Separatory Freezing and Cryoconcentration of Milk and Whey

I A Korotkiy, E V Korotkaya, E N Neverov, I B Plotnikov and D A Efremov
Kemerovo State University, Krasnaya st., 6, Kemerovo, Russian Federation
E-mail: krot69@mail.ru

Abstract. A significant problem in the food industry is the lack of raw materials. At the same time, a significant part of the resources is not used for obtaining food products. This kind of food raw material includes whey, which contains a large amount of valuable components, in particular whey proteins and lactose. The article discusses the results of research devoted to the development of industrial technology for separating freezing (cryoconcentration) of skim milk and curd whey to obtain concentrated skim milk, as well as whey protein concentrate in low-tonnage production conditions. The research was carried out on products manufactured by dairy enterprises in Kemerovo, Russia.

1. Introduction
In the technology of dairy products production there is a problem of full and rational use of milk. Particular attention is paid to increasing the efficiency of industrial production based on the principles of waste-free or low-waste technology with the fullest possible use of raw materials.

Since the cost of raw materials in the cost of dairy products is more than 80%, the dairy industry is considered one of the most energy-intensive and material-intensive sectors of the national economy [7].

At dairy enterprises, during the implementation of technological processes for the production of dairy products, significant quantities of secondary dairy raw materials are produced – skim milk, buttermilk and whey.

One of the promising methods for processing this type of by-products is cryoconcentration and separation freezing technologies, which make it possible to increase the efficiency of milk processing enterprises, the depth of processing of raw materials, quality and obtain new types of products.

The method is based on the crystallization of water contained in the product and the removal of ice crystals. Thus, the concentration of dry matter in the product increases to 30-40%. The advantage of the technology is the high quality characteristics of the product and the greater safety of valuable biological components due to the low temperatures of the technological process. Concentration occurs at low temperatures. As a result, the rate of biochemical changes is significantly reduced due to low temperatures, so the resulting concentrates have high quality indicators [9].

Partial removal of free moisture during cryoconcentration of liquid products in a dynamic and static mode and an increase in the concentration of dry matter in them creates unfavorable conditions for pathogenic microflora. This allows one to increase the storage time of concentrated products [2].
2. Research objects and research methods
The development of techniques and technology for cryoconcentration makes it possible to apply this method for separating freezing of milk, buttermilk and whey. Separative freezing technologies make it possible to obtain concentrates of whey proteins and lactose [6].

Separating whole milk produces skim milk. The component composition of whole milk, skim milk and the degree of transition of the components of whole milk to skim milk are presented in table 1 [11].

| Components          | Whole milk | Skim milk | Transition rate, % |
|---------------------|------------|-----------|--------------------|
| Mineral components  | 0.7        | 0.68      | 99.7               |
| Lactose             | 4.8        | 4.77      | 99.4               |
| Protein             | 3.3        | 3.27      | 99.3               |
| Dry matter          | 12.5       | 8.7       | 70.5               |
| Milk fat            | 3.8        | 0.05      | 1.3                |

Milk whey, as a by-product in the production of cheese, cottage cheese and casein, contains a significant amount of valuable components, therefore it is an important raw material resource for the dairy industry [5].

The global demand for whey ingredients shows a higher dynamic compared to other dairy products. Therefore, one of the most significant directions in the development of the dairy industry is the trend for deep processing of whey. This is stimulated by the constant improvement of technologies, hardware support of technologies, as well as regulatory legal acts that tighten the responsibility of enterprises for the utilization of milk whey without processing [1].

In recent years, ultra- and nanofiltration technologies, as well as electrodialysis, have been increasingly used for the processing of whey. The dairy industry in Russia does not stay away from global trends, but the domestic dairy industry has its own specifics. First of all, a popular product in Russia is cottage cheese, the by-product of the production of which is curd whey, which is more mineralized in composition than cottage cheese, while the production of a significant amount of cottage cheese is concentrated in small enterprises that are located quite far from each other, which creates significant difficulties to centralize the processing of curd whey. Thus, due to the lack of effective technologies for low-tonnage whey processing, possible annual losses amount to a significant amount. According to some estimates, the domestic dairy industry loses annually from 6 to 8 billion rubles due to insufficient dissemination of technologies for deep processing of whey [8].

Separation freezing is currently a promising industrial processing technology, in particular for the dairy industry. This process is realized through cryoconcentration in the temperature range from 0 °C to –15 °C. Low process temperatures allow the maximum preservation of the initial properties of the components obtained during separation, exclude protein denaturation and preserve thermolabile substances [3].

The Kemerovo State University is developing technologies for cryoconcentration and separation freezing of liquid food media, in particular, whey.

The whey was obtained in the production of cottage cheese by the acid method at the enterprises of the city of Kemerovo. All samples had organoleptic characteristics characteristic of this type of product. The chemical composition and physicochemical parameters of serum for cryoconcentration are shown in tables 2, 3.
Table 2. Chemical composition of serum samples for cryoconcentration.

| Manufacturer       | LLC “Skomoroshka” | IAPC “Podvorie” | LLC “Naturalnoe moloko” |
|--------------------|--------------------|-----------------|-------------------------|
| Water              | 94.2               | 94.9            | 96.0                    |
| Dry matter, including: |                  |                 |                         |
| proteins           | 5.9                | 5.1             | 4.0                     |
| fats               | 0.6                | 0.7             | 0.5                     |
| carbohydrates      | 4.6                | 3.8             | 2.7                     |
| minerals           | 0.6                | 0.7             | 0.8                     |

Table 3. Physicochemical parameters of serum samples for cryoconcentration.

| Manufacturer       | IAPC “Podvorie” | LLC “Skomoroshka” | LLC “Naturalnoe moloko” |
|--------------------|-----------------|-------------------|-------------------------|
| Acidity:           |                 |                   |                         |
| active, pH         | 4.4             | 4.6               | 4.5                     |
| titratable, T      | 87              | 73                | 63                      |
| Density, kg/m³     | 1027            | 1026              | 1026                    |
| Dynamic viscosity, Pa×s | 1.25×10⁻³     | 1.17×10⁻³         | 1.24×10⁻³               |
| Kinematic viscosity×10⁶, m²/s | 1.24             | 1.22              | 1.22                    |

The curd whey met the requirements of GOST 34352-2017. “Milk whey is a raw material. Technical conditions” and GOST 33957-2016. “Milk whey and drinks based on it. Acceptance rules, sampling and control methods”.

3. Results and discussion

Based on the research results, a technology for cryoconcentration of skim milk has been developed. The process of concentration itself takes place in cryoconcentrators of a capacitive type. The technological scheme for the production of concentrated skim milk is shown in figure 1.

Analyzing the obtained quantitative ratios, it was calculated that to obtain 1 ton of the finished product (concentrated skim milk with a dry matter content of 27.6%), it is necessary to use 2.98 tons of the original skim milk - raw material.

The melting of the crystallize is carried out due to the heat of condensation of the refrigerant. Thus, the supply of energy at all stages of separation freezing is carried out only for the movement of the feedstock and separation products, as well as for the transfer of heat between the separation products and the transfer of its surplus to the environment.

Skim milk is a valuable raw material in itself, cryoconcentration technologies make it possible not only to obtain a rather valuable product - concentrated skim milk, but also to use concentrated skim milk to obtain cottage cheese, cheeses, milk-protein concentrates [4].

By cryoconcentration of curd whey, whey protein concentrate was obtained, which had the corresponding organoleptic characteristics in terms of color, consistency, appearance, color and smell. Chemical composition and physicochemical parameters are shown in tables 4–5.

The resulting whey protein concentrate corresponds to a standard albumin with a dry matter content of 20%.
Table 4. Whey protein concentrate chemical composition.

| Component name                  | Component content, % |
|---------------------------------|-----------------------|
| Mass fraction of dry matter     | 20.2                  |
| Mass fraction of total protein: |                       |
| whey proteins                   | 12.9                  |
| casein proteins                 | 8.0                   |
| Total nitrogen                  | 2.0                   |
| Non-protein nitrogen            | 0.3                   |

Table 5. Physicochemical parameters of whey protein concentrate.

| Indicator name | Indicator value |
|----------------|-----------------|
| Acidity:       |                 |
| active, pH     | 4.35            |
| titratable, T  | 95              |

Figure 1. Block diagram of the production of concentrated skim milk.

The obtained laboratory data made it possible to work out the technological process and develop an industrial technology for the isolation of whey protein concentrate by the separation freezing method [10].

The technological process is a sequential combination of the following stages:
1) reservation of whey, separation of casein dust, cooling, accumulation;
2) double cryoconcentration;
3) thermal coagulation of concentrated whey;
4) separation of whey protein concentrate.

The technological scheme for the production of whey protein concentrate is shown in figure 2.

The process for the production of whey protein concentrate is a divergent flow. In this case, the input and output of material flows to the block of cryoconcentration apparatuses are equal and with a full cycle of double cryoconcentration (36 hours) is 15 tons. The branching of material flows occurs at the stage of cryoconcentration and with further thermal coagulation.

Thus, the yield of whey protein concentrate when processing 15 tons of whey is 93 kg.
At the same time, about 3.8 tons of lactose concentrate with a mass fraction of about 16% is also a useful product.

The total energy consumption for the implementation of all technological processes is 471 kW·h.

Figure 2. Technological scheme for the production of whey protein concentrate.
Thus, as a result of the research, the process of cryoconcentration of dairy by-products – skim milk and curd whey – has been studied. The dairy products obtained in the process of cryoconcentration – concentrated skim milk and whey protein concentrate – have been investigated. Also, schemes have been developed for industrial continuous production of these products for low-tonnage production – 10 tons per day for feedstock.

References
[1] Alinovi M, Mucchetti G, Wiking L and Corredig M 2020 Freezing as a solution to preserve the quality of dairy products: the case of milk, curds and cheese Critical Reviews in Food Science and Nutrition DOI: 2-s2.0-85088589002
[2] Buyanova I V, Lupinskaya S M, Ostromov L A and Mazeeva I A 2021 Innovative low temperature methods of milk whey processing IOP Conference Series: Earth and Environmental Science 640 (3) 158
[3] Casas-Forero N, Orellana-Palma and Petzold G 2021 Recovery of Solutes from Ice and Concentrated Fractions in Centrifugal Block Cryoconcentration Applied to Blueberry Juice Food and Bioprocess Technology 14 (6) 1155–1168 DOI: 2-s2.0-85103426921
[4] Duran T, Minatovicz B, Bai J, Mohammediarani H and Chaudhuri B 2021 Molecular Dynamics Simulation to Uncover the Mechanisms of Protein Instability During Freezing Journal of Pharmaceutical Sciences 110 (6) 2457–2471
[5] Gavrilov G B, Prosekov A Yu, Kravchenko E F and Gavrilova B G 2015 Handbook on the processing of whey. Technologies, processes and devices, membrane equipment (SPb: ID Professiya) 176 p
[6] Korotkiy I A, Korotkaya E V, Neverov E N, Fedorov D E and Gushchin A A 2021 The parameters selection for separating freezing-out for extracting the components from curd whey IOP Conference Series: Earth and Environmental Science 640 (4) 042016 DOI: 2-s2.0-85101715452
[7] Korotkiy I A, Plotnikov I B and Mazeeva I A 2019 Current trends in the processing of whey (2019) Technique and technology of food production Vol. 49 227–234
[8] Korotkiy I A, Plotnikov I B and Mazeeva I A 2019 Separated freezing of milk whey Dairy industry 11 33–34
[9] Meneses D L, Ruiz Y, Hernandez E and Moreno F L 2021 Multi-stage block freeze-concentration of green tea (Camellia sinensis) extract Journal of Food Engineering 293 110381
[10] Ostromov L A, Korotkaya E V and Malceva O M 2018 Effect of cryoconcentration on the dry matter content of skim milk Dairy industry 8 60–61
[11] Vidal-San Martin C, Bastias-Montes J M, Villagra-Jorquera C, Salinas-Huchenlao G, Flores-Rios A, Gonzáles-Diaz N, Tamarit-Pino Y and Muñoz-Fariña O 2021 Effect of cryoconcentration assisted by centrifugation-filtration on bioactive compounds and microbiological quality of aqueous maqui (Aristotelia chilensis (mol.) stuntz) and calafate (berberis microphylla g. forst) extracts pretreated with high-pressure homogenization Processes 9 (4) 692 DOI: 2-s2.0-85105015682