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# DETERMINATION OF OPTIMAL FREEZE-DRYING MODES OF COTTAGE CHEESE WHEY PERMEATE AS A SEMI-FINISHED PRODUCT IN THE PRODUCTION FOR ENTERAL NUTRITION PRODUCTS

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## Abstract

The large volumes of cottage cheese whey produced in the country cause a strong pressure on the environment of dairy processing enterprises. This valuable secondary milk raw material, containing more than 50% of the dry solids of natural milk, is not in demand among processors due to the lack of data on the complex use of the processes of its baromembrane separation and subsequent freeze-drying of permeate. Expansion of the practical application of the baromembrane technology of cottage cheese whey deep processing in the production of long-term semi-finished products for enteral nutrition is proposed to be carried out by experimentally determining the optimal values of the main operating parameters of the sublimation process for deep-purified permeate.

The main raw materials are fresh cottage cheese whey obtained in production conditions, which is the main part of secondary dairy raw materials on the territory of the Russian Federation in GOST R 53438-2009 (nationwide standard) and Jerusalem artichoke tuber extract (industrial type). To determine the physical and chemical properties of raw materials, semi-finished products and finished products, the main parameters of the processes of their baromembrane purification and subsequent freeze-drying, standard and generally accepted methods and certified laboratory equipment were used.

Results of the study were following: 1) the optimal modes of freeze-drying of cottage cheese whey permeate had been established, in which the particle size in the finished product had not exceeded 67.0 ± 2 microns, the solubility index reached 0.41 ± 0.01%, the bulk density was 587 kg/m$^3$; 2) physicochemical characteristics organoleptic and microbiological parameters of the finished dry permeate, which could be used in the production of products for enteral nutrition, exceeded the nearest analogues in protein content no less than 5 times, in the content of the mesophilic aerobic and facultative anaerobic microorganisms, CFU/cm$^3$ (g) - by at least an order of magnitude.

The results of experimental studies had shown that freeze-dried cottage cheese whey permeate (PCCW), obtained in laboratory conditions in accordance with the developed technology, exceeded the nearest industrial analogues in terms of physicochemical, organoleptic and microbiological parameters.

***Key words**: Sublimation, Permeate, Cottage Cheese Whey, Membrane Separations.*

## 1. Introduction

The technology of whey processing based on the processes of its baromembrane separation has found wide practical application [1, 12]. The peculiarity of these processes is that the osmotic pressure of the separated system is significantly less than the working pressure (0.1 - 3 MPa). In this case, the membranes retain mainly high-molecular compounds and pass low-molecular substances with the solvent flow [12]. Permeate is a practically pure solution of

lactose, vitamins and salts of the mineral complex of milk. Further fractionation of permeate through nanofiltration and reverse osmosis membranes makes it possible to obtain semi-finished products for the preparation of various food products and beverages used for enteral nutrition. The problem of increasing their shelf life is usually solved by using the freeze-drying process, which can preserve the native forms of all dry ingredients, including milk mixtures, as the basis of such products. In modern freeze-drying plants, the duration of drying in a thin layer is 1.5 - 2 hours, however, due to the low productivity, they have found only limited use in the production of high-quality products for enteral nutrition of personnel under the autonomous systems operating in extreme conditions [2-6, 9, and 11].

The aim of the study was to experimentally determine the optimal values of the main operating parameters of the freeze-drying process of deep-purified cottage cheese whey, as one of the basic components of the milk mixture.

## 2. Materials and Methods

As a raw material, the cottage cheese whey in nationwide standard [11], previously purified from casein dust and milk fat particles, was used. Further purification was carried out in three main stages: first, by complexing polysaccharides of liquid Jerusalem artichoke extract and part of whey proteins (in a volume ratio of 30 : 70) by separating of the sediment. As a result, the residual protein content in the treated whey should not exceed 0.4 - 0.5%. At the second stage the ultrafiltration has been used for residual part of some whey proteins removing to produce ultrafiltration (UF) permeate. And, thirdly, the final nanofiltration of this permeate has been used for its final purification from nitrogenous substances.

Control of the required degree of cottage cheese whey purification from protein compounds at each stage of its processing was carried out according to the nationwide standard [13] using a certified UDK 139 analyzer (VELP). Based on the data obtained using the laboratory photocolorimeter, the logarithmic unit of optical density (P) was calculated using the generally accepted formula:

$$P=\lg(S_0/S) \quad (1)$$

Where: $S_0$ - the flow of radiation, which is incident on the object, $S$ - the flow of radiation passed through the object.

Based on the analysis of the own research results [1, 3 - 6, 9, 10, and 12], which have been not contradicted by the data from other scientific publications, it is established that for deep purification of cottage cheese whey using its baromembrane separation, it is possible to use membranes with a delay threshold of 20,000 - 30,0000 Da (for ultrafiltration) and up to 200 Da (for nanofiltration). Operational data on membranes, the main physicochemical characteristics of raw materials and semi-finished products are summarized in Tables 1 and 2. The processes of baromembrane separation of the studied objects were carried out on certified equipment. The experimental data were obtained using standard methods and processed on a PC in Microsoft Office applications. The selectivity of the membranes φ was determined by calculation

**Table 1. Physicochemical characteristics of the research object (p = 0.95)**

| Performance factors | Raw material | Cottage cheese whey after pre-purifying and concentration | | |
|---|---|---|---|---|
| | | Stage 1 [8, 9] | Stage 2 [8, 9] | Stage 3 [8, 9] |
| **Lactose** | 4.4 | 4.4 | 4.1 ± 0.1 | 22.3 ± 0.2 |
| **Protein (N x 6.38)** | 1.1 | 0.4 | 0.035 ± 0.001 | 0.5 ± 0.01 |
| **Fats** | 0.1 | 0.1 | 0.05 ± 0.01 | 1,052 ± 2 |
| **Log. unit of optical density** | 0.26 | 0.13 | 0.05 | 0.28 |

**Table 2. Operational parameters of membrane**

| Performance factors | Membrane brands | | | | |
|---|---|---|---|---|---|
| | Ultrafiltration polyamide membranes (UPM-20) (Vladipor, Russia) | UPM-50 (Vladipor, Russia) | Biomax 100 (Merck Millipore) | Biomax 300 (Merck Millipore) | Alfa Laval NF |
| **Pressure, MPa** | 0.1 - 0.4 | 0.1 - 0.5 | 0.4 - 0.5 | 0.4 - 0.5 | 1.5 - 4.0 |
| **Retention threshold of membranes, Da** | 45000 - 50000 | 50000 - 55000 | 95000 - 100000 | 295000 - 300000 | 200 |
| **Temperature, °C** | 5 - 40 | 10 - 50 | 4 - 50 | 4 - 50 | 5 - 50 |
| **pH of the cleaning medium** | 2 - 10 | 2 - 12 | 1 - 14 | 1 - 14 | 2 - 10 |

according to the generally accepted formula:

$$\varphi = (W_2 \cdot k_2 / W_1 \cdot k_1) \cdot 100\% \quad (2)$$

Where: $W_1$- the volume of cottage cheese whey at the beginning of the baromembrane separation process, $k_2$- mass fraction of dispersed phase particles in it; $W_2$ and $k_2$- the volume of cottage cheese whey at the end of the baromembrane separation process and the mass fraction of dry solids, respectively, in this volume.

The ultrafiltration process was carried out with the following operating parameters:

- Working pressure in the apparatus of P (MPa) - up to 0.44 - 0.48.

- Flow rate of the separated system V (m/s) is 0.1 - 0.3.

- Temperature t, $^0$C - 12 - 15.

The nanofiltration process was carried out at the following operating parameters:

- Working pressure in the apparatus of P (MPa) - up to 4.5.

- Flow rate of the separated system V (m/s) up to 0.3.

- Temperature t, $^0$C - 12 - 15.

The adequacy of the obtained equations was checked using Fisher criteria, the calculated value of which cannot exceed the table value, depending on the number of experiments performed and the number of factors studied. The equations coefficients were analyzed in accordance with the Student's criteria (at a significance level of 0.05 when p = 0.95).

## 3. Results and Discussion

Permeates of pre-purified cottage cheese whey obtained using ultrafiltration membranes with a retention threshold of 100 kDa (PCCW 100) was used, concentrated by nanofiltration to the content of CD.S = 23 - 25% (NPCCW) had been taken for experimental studies of the freeze-drying process. At the first stage (Figure 1), the raw material was distributed on a tray in a uniform layer with 6-8 mm in its thickness and frozen up to t = -20 - 22 $^0$C. At the second stage, drying was carried out in a freeze-drying chamber with a residual pressure of 5 - 7 Pa. When the residual moisture content of the material was less than 45%, to intensify the freeze-drying process, the trays were heated after 8, 9, 10 and 11 hours since the second stage had been started.

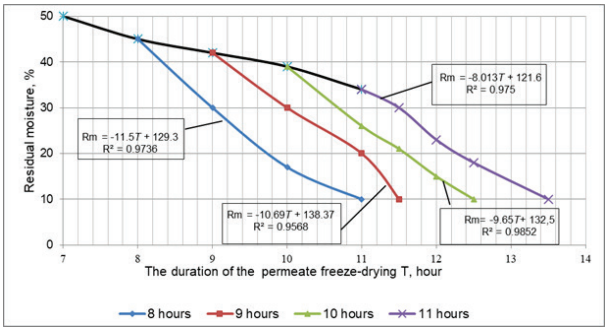


**Figure 1. Dependences of the residual moisture on the freeze-drying NPCCW duration**

Based on the analysis of experimental data, it was found that the rate of moisture removal was the same when the trays had been additionally heating and the residual moisture of samples (8 - 10%) was achieved, in general, in 3 - 3.5 hours. Therefore, to reduce the drying process, the optimal mode is the process when the tray heating begins in 8 or 9 hours since the second stage has been starting with a total duration of freeze-drying of 11 - 11.5 hours, which corresponds to the drying modes of samples № 1 and № 2 (Table 3).

The results of organoleptic studies showed (Table 3) that samples № 1 and № 2 have a uniform consistency with a crystal structure, and uniform color and a weakly marked the whey taste. Samples № 3, and 4 have a cream tone, which could be occurred due to longer exposure at the drying temperature.

To obtain a finished product with a residual moisture of 4.5 - 5 %, it is necessary to carry out the third stage-heat drying of the permeate after freeze-drying, where the heating temperature is an important parameter. The values of this parameter ranged from +25 to +45 $^0$C. The duration of the final stage was 3 - 3.5 hours. Table 4 shows the organoleptic evaluation of the finished dry permeate at different drying temperatures.

**Table 3. Results of organoleptic evaluation of sublimated permeate**

| Factor | Samples №1 (11 hours) | Samples № 2 (11.5 hours) | Samples №3 (12.5 hours) | Samples № 4 (13.5 hours) |
|---|---|---|---|---|
| **Color** | 4 | 5 | 4 | 4 |
| **Taste** | 4 | 4 | 5 | 3 |
| **Odour** | 5 | 5 | 4 | 4 |
| **Consistency** | 5 | 5 | 4 | 4 |
| **Total score** | 4.5 | 4.75 | 4.25 | 3.75 |

As a result of organoleptic evaluation (Table 4) it was found that samples № 1 and 2 at $T_{DP}$=35 ± 2 °C retained a uniform consistency and uniform color over the mass, and a weakly marked the whey taste. Samples № 3 and 4 at $T_{DP}$ =45 ± 2 °C acquired an intense cream color because of the increased temperature of the drying process, that might reduce the consumer quality of the finished product.

Thus, as a result of experimental studies, optimal modes of freeze-drying NPCCW were established: the duration of freeze-drying was 11 - 11.5 hours, the drying temperature was t = 25 - 35 °C, and the total dehydration time was 14 - 15.5 hours.

To determine the possibility of further use of freeze-dried permeate in food formulations, its physical and chemical properties (Table 5), functional and technological characteristics (Table 6) and microbiological indicators (Table 7) were studied in comparison with analogues presented on the modern market.

It was found that the samples of freeze-dried NPCCW obtained in the laboratory differed from their analogues by a smaller amount of protein - 0.5 % (4.5, and 2% for analogues) (Table 5) accordingly, the average particle size is not more than 69 ± 2 microns (Table 6), with a smaller number of mesophilic aerobic and facultative anaerobic microorganisms, CFU/cm³ (g) - 1.31 x $10^3$ (1.65 x $10^4$ and 1.79 x $10^6$ for analogues) (Table 7).

**Table 4. Organoleptic evaluation of the finished dry permeate at different drying temperatures**

| Factor | $T_{DP}$ = 25 ± 2 °C | $T_{DP}$ = 35 ± 2 °C | $T_{DP}$ = 40 ± 2 °C | $T_{DP}$ = 45 ± 2 °C |
|---|---|---|---|---|
| **Color** | 5 | 5 | 4 | 4 |
| **Taste** | 4 | 4 | 4 | 4 |
| **Odour** | 4 | 5 | 5 | 5 |
| **Consistency** | 5 | 5 | 4 | 4 |
| **Total score** | 4.5 | 4.75 | 4.25 | 4.25 |

**Table 5. Physical and chemical characteristics of the sublimated product obtained in the laboratory NPCCW**

| Factor | Sublimated NPCCW | Lactalis industry | Company MOL (Russia) |
|---|---|---|---|
| **Weight ratio, %:** | | | |
| **Lactose** | 87 ± 2 | 80 ± 2 | 85 ± 2 |
| **Moistures** | 4.0 ± 0.5 | 4.0 ± 0.5 | 4.5 ± 0.5 |
| **Protein (N x 6.38)** | 0.5 | 4.5 ± 0.001 | 2 ± 0.001 |
| **Milk acid** | 0.05 ± 0,01 | 0.5 ± 0.01 | 0.1 ± 0.01 |
| **Minerals** | 6.2 ± 0,2 | 9.5 ± 0.2 | 7.0 ± 0.2 |
| **Fats** | 1.2 ± 0,01 | 1.5 ± 0.01 | 1.4 ± 0.01 |
| **Group of purity** | I | I | I |

**Table 6. Main functional and technological properties of the obtained sublimated NPCCW**

| Factor | Sublimated NPCCW | Lactalis industry | Company MOL (Russia) |
|---|---|---|---|
| **Volume density (bulk), kg/m³** | 587 ± 2 | 611 ± 2 | 619 ± 2 |
| **Density (with seal), kg/m³** | 736 ± 2 | 731 ± 2 | 725 ± 2 |
| **Average particle size, microns** | 67 ± 2 | 79 | 81.7 |
| **Solubility index, %** | 0.41 ± 0.01 | 0.54 ± 0.02 | 0.63 ± 0.01 |
| **pH (reconstituted product with SV 6% )** | 6.2 - 6.4 | 6.4 - 6.6 | 6.8 - 7 |
| **Group of purity** | I | I | I |

**Table 7. Microbiological indicators of sublimated NPCCW**

| Factor | | Sublimated NPCCW | Lactalis industry | Company MOL (Russia) |
|---|---|---|---|---|
| **Quantity of the mesophilic aerobic and facultative anaerobic microorganisms, CFU/cm³ (g)** | | 1.31 x $10^3$ | 1.65 x $10^4$ | 1.79 x $10^6$ |
| **Weight of the product (g), which is not detected:** | CGB (coli-forms) | 0.1 | 0.1 | 0.1 |
| | Pathogenic (including *Salmonella*) | 30 | 30 | 30 |
| **Yeasts, CFU/g,** | | not detected | 60 | 100 |
| **Moulds, CFU/g,** | | not detected | 10 | 10 |

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## 4. Conclusions

- Optimal modes of cottage cheese whey permeate freeze-drying had been established, in which the particle size in the finished product had not exceeded 67 ± 2 microns, the solubility index reached 0.41 ± 0.01 %, the bulk density was 587 kg/m$^3$.

- Physicochemical characteristics organoleptic and microbiological parameters of the finished dry permeate, which could be used in the production of products for enteral nutrition, exceeded the nearest analogues in protein content - no less than 5 times, in the content of the mesophilic aerobic and facultative anaerobic microorganisms, CFU/cm$^3$ (g) - by at least an order of magnitude.

- The results of experimental studies had shown that freeze-dried cottage cheese whey permeate (PCCW), obtained in laboratory conditions in accordance with the developed technology, exceeded the nearest industrial analogues in terms of physicochemical, organoleptic and microbiological parameters.

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