

The final moisture content of dried honey is presented in Table 4.

Table 4

Moisture indicators of dried honey: sunflower, sweet clover, mountain

No.	Indicator	Sunflower	Donnik	Mountain
1	Humidity, %	5.08	5.24	4.17

Fig. 3 shows samples of powdered dried honey: sunflower, sweet clover, mountain.

The drying temperature is one of the essential factors of drying. Honey samples were dried at a temperature of 40 °C. An increase in the drying temperature above 40 °C shortens the duration of the process, however, it may lead to a decrease in the quality of the product.

a *b* *c*

Fig. 3. Samples of powdered dried honey: *a* – sunflower; *b* – sweet clover; *c* – mountain

6. Discussion of the results of the study of the physico-chemical and rheological properties on the regularities of the honey drying process

Based on the conducted studies, it was found that honey samples selected for freeze drying are high-quality and safe products (Table 1). The results obtained indicate that all the studied honey samples meet the regulatory requirements in terms of physico-chemical parameters [19].

The mass fraction of water in honey is an important indicator in the examination of honey. It was found that in the studied samples (Table 1) the mass fraction is less than 20 %.

Dynamic viscosity is highly dependent on temperature. It is an important criterion for the packaging and dosing of honey. Analysis of the data obtained (Table 2) shows a sharp decrease in viscosity with increasing temperature. It has been found that some varieties, for example sunflower, when the temperature increases by 15 °C (from 20 °C to 35 °C), the viscosity decreases more than 10 times – from 359.8 to 32.5 MPa x. However, the viscosity of the Gorny variety decreases to a lesser extent – from 114.3 to 25.6 MPa x, that is, more than 4 times.

It is established that honey, being a non-Newtonian liquid at temperatures up to 30–35 °C, but with an increase in temperature above 35 °C, noticeably changes its rheological parameters, its flow becomes close to Newtonian and practically does not depend on the deformation rate.

The viscosity of honey is greatly influenced by its humidity. From the results obtained (Table 3) it follows that with increasing humidity for all types of honey, the viscosity decreases.

Graph analysis (Fig. 1) changes in the relative humidity of honey at drying temperatures of 30 °C, 40 °C and 50 °C, the greatest loss of moisture is observed in the first 4 hours (a steep section of the curve), then the drying process proceeds less intensively (a gradual decrease in relative weight). According to the authors of paper [8], low humidity values are necessary for the stability of powders and prevention of re-sticking of powders. A comparison of the curves of dependence at different drying temperatures shows that the shortest drying time is observed at 50 °C for all types of honey. However, it is not recommended to raise the temperature above 50 °C due to the possibility of destroying the native properties of honey. Since the shortest duration of the freeze drying process is the main criterion, let's choose a temperature for the honey sublimation stage equal to 50 °C.

At the same time, it was found that solid varieties of honey with lower humidity during freeze-drying at the outlet had higher humidity values compared to liquid varieties (Table 4). It was also found that dried powdery samples from solid varieties of honey lost flowability during storage due to the adhesion of powdery particles of dried honey, in contrast to dried powdered samples from liquid honey varieties. Therefore, additional temperature treatment was required.

One of the essential factors of the drying process is the drying temperature. Otherwise, the dried honey samples stick together again due to high hygroscopicity. Honey samples were dried at a temperature of 40 °C. An increase in the drying temperature above 40 °C shortens the duration of the process, but this may lead to a decrease in the quality of the product. Due to the fact that it is important for us to preserve the healing properties of bee honey, let's opt for a drying temperature of 40 °C.

The freezing temperature was not lowered below – 50 °C, due to the fact that honey loses its healing properties when exposed to temperatures below minus 500 C.

The hypothesis of the research was to optimize the parameters of the freeze-drying temperature for the studied types of honey, at which the drying time will be minimal. At the same time, it is important to take into account that the dried honey samples retain high quality indicators, as well as preserve the biological and nutritional value of natural honey as much as possible.

Based on the research conducted to improve the technological parameters of freeze drying, it was established that the rational values of the drying process for all types of honey are: freezing temperature of honey – minus 40 °C; freeze drying temperature – 50 °C; drying temperature – 40 °C. However, it should be taken into account that dried honey is very hygroscopic due to the presence of sugars and the amorphous state after drying [8]. The drying process reduces the hygroscopicity of dry honey. Meanwhile, it should be noted that these recommendations are typical for these types of honey and climatic conditions of their production. Rheological characteristics of different varieties vary significantly in many respects, depending on the honey collection.

The main advantage of the obtained dry honey is that it has a high flowability, which ensures the accuracy of dosing and uniform mixing when used in food products, also facilitates storage and transportation.

To improve the technology of obtaining powdered dried honey, the process temperature parameters for the studied types of honey were determined, at which the drying time is minimal – up to 6 hours (Fig. 1). At the same time, it is important to take into account that the dried honey samples are products with high quality indicators, and also preserve the biological and nutritional value of natural honey as much as possible. Analysis of the research results showed that powdered dried honey of the "mountain" variety has the best indicators (humidity, flowability). That is, more liquid varieties of honey, for example, "mountain", under the same drying conditions, are dried better compared to solid ones, like "Donnik". Dried powdered honey varieties "Donnik" and "sunflower" during storage are prone to loss of flowability due to sticking. According to our hypothesis, before drying, solid varieties of honey should be subjected to additional processing to reduce viscosity. The main advantage of the resulting dry honey is that it has a high flowability, which ensures the accuracy of dosing and uniform mixing when used in food products, also facilitates storage and transportation.

Meanwhile, regularities requiring additional research have been established: the higher the moisture content of honey before drying, the higher the drying efficiency; dried powdered honey from hard varieties loses flowability during prolonged storage due to particles sticking together, unlike liquid varieties. Most likely, this is due to the fact that the efficiency of heat exchange in a liquid medium is higher than in a solid one.

7. Conclusions

1. Physico-chemical parameters of honey samples of varieties: sunflower, sweet clover, mountain have been studied. The results obtained indicate that all the studied honey samples comply with regulatory requirements. The mass fraction of water in all honey samples is less than 20 %. The highest humidity has honey of the "mountain" variety – 16.04 %. When the temperature of honey increases from 20 °C to 35 °C, the viscosity decreases by an order of magnitude. For example, for the sunflower variety, it is reduced from 359.8 to 32.5 MPa×s.

2. It was found that the optimal: freezing temperature of honey – minus 40 °C; freeze-drying temperature – minus 35–40 °C; drying temperature 40 °C. For example, for the Gorny variety, the moisture content decreased from 16.04 % to 4.17 %. Consequently, reducing the water content during the drying process significantly contributes to increasing the stability of honey during storage. The resulting powdered dry honey has a high flowability and a long shelf life. Powdered honey is in demand in the food, pharmaceutical and cosmetic industries, due to increased dosing accuracy due to the flowability of dried honey.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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Data availability

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