European Online Journal of Natural and Social Sciences 2018; www.european-science.com
Vol.7, No 2 pp. 460-470
ISSN 1805-3602

Freeze Drying Of Sheep Milk: Calculating Process Duration

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Received for publication: 27 November 2017.
Accepted for publication: 28 April 2018.

Abstract

We have established the extremes for different forms of moisture binding as a parameter, characterizing the safety and quality of sheep milk products. Monomolecular adsorption area for the sheep milk produced in the South Kazakhstan Region is within the following water activity interval: 0.29<aw<0.38. As for the sheep milk produced in the Almaty Region, this interval is 0.27<aw<0.35. In this case, moisture binding energy is characterized by average strength and is in the range from 3.189·103 to 2.557·103 J/mole.

Polymolecular adsorption area for the sheep milk produced in the South Kazakhstan Region is within the following water activity interval: 0.38<aw<0.63. As for the sheep milk produced in the Almaty Region, this interval is 0.35<aw<0.65. Moisture binding energy is in the range from 1.25·103 to 1.045·103 J/mole.

Capillary binding area for the sheep milk produced in the South Kazakhstan Region is within the following water activity interval: 0.63<aw<0.96. As for the sheep milk produced in the Almaty Region, this interval is 0.65 < aw > 0.97 In this case, moisture binding energy is similar in value to the free moisture energy – 99.44·102 - 74.19·102 J/mole depending on the kind of milk.

Keywords: Sheep Milk; Freeze Drying; Moisture Binding Forms; Water Activity; Moisture Binding Energy.

Introduction

Agribusiness 2020 Program of Agro-Industrial Development in the Republic of Kazakhstan for 2013 – 2020 provides for the creation of conditions required to increase the competitive capabilities of agribusiness entities in the Republic. Provisions cover the livestock industry – 43% of total gross output (Analytical service of Rating Agency, 2010; Agribusiness 2020, 2013).

Sheep breeding is one of the most important sectors of animal husbandry in the Republic. However, dairy market analysis has shown that Kazakh dairy products are made mainly of cow, partly fermented mare and camel milk (Analytical service of Rating Agency, 2010; Meat encyclopedia).

In the Republic of Kazakhstan, the main purpose of sheep farming is to produce meat and wool. Sheep milk, however, is not used in industrial quantities, although world experience shows a steady increase in the volume of sheep milk consumption due to its unique properties. Sheep milk contains various vitamins and microelements (Analytical service of Rating Agency, 2010; Meat encyclopedia).

Thus, sheep milk is widely used in Italy, Greece, Crimea, Transcaucasia, Central Asia and North Caucasus as a base for such dairy products as ayran, yoghurt, cheeses and butter, matzoon, and katuk.

In the framework of a thesis, we have studied the process of developing a methodology for auditing sheep milk powder production based on demand fulfillment records. Sheep milk, produced seasonally, is a highly demanded product – both the people and the processing enterprises are willing to purchase it.

In this regard, there is a good background for producing not only sheep milk, but also new dairy products made of sheep milk powder: yogurt, some types of soft cheese, ayran and katuk. The process of preparing standardization and certification documents is another pressing problem. These documents are required if the enterprise is going to meet all the requirements for product safety and quality specified by: the State *System* of the *Technical Regulation of the Republic* of *Kazakhstan*, the Customs Union, and the Eurasian Economic Union.

These documents include national standards as general requirements, methods of inspection and testing (method of measurement), and management standards.

Our review of the regulatory component has showed that there is almost a complete lack of control over the process not only in Kazakhstan, but also in CIS countries. This can be explained by the fact that we are only beginning to develop this area with the purpose of preserving national production and traditions in the single economic space (Milk processing – standardization; Myrkhalykov, 2015).

We have found that provisions of the *Customs Union Technical Regulation on Safety of Milk and Dairy Products (TR TS 033/2013)*, presented in Table 1 (Appendix to *TR TS* No. 5), describe identification indicators for raw milk provided by different farm animals, including sheep. According to these indicators, milk composition should be the following: fat (not less than 6.2%), protein (not less than 5.1%), dry substances (18.5% an average). At a temperature of 20 °C, milk density should be not less than 1034 while the acidity (°T) – not higher than 25 (*Customs Union Technical Regulation on Safety of Milk and Dairy Products*, 2013).

These data could be a basis for a sheep milk standard designed in respect to physical-chemical characteristics. We have analyzed the National Standard of the Republic of Kazakhstan ST RK 2117-2011 – KAZAKH NATIONAL DAIRY PRODUCTS. In the Section 4 titled as *Classification*, general characteristics are provided for a sub-group "...from goat and sheep milk" depending on the type of milk raw materials used in dairy production. However, there are no organoleptic and physical-chemical characteristics outlined specifically for sheep milk and sheep milk products. Sections 5.3 of the ST RK 1760-2008, ST RK 166-97 and ST RK 1005-98 provide for the requirements for raw materials used in producing national sheep milk products. Each document is devoted to a particular type of milk: cow, camel and mare milk, respectively (ST RK 166-97; ST RK 1005-98; ST RK 1760-2008).

We have also drawn our attention to the availability of National Standards that can be used to determine the organoleptic and physical-chemical characteristics of sheep. As result, we have found the following documents: ST RK 1734-2007 Milk and dairy products. Acceptance rules and test methods; ST RK 1508-2006 Radiation monitoring. The selection of milk samples and milk products. General requirements; ST RK 1732-2007 Milk and dairy products. Sensory method of determining the quality indicators; RK 1733-2007 Milk and dairy products. General technical specifications; RK 1735-2007 Milk and dairy products. Packing, marking, transportation and storage; ST RK 2152-2011 Identification of milk products. General provisions (*Normative* *documents on standardization* 2015). These Standards are taking into account the International and National Standards, and can be used while testing sheep milk and sheep milk products.

However, we believe that the process of producing such products requires a number of adopted national standards similar to ST RK 1760-2008, ST RK 166-97 and ST RK 1005-98. Our first objectives are to develop a STO Standard that can be used to identify the product, to put the ISO certificate on the application, and to provide product description. This will allow respecting the copyrighted technology and/or finished products.

As mentioned above, we propose to bring a focus on sheep milk powder since sheep milk lactation is seasonal and cannot fulfill the further demand for sheep milk products.

We have also studied the samples of raw sheep milk and sheep milk powder made of milk produced in the Almaty and the South Kazakhstan Regions. As a result, we have data on a number of sheep milk quality and safety indicators that allow drawing a conclusion about the possibility of using sheep milk powder for producing various types of dairy products. These indicators meet the requirements of the *Customs Union Technical Regulation TR TS 033/2013* (Myrkhalykov et al., 2015).

The next stage of our research is the problem of choosing the optimal drying method that would allow saving the nutritional properties of sheep milk. Moisture content in powdered milk affects this indicator, as its storage time is specified in the Finished Product Standard. If the content of moisture is above the limit, undesirable bacteria will grow. This will disrupt the quality of the product and reduce its shelf life.

In our opinion, freeze drying will allow achieving the best product quality. However, this method has significant disadvantages: long duration and great energy costs. Energy costs will be lower if we reduce the drying process duration by creating such freeze drying conditions that will allow taking into account the thermodynamic state of moisture in the product, hygroscopic state of the environment, as well as the nature and form of moisture transfer from the dried product surface. There are being created various methods to measure this parameter (process duration) (Lykov, 1950; Guygo et al., 1972; Ginzburg, 1973; Komovnikov et al., 1085; Shingisov and Chomanov, 2006). The most common method was introduced by A.V. Lykov (1950). However, his assumption about the constant nature of moisture factors limit the application of this method, as these factors do actually change with a decrease in humidity and increase in temperature. V. V. Krasnikov has introduced the method that involves the stage of generalizing drying curves into a single curve in the $N\tau - \tau$ coordinate system. However, this method does not reveal the mechanism of the process and does not take into account the conditions affecting the behavior of the product being dried. These two aspects are necessary for selecting optimal drying conditions.

Thus, out analysis of existing computational methods for calculating the freeze drying process duration shows that there are no uniform and universal computational methods. Each particular case requires an individual approach according to its characteristics. Besides, the driving force of all existing computational methods is followed from the differential: temperature Δt, pressure ΔP, concentration ΔWith, density $\Delta\rho$ etc. These are the parameters that are not fully integrated into the thermodynamic state of moisture in the product, hygroscopic state of the environment, as well as the nature and form of moisture transfer from the dried product surface.

Therefore, **the purpose of this research** is to analyze the cases of using water activity parameter as a driving force of moisture transfer in the light of the thermodynamic state of moisture in the product; to use it to measure the moisture binding energy and relative humidity as a parameter characterizing the hygroscopic state of environment; to stabilize the thermal balance; and to formulate the equation for calculating the freeze drying process duration.

According to (Shingisov and Chomanov, 2006), convective heat transfer under the pressure and temperature typical for freeze drying is very weak. It is most noticeable only at the pressure >100 Pa. At the same time, radiant heat transfer coefficient will increase with the increase in deferential temperature while the convective heat transfer coefficient remains constant. Thus, radiant heat transfer plays the main role in heat removal by sublimation during the freeze drying (Novikov and Wagner, 1969).

Thus, we can assume that the mechanism of energy exchange between the surface of the product and the environment is as follows. In the case of high moisture content, first freeze-drying cycle is characterized by the situation when distant vapor molecules begin to equalize own energy with the energy of humid air molecules to E1. Some of the E2 vapor molecules condensate on the surface releasing extra E1. Thus, we can assume that interaction between the water and the elements, as well as the effect on physical-chemical characteristics, depends not so much on the moisture content, as on its thermodynamic state (*Duckworth*, 1974; Kamerbaev, 2001; Shingisov, 2004).

Thermodynamic state of moisture affects the character of moisture transfer within and from the product surface, as well as the thermodynamic stability of the product structure.

In recent years, many researchers have dawn enough attention to the need in studying the moisture content in food in combination with intensive and extensive parameters, including water activity (a_w). The a_w value allows measuring the kinetics of the freeze drying process, as well as determining how the moisture interacts with the dried product surface. This makes it possible to calculate the drying process duration.

Theory

According to the heat balance equation formulated for freeze drying, *q* energy, extracted from or transferred to the product (convection, induction, radiation, etc.) should be equal to the $r \cdot j$ energy, extracted with weight loss (water vapor) (Guygo et al., 1972):

$$q = r \cdot j \qquad (1)$$

If we consider the drying speed under the assumption that heat transfer rate is proportional to the amount of unfrozen moisture, the heat balance equation for an infinitesimal time interval $d\tau$ will be:

$$q d\tau = (a_н - a_к)\ \rho_0 \cdot r \cdot d\delta \qquad (2)$$

Where, a_{w-} – water activity at the beginning and at the end of the drying process;

ρ_0 – dry product density.

If we apply the expression q from (2) to (1) and pass to integrals, we will get:

$$\int_0^\tau d\tau = A \cdot \int_0^\delta d\delta + C_1 \qquad (3)$$

If we take total water activity as *A*, we will get:

$$A = \frac{(a_н - a_к) \cdot \rho_0 \cdot r}{q} \qquad (4)$$

If we apply (4) to (3), we will get:

$$\tau = \frac{(a_н - a_к) \cdot \rho_0 \cdot r \cdot \delta}{r \cdot j} \qquad (5)$$

or

$$\tau = \frac{(a_н - a_к)\rho_0 \delta}{j} \tag{6}$$

Equation (6) can be applied after changing the intensity of moisture evaporation and increasing water activity during the freeze drying process.

The article (Shingisov et al., 2016) is devoted to the issues of surface evaporation through the lens of sublimation, and thermodynamic theory with due account for the surface water condition changing during the freezing and freeze-drying processes. The article provides the equation for measuring the intensity of moisture transfer from the product surface based on the difference in thermodynamic water activity and relative humidity:

$$j = 0.622 \frac{\alpha}{\mu \cdot C_p} \cdot \frac{P''_в}{B}(a_w - \varphi) \tag{7}$$

Based on (7), (6) will be as follows:

$$\tau = \frac{\rho_0 \cdot \delta \cdot \mu \cdot c_p}{0.622 \cdot \alpha_T} \cdot \frac{B}{P''_в} \cdot \ln \frac{a_н - \varphi}{a_к - \varphi} \tag{8}$$

Thus, equation (8) is an interesting solution when it comes to calculating the freeze drying process duration: dry product size (δ), heat and moisture transfer conditions (α_T), product structure impact (μ), water activity (a_w) and relative humidity (-φ).

Materials and Methods

Research material: sheep milk produced by *Ovis aries* in the South Kazakhstan and Almaty Regions.

Moisture content in the samples of sheep milk was measured by standard method for drying the product to constant weight in a convection oven ShK-80 (Russia) at 105°C.

Water activity a_w was measured with the Agualab 4 (Russia) before and after drying.

Heat capacity C_p, barometric pressure B and air density ρ_o are available from the literature.

Relative humidity φ was measured with dry and wet thermometer from the inside of the sublimation chamber.

Coefficient α_T was taken as $\alpha_T = 3$ W/(m^2·K).

Evaporative resistance coefficient μ was measured as follows: At the beginning of the experiment, sheep milk samples frozen as plates (size: 0.35x0.25 m; thickness: δ=0.004 m) were placed to the refrigerating chamber for 16 hours at a temperature of -18÷-20 ºC. Then, they were subjected to freeze drying at the temperatures of -10 °C. The drying process has been stopped every 3 hours. The samples were taken out of the machine and measured in order to determine how the water activity changes. The effect of dried product thickness on the drying process duration was studied as follows. Sheep milk samples were frozen in the same form – as plates, and under the same temperature conditions. The thickness of plates, however, varied: δ = 0.002, 0.004, 0.006 and 0.008 m. Residual pressure in the sublimation chamber was maintained in the range equal to 3524 Pa.

Changes in the moisture content were measured with the following formula:

$$W_{пр} = \frac{G_н - (G_н - G_р)}{G_н} \cdot 100\% \tag{9}$$

Where: $W_{пр}$ – – moisture content at the time of measurement,%;

$G_н$ – initial weight, g;

$G_н$– weight at the time of measurement.

Results and Discussion

Water activity index of freeze-dried sheep milk samples

The average moisture content in sheep milk, produced in the South Kazakhstan Region, amounted to W_H=86.45 %. As for the sheep milk produced in the Almaty Region, this parameter was W_H=88.44 %. These data allow us to set the X-axis limits for W_H not exceeding 90%. The Y-axis is for a_w. Based on obtained data, we have drawn the curves of a_w dependence on $W_н$ (Figure 1).

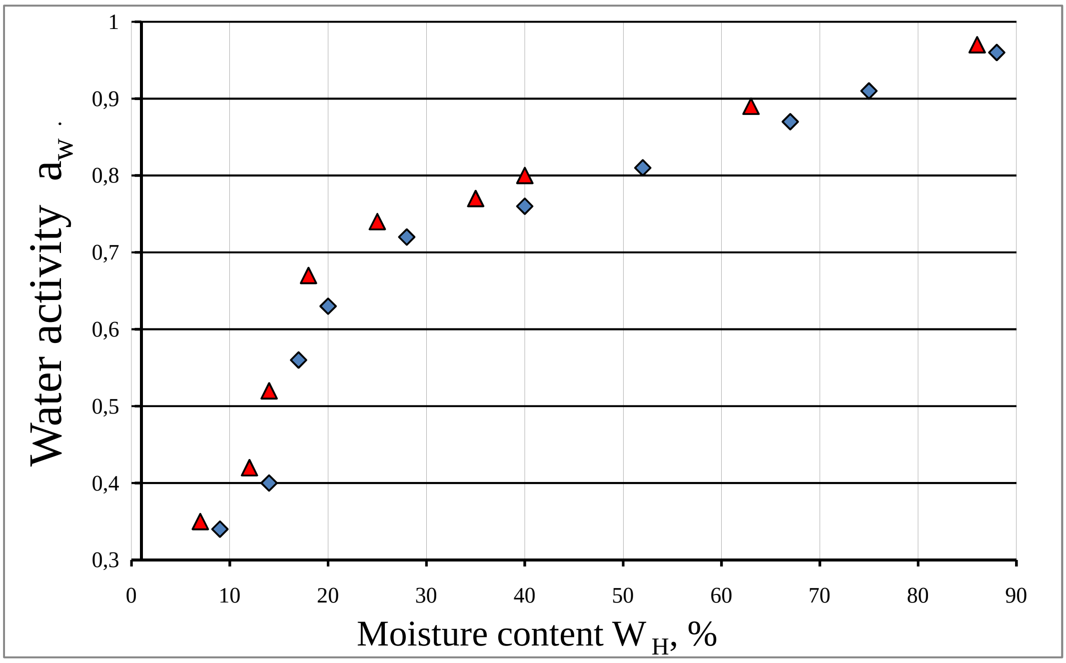

Figure 1.Water activity dependence on moisture content in the samples
Note: ∆ - sheep milk produced in the Almaty Region; ◊ - sheep milk produced in the South Kazakhstan Region.

Figure 1 shows that the curve of moisture desorption isotherms of sheep milk has a complex form. We can clearly distinguish three intervals on the aw = f (Wпр) curve with their boundary points: A and B – for sheep milk produced in the South Kazakhstan Region; C and D – for sheep milk produced in the Almaty Region.

The first interval is the monomolecular moisture adsorption that characterizes the formation of strong hydration complexes with high moisture binding energy and strict orientation of water molecules in relation to the dry product surface. These water activity intervals are in the range of 0 $<a_w<$ 0.38 (the case of sheep milk produced in the South Kazakhstan Region) and 0 $<a_w<$ 0.35 (the case of sheep milk produced in the Almaty Region).

The second interval is the multi-molecular moisture adsorption, characterized by the mobility of water molecules straining the flexible protein chains to assume the most favorable conformation. These water activity intervals are in the range of 0.38 $<a_w<$ 0.63 (the case of sheep milk produced in the South Kazakhstan Region) and 0.35 $<a_w<$ 0.65 (the case of sheep milk produced in the Almaty Region). In this interval, moisture binding energy is significantly reduced while the internal energy increases. Hence, mobility of water molecules also increases. Thus, microorganisms have better chances to cultivate.

The third interval is characterized by the diffusion of water molecules in the intermolecular space of protein complex. It corresponds to the capillary state of moisture in micro - and macro-cells. These water activity intervals are in the range of $0.63<a_w<0.96$ (the case of sheep milk produced in the South Kazakhstan Region) and $0.65<a_w<0.97$ (the case of sheep milk produced in the Almaty Region). In this interval, moisture binding energy is low due to weak bonds. This increases the flexibility of protein chains and leads to further moisture adsorption that entails the separation of these chains (diffusion).

Monomolecular layer of moisture is known to be the optimal level of moisture content when it comes to freeze drying, since moisture is firmly bound and non-reactive, and therefore, cannot be solvent. Based on the foregoing, the optimum values of water activity are: $a_w = 0.36 \div 0.38$ (at storing sheep milk produced in the South Kazakhstan Region), which corresponds to u_p= 0.32-0.36 kg in 1 kg of dry matter; and $a_w = 0.34 \div 0.35$ (at storing sheep milk produced in the Almaty Region), which corresponds to $u_p = 0.31 \div 0.34$ kg in 1 kg of dry matter.

Based on the conditional moisture distribution in milk (strongly bounded and weakly bounded), we can assume that the boundary showing the extremes of the moisture binding in sheep milk, produced in the South Kazakhstan Region, will be at a_w = 0.65. As for the sheep milk produced in the Almaty Region, this parameter is a_w = 0.63.

Studying the change in the evaporative resistance coefficient (μ) values

Freeze drying process analysis in terms of dynamics has shown that different cells (cracks, channels, capillaries) have been formed in the frozen sheep milk plates during the freeze drying. They have been formed due to the deepening of the sublimation surface. In these cells, vapor molecules can move from the sublimation area to the product surface (Figure 2).

Figure 2. Freeze drying of sheep milk

Product surface layers dehydrate due to moisture evaporation; moisture concentration decreases along with moisture evaporation entailing an increase in the value of evaporative resistance coefficient (ERC) - μ.

Research results regarding the dependence of μ on water activity are presented in Figure 3.

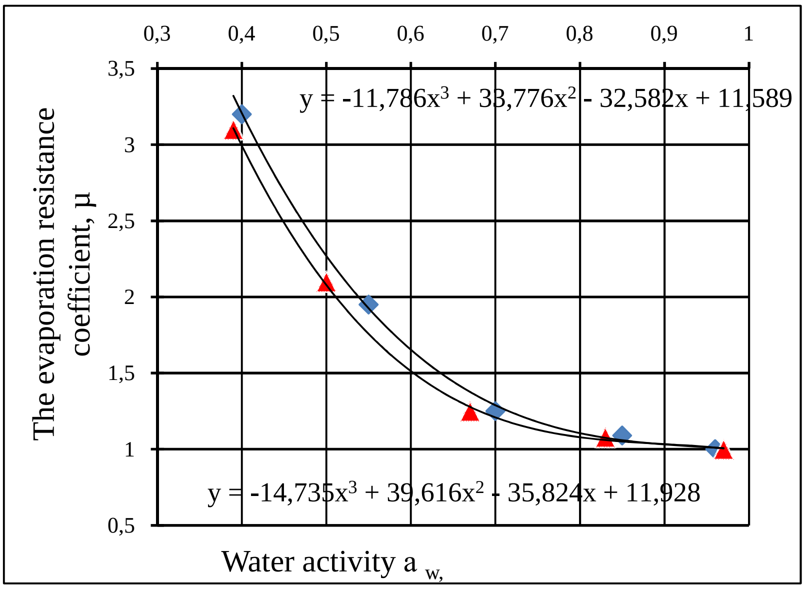

Figure 3. ERC dependence on water activity

Note: ∆ - sheep milk produced in the Almaty Region; ◊ - sheep milk produced in the South Kazakhstan Region..

Figure 3 shows that the curve of water activity regularities has a complex form. For example, if water activity intervals are in the range of $1.0 < a_w > 0.65$ (the case of sheep milk produced in the South Kazakhstan Region) and $1.0<a_w>0.63$ (the case of sheep milk produced in the Almaty Region), ERC increases monotonically. If water activity intervals are in the range of $0.38<a_w<0.65$ (the case of sheep milk produced in the South Kazakhstan Region) and $0.36 < a_w < 0.68$ (the case of sheep milk produced in the Almaty Region), ERC increases sharply. Based on these data, we can assume that weakly bounded moisture has been extracted from the sheep milk samples, which water activity intervals were in the range of $1.0 < a_w > 0.65$ (the case of sheep milk produced in the South Kazakhstan Region) and $1.0<a_w>0.63$ (the case of sheep milk produced in the Almaty Region).

There are no physical-chemical bounds in the second interval: $0.38<a_w<0.65$ (the case of sheep milk produced in the South Kazakhstan Region) and $0.36<a_w<0.68$ (the case of sheep milk produced in the Almaty Region). In this case, medium strength water configuration is formed by polar molecules attracted to each other by *dipole-dipole* attractions. In these intervals, water is bind to the casein and p-lactoglobulin micelles, the membranes of milk fat balls and free phospholipids, as well as to lactose and minerals.

Thus, we can assume that in cells, ERC value increases in 3 times an average.

Approximation of the ERC dependence on water activity is satisfactorily described by the following equation:

$$Y = A\cdot x^3 + B\cdot x^2 + D\cdot x + C \qquad (10)$$

Where: A, B, C, D, and C are constant coefficients.

The values of A,B, D and C coefficients are presented in Table 1.

Table 1. Values of A, B, D and C coefficients

Sheep milk	A	B	D	C	Approximation validity (R^2)
South Kazakhstan Region	-11.786	33.776	-32.582	11.589	0.9975
Almaty Region	-14.735	39.616	-35.824	11.928	0.9995

Studying the effect of dry sample thickness on the drying time

We have measured the drying duration of sheep milk samples with different thickness (samples were frozen as plates with the following parameters: thickness – 0.002, 0.004, 0.006 and 0.008 m; size: 0.35x 0.25 m).

The results are presented in Figure 4.

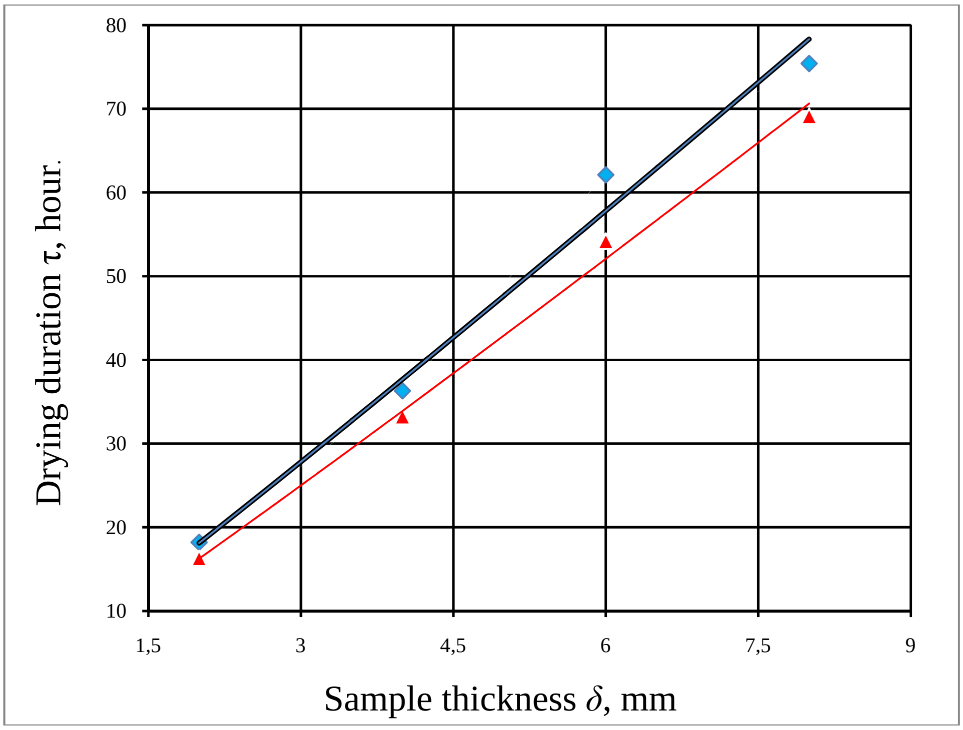

Figure 4. Product thickness impact on the drying time
Note: Δ - sheep milk produced in the Almaty Region; ◊ - sheep milk produced in the South Kazakhstan Region.

Conclusions

1. Merino sheep milk, produced in the South Kazakhstan and Almaty Regions, can be used in industrial production, since their quality and safety parameters meet the requirements of the *Customs Union Technical Regulation on Safety of Milk and Dairy Products*.
2. There should be adopted standards for raw sheep milk and sheep milk powder that would provide for the product quality and safety parameters.
3. Freeze drying method was applied to increase the shelf life of dried sheep milk as one of the significant parameters and to maximize the amount of preserved nutrients.

4.We have established that boundary points showing the extremes of moisture binding in sheep milk are located in the surface of monomolecular moisture adsorption at the water activity intervals of 0.29 $< a_w <$ 0.38 (the case of sheep milk produced in the South Kazakhstan Region) and 0.27 $< a_w <$ 0.35 (the case of sheep milk produced in the Almaty Region). In this interval, moisture binding energy is characterized by moderate strength and ranges from $3.189 \cdot 10^3$ to $2.557 \cdot 10^3$ J / mol.

Polymolecular moisture is adsorbed at the water activity intervals of $0.38 < a_w < 0.63$ (the case of sheep milk produced in the South Kazakhstan Region) and 0.35 $< a_w <$ 0.65 (the case of sheep milk produced in the Almaty Region). this interval, moisture binding energy ranges from $1.25 \cdot 10^3$ to $1.045 \cdot 10^3$ J / mol.

Capillary moisture bonds were found at the water activity intervals of $0.63 < a_w < 0.96$ (the case of sheep milk produced in the South Kazakhstan Region) and $0.65 < a_w > 0.97$ (the case of sheep milk produced in the Almaty Region). In this interval, the amount of moisture binding energy is similar to the amount of free moisture energy – from $99.44 \cdot 10^2$ to $74.19 \cdot 10^2$ J/mole, depending on the kind of milk.

This research shows that evaporative resistance coefficient μ increases in three times due to cell formation at the intervals with adsorption moisture binding.

Comparative analysis of experimental data and data calculated with the formula (8) shows that the difference between them is not more than 11%. This proves that our foundations of regularities in forming equations to calculate drying duration are valid. Based on these foundations, we have established the drying operational parameters.

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