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## DETERMINATION OF THE PARAMETERS OF FREEZE-DRYING HONEY

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*The article presents the results of studies of freeze-drying of various types of honey. The purpose of the research was to determine the optimal parameters for the freeze-drying of honey. The physical and chemical parameters of three types of honey produced in the East Kazakhstan region were previously studied. The results showed that the studied prototypes meet the regulatory requirements for the quality of natural honey. It has been established that in terms of moisture content, sunflower honey differs from mountain honey and sweet clover (15%, 17.3 and 17.2%, respectively), and also contains a high amount of reducing sugars (87.9%), however, the mass fraction of sucrose is 2.5 %. It is noted that the highest content of sucrose in mountain honey is 4.5%. The diastase number is an indicator of the quality and naturalness of honey and, according to the requirements of GOST, should be at least 8 Gote units, the results show that the studied samples of honey contain from 13.1 to 15.1 units, which confirms the influence of the geographical origin of honey. According to the value of water activity, all types of honey are products with low humidity and long-term storage. The optimal technological modes of freeze-drying of honey have been established, which range from minus 30°C to minus 40°C degrees.*

**Keywords:** honey, freeze drying, technological parameters, temperature, humidity, sublimation.

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## БАЛДЫ СУБЛИМАЦИЯЛЫҚ КЕПТІРУ ПАРАМЕТРЛЕРІН АНЫҚТАУ

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Мақалада балдың әртүрлі түрлерін сублимациялық кептіруді зерттеу нәтижелері келтірілген. Жүргізілген зерттеулердің мақсаты балды сублимациялық кептірудің оңтайлы параметрлерін анықтау болды. Шығыс Қазақстан облысында өндірілетін балдың үш түрінің физикалық-химиялық көрсеткіштері алдын ала зерттелді. Нәтижелер зерттелген тәжірибелік үлгілер табиғи балдың сапасына қойылатын нормативтік талаптарға сәйкес келетіндігін көрсетті. Құрамына балының ылғал мөлшері тау мен түйежоңышқадан (сәйкесінше 15%, 17,3 және 17,2%) ерекшеленетіні анықталды, сонымен қатар азайтатын қанттың (87,9%) жоғары мөлшері бар, бірақ сахарозаның массалық үлесі 2,5% құрайды. Тау балындағы сахарозаның ең жоғары мөлшері 4,5% құрайды. Диастаз саны балдың сапасы мен табиғилығының көрсеткіші болып табылады және МС талаптарына сәйкес кемінде 8 Гот бірлігі болуы керек, алынған нәтижелер бойынша зерттелген бал үлгілерінің құрамында 13,1-ден 15,1 бірлікке дейін бар екендігі байқалады, бұл балдың географиялық шығу тегінің әсерін растайды. Судың белсенділігі тұрғысынан балдың барлық түрлері ылғалдылығы төмен және ұзақ сақталатын өнімдерге жатады. Балды сублимациялық кептірудің оңтайлы технологиялық режимдері орнатылды, олар минус 30С-тан минус 40С градусқа дейін.

Негізгі сөздер: бал, сублимациялық кептіру, технологиялық параметрлер, температура, ылғалдылық, сублимация.

## ОПРЕДЕЛЕНИЕ ПАРАМЕТРОВ СУБЛИМАЦИОННОЙ СУШКИ МЕДА

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В статье представлены результаты исследований сублимационной сушки различных видов меда. Целью проводимых исследований было определение оптимальных параметров сублимационной сушки меда. Предварительно были изучены физико-химические показатели трех видов меда, производимого в Восточно-Казахстанской области. Результаты показали, что исследуемые опытные образцы соответствуют нормативным требованиям, предъявляемым к качеству натурального меда. Установлено, что по содержанию влаги подсолнечниковый мед отличается от горного и донника (15%, 17,3 и 17,2% соответственно), а также содержит высокое количество редуцирующих сахаров (87,9%), однако массовая доля сахарозы составляет 2,5%. Отмечено, что самое высокое содержание сахарозы в горном меде и составляет 4,5%. Диастазное число является показателем качества и натуральности меда и согласно требованиям ГОСТа должно быть не менее 8 единиц Готе, по полученным результатам видно, что исследованные образцы меда содержат от 13,1 до 15,1 единиц, что подтверждает влияние географического происхождения меда. По значению активности воды все виды меда относятся к продуктам с низкой влажностью и длительного хранения. Установлены оптимальные технологические режимы сублимационной сушки меда, которые составляют от минус 30°С до минус 40°С градусов.

Ключевые слова: мед, сублимационная сушка, технологические параметры, температура, влажность, сублимация.

### *Introduction*

#### **Justification of the choice of the article and goal and objectives**

Honey is a food that consumers associate with nature and ecology, valued for its complex content and considered functional food [1].

Honey is a sweet product with a pleasant aroma, produced by bees from the nectar of flowers, honeydew, or a mixture of these sugary liquids. The composition of honey is very complex: it contains about a hundred different components valuable for the body - glucose, levulose, sucrose, vitamins, microelements, mineral, hormonal, antibacterial and other substances [2, 3, 4]. Despite the value of honey, only a small amount of it is used on an industrial scale [5].

Honey in its natural form has a number of disadvantages due to its high viscosity and density, which cause difficulties in transportation and dosage [6,7]. Also, honey can change its properties as a result of crystallization, which can contribute to the development of osmophilic yeast and fermentation.

Various methods have been developed for drying honey: spray drying, vacuum drying and freeze drying.

Freeze drying (lyophilization) is one of the most progressive and efficient drying methods in the food industry and allows you to get finished products that are as close as possible in terms of quality to native raw materials [8,9, 10].

Freeze drying makes it possible to preserve to the maximum extent not only the chemical composition of the product, but also its rheological properties [11]. Quantitative changes in the chemical composition of sublimated products are minimal.

Due to the lack of conditions for the development of microorganisms, freeze-dried products are the least susceptible to microbiological spoilage. The quality of sublimated products depends on many factors: the physical and chemical composition of the product itself, the technological modes of dehydration, the type of packaging, etc[12,13].

Thus, the development of technology for freeze-drying honey is an urgent task.

#### ***Materials and Research Methods***

The physicochemical parameters of honey were determined in accordance with the regulatory documents. (GOST 19792-2017) [14]

The mass fraction of water in honey was determined by the refractometric method using a digital refractometer model SNEL-104[3].

The mass fraction of reducing sugars and sucrose - by colorimetric method, calculating the optical density of a solution of potassium ferricyanide after it reacts with the reducing sugars of honey[4].

The diastase number was measured on FEK-3 by the method of colorimetric determination of the amount of the substrate cleaved under the conditions of the enzymatic reaction, and its subsequent calculation[15,4].

The total acidity was determined by the titrimetric method.

To assess the content of hydroxymethylfurfural (HMF), the Selivanov-Fige method was used, based on the formation in an acidic medium of the product of the interaction of HMF with resorcinol, painted in cherry red, with a positive reaction and the content of HMF not less than 25.0 mg/kg.

Determination of water activity (Aw) in the studied samples of honey was carried out using a portable high-speed device AquaLab PAWKIT Meter (USA)[16].

Studies to determine the technological modes of freeze-drying of honey were carried out on a laboratory unit ALPHA 1-2 LDplus

#### ***Results and their discussion***

The object of research was honey from apiaries of the East Kazakhstan region of three types: sunflower, sweet clover with sainfoin and mountain flower. To determine the quality and naturalness of honey, a study of physical and chemical indicators was carried out: mass fraction water, reducing sugars and sucrose, diastase number, total acidity and hydroxymethylfurfural content.

The research results are presented in table 1.

Table 1 Physical and chemical indicators

Name of indicator	Type of honey			Requirements according to GOST
	Sunflower	Sweet clover with sainfoin	mountain flower	
Mass fraction water, %, no more	15,0	17,2	17,3	20
Mass fraction reducing sugars, %, at least	87,9	86,5	83,3	65
Mass fraction sucrose, %, no more	2,5	3,9	4,5	5
diastase number units Gotha, at least	14,6	15,1	13,1	8
General acidity, cm <sup>3</sup> NaOH, no more	2	3	2	1-4
Quality response to HMF	negative	negative	negative	negative

The results obtained indicate that all the studied samples of honey meet the regulatory requirements GOST.

The mass fraction of water in honey is an important indicator in the examination of honey. Honey containing less than 20% free water is not fermented by yeast. Honey with a moisture content of more than 21% turns sour at lower or higher temperatures [8-17]. In the studied samples of three types of honey, the mass fraction of water ranged from 15.0% to 17.3%, which does not exceed the permissible limits. The diastase number of Gote units in honey shows its quality and naturalness and must be at least 8, and the higher its number, the better the honey. In the studied samples of honey: sunflower, donik with exaracet and mountain, the diastase number was 14; 15.1 and 13.1 Gote units, which confirms its high quality and naturalness. The mass fraction of sucrose ranged from 2.5 to 4.5%, while its smallest amount is noted in sunflower honey and the largest in mountain honey. To determine the quality and maturity of honey, we studied the content of reducing sugars, which consist of glucose and fructose. If this indicator is below 80%, then it is considered that the honey was subjected to heat treatment or falsified honey. In our experimental samples, the mass fraction of reducing sugars ranged from 83.3 to 87.9%, which confirms the originality and quality of honey. [8-17]. The results of the study on the content of hydroxymethylfurfural in honey samples showed a negative reaction, which means the absence of this carbohydrate and confirms the quality of the product.

However, the moisture index does not reflect the entire complex of interactions that are

present in the food product and in which water is a participant. At the same time, there is the “water activity” indicator, which is the main criterion for characterizing the state of water in food products and is widely used throughout the world, both to predict the technological properties of products, and the quality of the food product and its shelf life [18,19,20] Water activity itself is a dimensionless quantity and is defined in the range from 0 to 1.

Water activity is one of the most critical parameters in determining the quality and safety of products that are consumed every day. Water activity affects the shelf life, safety, structure and smell of foods. [18,20,21]

Considering that water activity is important for monitoring the safety and quality of honey, we conducted studies to determine the water activity for the studied types of honey (Table 2) [22, 23].

The water activity of honey water depends mainly on the content of fructose and glucose. During crystallization, glucose begins to crystallize first. Fructose has a higher solubility and stays in solution longer. All five hydroxyl groups of glucose interact with water molecules. After crystallization, glucose is found in the form of glucose monohydrate, each glucose molecule fixes only one molecule of water. Consequently, less water is fixed in the crystallized state. The free water content is higher and corresponds to the water activity.

The type of honey has different ratios of fructose and glucose. Therefore, glucose crystalli-

zation has a stronger effect on those types of honey that contain relatively more glucose. The dif-

ference between the water activity of different types of honey lies in its varied sugar composition.

Table 2 Water activity in honey

Type of honey	Water activity ( $A_w$ )			
	$A_{w1}$	$A_{w2}$	$A_{w3}$	$A_{w\text{среднее}}$
Sunflower	0.54	0.56	0.56	0.55
Sweet clover with sainfoin	0.48	0.55	0.55	0.52
Mountain flower	0.57	0.59	0.58	0.58

Water activity in honey was in the range of 0.5-0.58, the temperature of honey samples was  $23.2 \pm 0.20^\circ\text{C}$ . The data obtained show that all types of honey in terms of water activity (0.55; 0.52; 0.58) are products with low moisture content. Thus, honey samples are safe products, protected from microbiological spoilage. This is due to the fact that with a decrease in water activity, the possibility of using moisture for the metabolism of microorganisms decreases [3,9].

Based on the research, it was found that honey samples selected for freeze-drying are high-quality and safe products.

As you know, the technology of freeze drying includes two main stages: preliminary freezing of the materials to be dried and subsequent removal of the frozen part of the moisture in vacuum by the ice-vapor phase transition. At the same time, the level of preservation of the native properties of preserved objects depends decisively on the methods and modes of preliminary freezing, on the temperature level, removal of moisture by sublimation, and the temperature of drying the drying object [24,25, 26].

Studies to determine the technological modes of freeze-drying of honey were carried out on a laboratory unit ALPHA 1-2 LDplus (Figure 1).



Figure 1 - ALPHA 1-2 LDplus laboratory setup

Preliminary freezing of the studied honey samples was carried out at temperatures:  $-20^\circ\text{C}$ ,  $-30^\circ\text{C}$  and  $-40^\circ\text{C}$ . The results obtained showed that the process of freeze drying for all types of bee honey from the initial sample temperature of  $-40^\circ\text{C}$  has a minimum drying time (from 7 to 9 hours), despite the decrease in the intensity of the drying process in the initial period [27].

Then the frozen samples were subjected to freeze drying. The process of freeze drying is carried out in two stages: at the first stage, under conditions of temperature and pressure below the cryohydration point, the crystallized mois-

ture is sublimated into the environment, while most of the moisture - 80 - 90% - is removed from the product. At the second stage, heat is supplied to the product (up to a temperature of  $40 - 50^\circ\text{C}$ ), thereby removing the remaining part of the moisture [28,29].

To select the sublimation temperature, a study was made of the change in the relative mass of honey and the drying rate over time at a certain temperature and a layer thickness of 10 mm. The freeze-drying temperature was varied from  $-5$  to  $-45^\circ\text{C}$  in increments of 5 degrees (Figure 2)[23]. The freezing temperature was

not reduced below  $-50^{\circ}\text{C}$ , due to the fact that honey, when exposed to temperatures below

minus  $50^{\circ}\text{C}$ , loses its healing properties [9].

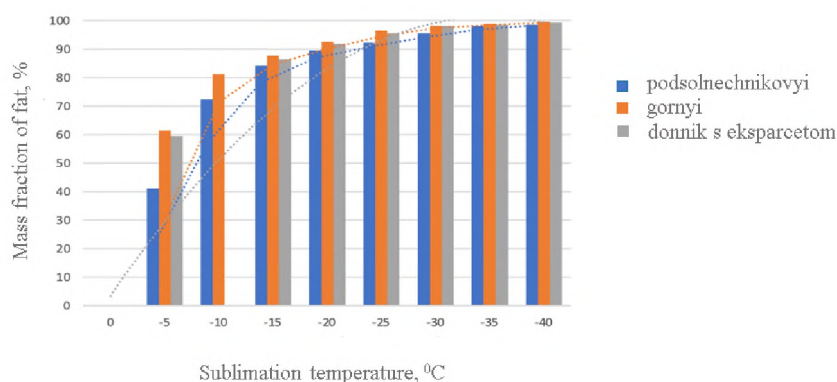


Figure 2 – The dependence of the amount of moisture removed from the sublimation temperature

The obtained research results showed (Figure 2) that the maximum proportion of frozen moisture in the process of sublimation is observed at temperatures from minus  $30^{\circ}\text{C}$  to minus  $40^{\circ}\text{C}$ , depending on the types of honey [30].

The drying temperature is one of the essential drying factors. Honey samples were dried at a temperature of  $40^{\circ}\text{C}$ . Increasing the post-drying temperature above  $40^{\circ}\text{C}$  can lead to a decrease in the quality of the product, but this reduces the duration of the process.

When carrying out freeze drying, it is necessary to determine such parameters of the process temperature for the studied types of honey, at which the drying time will be minimal [24]. At the same time, it is important to take into account that dried honey samples are products with high quality indicators, as well as retaining the biological and nutritional value of natural honey as much as possible [31].

### Conclusions

Three types of honey from the Eastern region of Kazakhstan were selected for freeze-drying: sunflower, sweet clover with sainfoin and mountain flower. To control the naturalness and safety of honey samples, a study of their physical and chemical indicators was carried out: mass fraction water, reducing sugars and sucrose, diastase number, total acidity and hydroxymethylfurfural content.

The results showed that the studied prototypes meet the regulatory requirements for the quality of natural honey. It has been established that in terms of moisture content, sunflower honey differs from mountain honey and sweet clover

(15%, 17.3 and 17.2%, respectively), and also contains a high amount of reducing sugars (87.9%), however, the mass fraction of sucrose is 2.5 %. It is noted that the highest content of sucrose in mountain honey is 4.5%. The diastase number is an indicator of the quality and naturalness of honey and, according to the requirements of GOST, should be at least 8 Gote units, the results show that the studied samples of honey contain from 13.1 to 15.1 units, which confirms the influence of the geographical origin of honey.

According to the value of water activity, all types of honey are products with low humidity and long-term storage.

The results obtained indicate that all the studied samples of honey meet the regulatory requirements and confirm its high quality and naturalness.

All types of honey samples in terms of water activity (0.55; 0.52; 0.58) are products with low moisture content. Thus, honey samples are safe products, protected from microbiological spoilage[3].

When selecting the technological parameters of freeze-drying, it was determined that the optimal values of the drying process for all types of honey are: honey freezing temperature - minus  $40^{\circ}\text{C}$ ; freeze-drying temperature - minus  $35-40^{\circ}\text{C}$ ; drying temperature  $40^{\circ}\text{C}$ .

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