Membrane purification of secondary milk raw materials: intensification of processes

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Abstract. Further development of the human community depends on two main tasks to be performed, i.e. providing the population with high-grade food and preserving the environment due to the rational use of natural raw materials. In this regard, processing of secondary milk raw materials is considered a controlled impact on a complex biotechnological system. The impact was divided into preliminary treatment and final processing and is based on two principles; firstly, secondary dairy raw materials must be standardized before the initial stage of its deep processing, involving fractionation; and secondly, the first step of the baromembrane purification of secondary milk raw materials should ensure the purification of the permeate from proteins and milk fat. The purpose of the study was to theoretically substantiate and experimentally determine the optimal operating parameters of the baromembrane processes in the technology for obtaining highly purified whey permeate. The authors have experimentally established that the deep baromembrane purification of milk whey ensures the optimal values of the working pressure and circulation rate of the separated system that are $P=0.45-0.5$ MPa and $V=0.15-0.2$ m/sec in the channel of the ultrafiltration apparatus and $P=1.5-2.5$ MPa and $V=0.16-0.26$ m/sec in the channel of the nanofiltration apparatus, respectively.

1. Introduction

Ecological modernization of food industry enterprises is one of the main tasks for all its branches; at dairy industry enterprises this will significantly reduce or even eliminate the negative impact of modern production on the environment [1, 2]. The problem can be solved by membrane equipment used for separating milk into fractions without residues; combining these fractions enables obtaining finished high-quality products [3-6]. The traditional scheme for processing whole and/or part-skim milk (for cheeses, curd products, and casein productions) provides for the removal of about half of the dry matter in the form of whey as a by-product or waste; its cost is considerably lower than the initial raw material. However, its unique composition, physicochemical properties, and biological value provide basis to consider whey as a full-fledged type of secondary milk raw material, for example, for the production of highly purified lactose.

The purpose of the study was to theoretically substantiate and experimentally determine the optimal operating parameters of the baromembrane processes in the technology of highly purified whey permeate as a nutrient for milk whey.

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2. Materials and methods

In the experimental study, we used curd whey obtained in the manufacture of basic products in accordance with ГОСТ 31534-2012 [7] at the Stavropol Dairy Plant (Table 1).

| Parameter       | Curd whey |
|-----------------|-----------|
| Lactose         | 5.1±0.2   |
| Protein         | 1.0±0.001 |
| Fat             | 0.1±0.01  |
| Density, kg/m³  | 1023      |

The data analysis [8-12] showed that at present it was advisable to use polymer membranes of the UPM 20, UPM 50, UAM 150, UAM 500, and HyStream brands for ultrafiltration of curd whey, while the Alfa Laval NF brand with delay threshold from 100 to 200 Da was good for nanofiltration of permeate (Table 2).

| Main parameter       | Membrane type |
|----------------------|---------------|
|                      | UPM-20        |
|                      | UPM-50        |
|                      | UAM-150       |
|                      | UAM-500       |
|                      | HyStream      |
|                      | Alfa Laval NF |
| Delay threshold, kDa | 15-20         |
|                      | 40-50         |
|                      | 20-25         |
|                      | 50-55         |
|                      | 45-50         |
|                      | 0.1 – 0.2     |

To determine the physicochemical characteristics of the research objects—separated curd whey and its permeates, including those thickened by the baromembrane concentration, generally accepted and standard procedures were applied. The whey was ultrafiltrated using a laboratory baromembrane installation “Millipore” while selecting semi-permeable membranes. The filtering surface area was 0.05 m². The basic operating mode of the installation was periodic, but continuous implementation of the process was possible in case of a constant supply of fresh raw material. An Alfa Laval TestUnit M20 was used for nanofiltration of the whey permeate. Permeate and retentate were sublimated on a “Scientz-ND” unit. The experimental data were processed using Microsoft Office 2010 and Statistica 12.0 programs. We used an orthogonal design of a full factorial experiment, which allowed obtaining independent estimates of the effects and determining the optimal implementation modes on their basis.

3. Results and discussion

3.1. Theoretical aspects of intensified membrane purification of secondary milk raw material

The industrial processing of secondary dairy raw material should be considered as a directed and controlled impact on a complex biotechnological system. So, all types of such exposure should be divided into two main groups, namely, preliminary treatment and final treatment. The basis of the concept of deep processing secondary milk raw material is schematically shown in Figure 1. One of the main principles of the proposed concept can be formulated as follows: secondary milk raw material should be standardized before the initial stage of its deep processing that provides for fractionation. The need to comply with this principle is primarily due to the basic conditions for the effective baromembrane processes, i.e.

- standard, commercially available equipment;
- a unified series of membranes with specified values of permeability for the standard permeate of secondary dairy raw material and selectivity for whey proteins; and
- measures aimed at clarifying or adapting the main modes of the baromembrane equipment.
operation should be excluded from the commissioning works, depending on the physical and chemical indices of the secondary milk raw material.

Figure 1. Concept (a fragment) of deep processing of secondary milk raw materials.

To implement this principle, the initial stage of the subsequent deep processing of secondary milk raw materials, including milk whey, is based on the membrane equipment and should provide for the following technological operations:

- pasteurization by the traditional method or sanitation using microfiltration equipment;
- cleaning from mechanical impurities, casein particles, and milk fat by centrifugal forces, including during the vibrational motion of the system being divided; and
- isolation of a part of the protein fraction in one way or another, for example, by adding a complex of plant polysaccharides (Jerusalem artichoke extract) to the previously purified milk whey and subsequent precipitation of the formed high-molecular complex “whey proteins - polysaccharide.”

The method to be chosen for pretreatment of secondary milk raw materials was determined by its physical and chemical properties and the requirements of the technology for further processing; its effectiveness depended on the composition and the extent of application of the equipment at the enterprise. In this case, it was possible to use thermocoagulation, sorption, chromatography, membrane filtration, etc. The boundary varying conditions for methods and mutually influencing modes of each specific technological operation must be established on the basis of indicators that determine the compliance of the received raw material with the “standard” requirements. The implementation of the proposed concept was caused by experimental research (figure 2) and interrelated practical tasks that were
• substantiation of the composition and content of the main parameters of standardized secondary milk raw materials; and
• determination of the permeate permeability parameters, membrane protein selectivity, and the type of membrane equipment for purification of, for example, whey.

![Flowchart]

**Figure 2.** The main fragment of the tasks of processing secondary milk raw materials and general method of performing them experimentally.

We adopted a sieve model of the baromembrane separation of secondary milk raw material and, accordingly, ultrafiltration as the first stage of its processing. So, the content of the “standard” parameters was determined primarily by the efficiency of ultrafiltration separation of standardized raw material. There was formulated the second principle that served as a basis for the concept proposed, namely, the first step of the baromembrane purification of secondary milk raw materials should ensure the purification of the permeate from proteins and milk fat. This principle provided favorable conditions
for the isolation of lactose from secondary milk raw materials at subsequent stages of its deep processing. Various combinations of these processes depend, first of all, on the cost of technologies and equipment necessary for their implementation, the volume of raw materials, the required depth of its processing, and the market price of the finished product. Taking into account the peculiarities and working conditions of the major milk processors in Russia, the most promising is the implementation of membrane processes that allow concentrating and fractionating the main components of whey purified from fat and proteins, varying in volumes of processed raw materials and directions for further use of all its components. In accordance with the concept proposed, the optimal ranges of values of the operating pressure and the rate of milk whey circulation in the channel of the baromembrane apparatus were determined experimentally.

3.2. Optimal values of main modes of baromembrane cleaning the milk whey.

We established that, before the nanofiltration permeate concentration, it was advisable to conduct ultrafiltration of whey on UPM-50 or HyStream membranes, depending on their service life and peculiarities of the washing procedure. The obtained permeate samples were noted for high quality—the residual content of total nitrogen was less than 0.02% (table 3).

Table 3. Main physical and chemical parameters of whey permeate.

| Parameter          | UPM-20        | UPM-50        | UAM-150       | UAM-500       | HyStream     |
|--------------------|---------------|---------------|---------------|---------------|--------------|
| Total nitrogen, %  | 0.01±0.001    | 0.015±0.001   | 0.018±0.001   | 0.02±0.001    | 0.016±0.001  |
| Lactose, %         | 4.6±0.2       | 4.6±0.2       | 4.7±0.2       | 4.8±0.2       | 4.6±0.2      |
| Fat, %             | 0.1±0.01      | 0.1±0.01      | 0.1±0.01      | 0.1±0.01      | 0.1±0.01     |
| Density, kg/m³     | 1022±2.0      | 1022±2.0      | 1022±2.0      | 1022±2.0      | 1022±2.0     |

The subsequent nanofiltration of the permeate resulted in obtaining samples of a mineral complex solution of milk whey that were almost free of high molecular weight components or lactose concentrates, containing minor impurities of protein substances, in comparison with the traditional production methods (table 4).

Table 4. The main physical and chemical parameters of the mineral complex solutions of milk whey (permeate-N) and lactose concentrate (retentate-N).

| Parameter          | Permeate-N | Retentate-N |
|--------------------|------------|-------------|
| Total nitrogen, %  | 0.005±0.001| 0.005±0.001 |
| Lactose, %         | 0.1±0.01   | 21.6±0.2    |
| Fat, %             | -          | 0.5±0.01    |
| Density, kg/m³     | 1015±2.0   | 1056±2.0    |

In terms of physical and chemical characteristics, both semi-finished products met the requirements of traditional technologies for their further deep processing. Subsequent freeze drying of the retentate-N samples made it possible to obtain a product whose quality indices were superior to known analogues (table 5).

Table 5. Physical and chemical characteristics of the retentate-N sublimed.

| Parameter          | Retentate-N | LACTALIS INDUSTRIE | «MOL» Company |
|--------------------|-------------|---------------------|---------------|
| Weight fraction of|             |                     |               |
| lactose, %         | 89±2        | 80±2                | 85±2          |
| moisture, %        | 4.0±0.5     | 4.0±0.5             | 4.5±0.5       |
| protein (N x 6.38), % | 0.4      | 4.5±0.001           | 2±0.001       |
| fat, %             | 1.1±0.01    | 1.5±0.01            | 1.4±0.01      |
| lactic acid, %     | 0.04±0.01   | 0.5±0.01            | 0.1±0.01      |
| Purity group       | I           | I                   | I             |

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Sequential iteration of the main processes in accordance with the scheme proposed (figure 2) made it possible to determine the optimal values of the main modes of baromembrane purification of the milk whey. The optimal values of the working pressure and circulation rate of the separated system were $P=0.45-0.5$ MPa and $V=0.15-0.2$ m/sec in the channel of the ultrafiltration apparatus and $P=1.5-2.5$ MPa and $V=0.16-0.26$ m/sec in the channel of the nanofiltration apparatus, respectively.

4. Conclusions
The research enabled us to obtain data that substantiate the feasibility of further testing the concept of deep processing secondary dairy raw materials. The concept is based on the following principles:

- secondary dairy raw materials must be standardized before the initial stage of its deep processing, providing for fractionation;
- the first stage of the baromembrane purification of secondary milk raw materials should ensure the purification of permeate from proteins and milk fat.

The legitimacy of the developed concept was confirmed by the experimentally obtained optimal values of the main operating parameters of the baromembrane whey separation.

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