Membrane process for the extraction of casein and whey proteins from skim milk

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Abstract. In deep processing of milk, microfiltration is used to isolate native micellar casein. The need to reduce its cost by increasing the efficiency of this process determines the relevance of research work in this area. The purpose of our research is to analyze the a priori information. This will determine the practical value and prospects of subsequent experimental determination of optimal parameters of the skim milk microfiltration process. The main steps of information search by keywords: selection of databases (Scopus, WOS, ScienceDirect, GoogleScholar, etc.) and the most authoritative editions (J. of Dairy Science, J. Membrane Science, J. Membranes), where appearance of publications with practical application in the research subject is noted since 2007, bibliography analysis of scientific articles. Non-academic materials are excluded from the search because they lack full descriptions of research methods, which complicates the reproducibility of the presented results. Analysis of publications devoted to methods of increasing the efficiency of membrane separation of dairy raw materials showed that most of them are partial solutions to this problem. With the limitations - the properties of separation objects, membrane materials, types of apparatuses, etc. cause difficulties in the practical use of the results under changing physical and chemical characteristics of natural milk. But always the main operating parameters of the skim milk microfiltration process are the transmembrane pressure, the circulation rate of the separated system in the apparatus and its temperature. Optimal conditions of milk microfiltration for separation of native micellar casein should be sought experimentally on the basis of creating mathematical models of the process followed by their analysis by numerical methods, as the data given by the authors should be considered as indicative, depending on raw materials, membranes and separation technology.

1 Introduction

Waste-free, environmentally friendly production of dairy products nowadays can only be organized based on deep processing technology of the original raw materials. Thanks to modern advances in membrane technology, milk can be separated into several major components used to produce food and beverages. Of particular interest to the food industry are micellar casein concentrates, which have unique natural functional and nutritional

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properties and are produced in native form using microfiltration from skim milk. Such semi-finished products can be used for protein enrichment of various food products, as well as for standardization of milk in the technology of cheese products, yogurt, various drinks, including long-term storage, which contributes to a significant reduction in their cost. However, adjustments to the classic technology of milk processing often lead to a significant change in organoleptic characteristics of the resulting products - from sweet and fragrant to tasteless.

On the other hand, despite all the achievements in the field of microfiltration separation of skim milk, the effectiveness of this process, is still low. This is due to the complexity of the composition of the feedstock and changes in its physical and chemical parameters during microfiltration separation depending on a number of factors. Next, the material, membrane manufacturing method, type of apparatus, the method of organization and conduct of the microfiltration process, technological requirements for physical and chemical characteristics of retentate and permeate, have a significant impact on the results obtained. Insufficient completeness of the theory of membrane processes and practical results of its application make it necessary to carry out their own research work in this area, based on the analysis of a priori, including patent information collected from scientific publications and subsequent obtaining new own experimental data.

The purpose of this literature review and meta analysis is to obtain the initial data necessary for the experiments with the process of microfiltration of skim milk and extracting casein fractions from it. Figures and tables Figures and tables, as originals of good quality and well contrasted, are to be in their final form, ready for reproduction, pasted in the appropriate place in the text. Try to ensure that the size of the text in your figures is approximately the same size as the main text (10 point). Try to ensure that lines are no thinner than 0.25 point.

2 Milk processing based on the application of membrane separation processes

The process of microfiltration of milk using membranes with an average conditional pore diameter (CPD) of 0.1-0.2 μm can be used for separation of two main groups of milk proteins from these raw materials: casein micelles (~150 nm) and whey proteins (~2-15 nm). Retentate, whose protein fraction is represented mainly by casein micelles, is generally used in the production of cheese and cheese products. The permeate containing whey proteins, lactose and the mineral complex of the milk is usually subjected to ultrafiltration/diafiltration and used in the production of infant formulas. The general scheme of milk processing [1, 2], which provides for its microfiltration fractionation (Figure 1), includes preliminary treatment of this raw material: collection and storage (1), degreasing (2), inactivation by some means, primarily of pathogenic microflora (3,4,5) and stabilization of the separated system temperature (6) before its feeding into the baromembrane devices (7,8).

Possible variants of milk processing include the following raw milk flows: 1-2-3-6-7-8-10-11-12-13-14, 1-2-4-6-7-8-9-10-12-13-14, 1-2-5-6-9-10-11-12-13-14 or their reduced variations. The physical and chemical properties of raw materials in this case will be determined by the modes of each technological operation and the choice of flow variation.

The cross-flow microfiltration process is characterized by the following parameters:
- the value of operating pressure (TMP), defined as the difference of pressures in the circuit of the baromembrane apparatus between the sides of retentate and permeate;
- The mass flow rate of permeate passing through the membrane (Jp);
- circulation velocity of the system to be separated in the channel of the baromembrane apparatus (V);
- coefficient of volume reduction, i.e. the ratio of raw material flow rate to retentate flow rate.

Fig. 1. General scheme of the baromembrane milk separation.

There are two basic methodical approaches to conditions of microfiltration process: determination of the best variant of the whole technological line based on specially chosen criteria [1], or maintenance of optimum regimes of all technological operations connected with it. In any case working parameters of microfiltration itself are determined by a number of interrelated variables. Currently, these relationships have not been fully defined in explicit form for the microfiltration process of milk separation. Yet, research, including various modeling methods in this area have been conducted for a long time [3-5]. The implementation of the baromembrane method of dairy raw milk fractionation under industrial conditions is based, as a rule, on the available expert knowledge and empirical data, which are associated mainly with many years of experience of specialists [6]. Preliminary data on optimum operating conditions of corresponding equipment are usually determined on the basis of analysis of results of experimental studies [7–13]. The solution of the problem by modeling of the microfiltration process [14] is actually based on the analysis of experimental data and is usually limited by design features of the baromembrane apparatus. Comparison of the main operating parameters of the microfiltration process carried out on different types of membranes [13] shows that, all the rest being equal, their characteristics have a significant impact on the filtration area and therefore on cost of equipment. Since it is the filter element that is the main part of any baromembrane
One of the first tasks of optimization of the technological scheme for micellar casein concentrate 15–18]. Ceramic membranes, which are more expensive than polymeric ones, provide under industrial conditions permeate flow rates up to 75-80 kg/m² h⁻¹, while polymeric membranes are only about 25 kg/m² h⁻¹ [17], being characterized by low capital and operating costs and low cost (price/square meter). However, polymeric membranes are more demanding to washing regulations, have a shorter service life than ceramic ones and are more prone to contamination [14]. It should be noted that the development of membranes that differ from imported ones in some performance characteristics (mechanical and chemical resistance, permeability, selectivity, thermal, cost, etc.) is quite successful in our country [19].

Currently for microfiltration treatment of dairy raw materials the most widespread are units completed with tubular ceramic or polymeric membranes, as a rule, of roll type. From about 2008 up to the present time in RF the developments of CJSC SPA «Elevar» (table 1) represent the interest in the sphere of membrane separation including milk raw materials, originally applied mainly for «cold pasteurization» of SM.

<table>
<thead>
<tr>
<th>Plants</th>
<th>Retentate capacity, l/h</th>
<th>Surface area, m²</th>
<th>Power, kW</th>
</tr>
</thead>
<tbody>
<tr>
<td>MFS-2x2x19M41</td>
<td>15000</td>
<td>32,6</td>
<td>62</td>
</tr>
<tr>
<td>MFS -2x2x19(7) M41</td>
<td>10000</td>
<td>22,3</td>
<td>43</td>
</tr>
<tr>
<td>MFS -2x2x7 M41</td>
<td>5000</td>
<td>12,0</td>
<td>25</td>
</tr>
<tr>
<td>MFS -2x2x7(3) M41</td>
<td>3500</td>
<td>8,6</td>
<td>19</td>
</tr>
<tr>
<td>MFS -2x2x3 M41</td>
<td>2300</td>
<td>5,2</td>
<td>14</td>
</tr>
</tbody>
</table>

The plants, depending on the capacity, can be placed on the area of 6-3 m², control options - from manual to fully automatic, are completed (replacement every 5 years) with ceramic multi-channel membrane elements [20]. In addition to capital costs, for example for the installation of MFS -2x2x7M41 the following daily basic operating costs are needed: steam - 1200 kg, water for CIP-washing - 6 m³, cooling water - 6 m³, detergents - 40 kg, the operator's salary. In accordance with the recommendations of the manufacturer of this equipment [20] in case of SM microfiltration the circulation speed of the separated system must be 5-6 m/s, and the TMP value in the channel of the baromembrane apparatus should be not more than 0.06-0.08 MPa. In this case the recommended value of concentration factor (ratio of treated milk volume to the difference of treated milk and permeate volumes) is 1:20, which corresponds to 95% yield of treated milk [20]. It should be noted that while CJSC SPA «Elevar» previously preferred foreign membrane manufacturers (Germany), at present, for example, LLC «Vladisart» quite successfully develops and produces in industrial quantities domestic analogues. However, despite the similarity of the basic control algorithms of membrane units of most of their manufacturers,
sometimes the replacement of imported membranes with domestic analogues may require adjustment of both their basic operating parameters and washing modes, including the selection of detergents [19].

With all the attractiveness of the baromembrane method of dairy raw material fractionation, the analysis of the research results [21], showed that only about 40% of whey proteins can be isolated from SM during its microfiltration using polymer membranes and a total of 60% when double the volume of water is added to the obtained MF retentate during diafiltration. The retentate obtained by this process is a concentrated colloidal suspension [22] containing casein in micellar form, lactose, minerals and some whey proteins. And permeate is obtained enriched with α-lactalbumin and β-lactoglobulin and can be used to produce native whey protein concentrates.

It should be noted that the baromembrane separation of SM (Figure 2) in about 30 minutes from the beginning of the process there is almost 20% decrease in permeability of polymer membrane by permeate [21] with its further decrease at a rate of about 1-1.5 (l/m² hour)/hour. At the same time diafiltration with deionized water contributes to intensification of the process of whey protein extraction in the range of 52-68%, depending on the operating parameters of both processes [23]. An increase in TMP, although resulting in a higher initial permeate flow rate, also results in an accelerated blocking of the pore space of the membranes [24]. The increase in the circulation rate of the separated system in the circuit of coil-type apparatus is very limited compared to the option of using ceramic membranes. This results in a risk of intensive formation of deposits on the membrane, which significantly increases its selectivity for whey proteins [25]. The combination of low and uniform over the length of the membrane channel TMR (UTP mode) in devices equipped with ceramic membranes with a high V value significantly reduces this risk.

Fig. 2. Permeability of polymer membranes in a roll-type apparatus (Marella et al., 2021).

Analysis of the results of experiments performed using ceramic membranes (0.1 μm of TechSep type) [26] shows that at 50 °C the Jp value can reach 80 kg/m²h with a casein selectivity of the membranes of about 80%.

The process of SM baromembrane separation in the UTP mode can be done in a plant where a high speed of the separating system is achieved by organizing the relative motion of the membrane itself. For example, by rotating the apparatus at an angular velocity corresponding to about n=1000 rpm [27]. If in terms of practical implementation of such design of membrane unit for microfiltration of milk may be quite reasonable doubts, from the position of research of the process itself, analysis of experimental data [28, 29] obtained with its help are, in our opinion, interesting (figure 3).
At membrane apparatus rotational speeds in the range of 1044-1492 rpm, the maximum value of \( J_p \) (permeate flow) is reached at a transmembrane pressure \( \text{TMP} \) value of about 0.125 MPa, while at 1931 rpm the \( J_p \) value continues to increase linearly with increasing \( \text{TMP} \). There is reason to assume that under such hydrodynamic conditions there is complete destruction of the «secondary layers» [30] sediments formed on membrane surface.

Analysis of the results of experimental studies (Fig. 4), performed practically under the same conditions, but using polymeric membranes with an average conventional pore diameter of 0.15 µm [29] showed that the maximum value of \( J_p \) index is achieved at a value of transmembrane pressure \( \text{TMP} \) about 0.25 MPa and no longer depends on it.

Based on the analysis of the data presented in Figures 3 and 4, we can conclude the following:

- for SM microfiltration separation both ceramic and polymeric membranes are almost equally possible, the choice should be determined on the basis of economic indicators;

- ceteris paribus for polymeric and ceramic membranes the main operating parameters of SM microfiltration process are the operating pressure \( \text{TMP} \), the circulation speed \( V \) of the separated system in the channel of the baromembrane apparatus and process.
temperature $t$, whose values are determined by the operating characteristics of membranes, type and design features of the baromembrane apparatus.

4 Influence of diafiltration on the microfiltration separation of skim milk

The use of microfiltration process using membranes with an average conventional pore diameter in the range of 0.1-0.2 μm for separation of casein fraction from SM has been known for more than 20 years [31]. With all the attractiveness of this method of fractionation of dairy raw materials, the question of increasing its efficiency and, accordingly, the profitability of such production on an industrial scale is still not completely solved. To date, the most common technological method for increasing the purity of the resulting casein fraction is diafiltration of MF-retentate. In one of the works performed on this topic [12], the feasibility of using DF is experimentally substantiated, but this is likely to involve not only the use of special ceramic membranes, in which the average conventional pore size varies along the length of the membrane channel, but also special conditions for the process of baromembrane separation. Later work [23] presents data on optimization of microfiltration process using polymeric membranes (from polyvinylidene fluoride - Parker Process Advanced Filtration Division, Oxnard, California, USA) with pore size about 0.5 μm) and diafiltration during SM fractionation. To analyze the data presented by the authors, the following basic concepts and notations should be used:

- Total solids content (TS), a value obtained by an officially recognized method of analysis [17];
- total protein nitrogen (TPN), non-casein nitrogen (NCN), non-protein nitrogen (NPN) content were determined based on the Kjeldahl method, using a multiplication factor of 6.38 to convert nitrogen to protein
- true protein content (TP) calculated as the difference between TPN and NPN;
- casein content (CN), the difference between TPN and NCN;
- serum protein content (SP) - difference between NCN and NPN.
- SP removal (%) was calculated as the ratio of the mass fraction of SP in the permeate to the mass fraction of SP in the initial OM multiplied by 100;
- total flow (LMH) - permeate flow rate in liters per square meter of filtration area per hour;
- calculation of membrane selectivity index with respect to casein or whey protein was performed according to recommendations [17], which in principle does not contradict with terminology accepted in RF [24]:

$$R_{ej}=1-C_p/C_f$$

where: $C_p/C_f$ is the ratio of the mass fraction of the substance in the permeate and in the initial separated system;
- transmembrane pressure was determined as:

$$TMP = 0.5(P_{in} - P_{out}) - P_{per}$$

$P_{in}$, $P_{out}$, $P_{per}$ – pressure at the inlet, outlet of the microfiltration unit and in the permeate line, respectively.

From the analysis of the experimental data [23] (Figure 5) the authors, most likely draw a conclusion about the significant influence of diafiltration level (DF) on the membrane permeability (expressed as LMH) at TMR 34.5 and 62.1 MPa, noting that at TMR = 103.4 MPa the LMH index remains almost unchanged, and increasing the DF level with
simultaneous increase in TMR leads to a significant drop in the membrane permeability $Q$, which is in agreement with the results obtained in another similar work [23].

![Graph showing LMH index at different TMP values](image)

**Fig. 4.** Average (3-fold repetition) values of the LMH index obtained from laboratory studies. Experiments were performed at 24 °C, concentration factor 4, the amount of diafiltration water, measured in % of the original volume of the separated system, $p \geq 0.95$ [23].

The conclusion made by the authors agrees well and is confirmed by the experimental data (Fig. 6) on the instantaneous flow obtained at TMR=34.5 kPa [23], which does not contradict the data of other researchers [13, 33, 34].

![Graph showing instantaneous flow at different DF%](image)

**Fig. 5.** Instantaneous flow at TMR = 34.5 kPa. Instantaneous fluxes were obtained by measuring permeate flux at different time intervals throughout the process and averaging over this time interval. All experiments were performed at 24 °C, concentration factor 4, amount of diafiltration water, measured as a percentage of the original volume of the separated system, $p \geq 0.95$ (Marella et al., 2021).

It is quite possible, that growth of TMR can promote increase of level of concentration polarization in a microfiltration apparatus near-membrane zone and decrease in LMH indicator, that is confirmed by data on selectivity of a membrane concerning WP (figure 7), received in the further experimental research [23].
Data on the removal of serum protein (SP), all experiments were performed at 24 °C, concentration factor 4, the amount of diafiltration water, measured as a percentage of the initial volume of the separated system [23].

As a result of processing of these experimental data, the authors obtained an empirical expression, using which it is possible to predict with sufficient accuracy the selectivity of the membrane under the conditions of the studies performed:

\[ R_{ej} = 58,705 - 0.0015 \times \text{TMP}^2 + 0.18 \times \text{DF} \]  

where: DF represents the amount (% wt.) of water added per volume of feedstock. It should be noted that these experimental studies did not consider influence of the circulation speed of the system to be separated in the membrane channel on the level of concentration polarization in the membrane zone and on the actual (depending on the conditions of the separation process) performance characteristics of the membrane. This justifies the necessity to carry out our experimental investigations of complex influence of TMP and V on permeability and selectivity of polymer membranes for SM microfiltration.

5 Permeate flux during microfiltration of skim milk on polymeric membrane

The results of analysis of these studies [34–36] allow to conclude that if the purity index of separation of micellar casein (MC) and whey proteins (WP) using ceramic membranes (0.1 µm) depends on their selectivity and SM circulation rate in the circuit of microfiltration apparatus, in case of application of its spiral wound modules (SWMs) and polymeric membranes - on properties of the complex "membrane + its contaminating layer", which is conditioned by lower speed of tangential flow in roll filter elements. The phenomenon of concentration polarization, respectively an increase in hydraulic resistance to permeate flow, as well as a decrease in TMP along the membrane channel are the main problems in microfiltration of dairy raw materials on polymeric mainly hydrophobic membranes, especially in spiral wound modules [37, 38].

Another aspect of the problem of improving the efficiency of microfiltration separation of skim milk is that SM is a very complex liquid polydisperse system where proteins are
represented by two main, different in molecular weight, fractions - MC and WP, which interacting with the membrane itself and/or with each other can form protein deposits on the membrane surfaces [39]. This eventually leads to a change in the permeability and selectivity of polymeric membranes. However, the question of the degree of influence of MC or WP on the intensity or mechanism of formation of such deposits remains open. Experimental data (Table 2) on the results of SM (skim milk) and CFSM microfiltration are given in [11] skimmed milk with casein preliminarily isolated on a ceramic membrane up to 0.02% wt.) using a polymeric membrane (0.3 μm) on a roll-type apparatus.

**Table 2. Mean values of microfiltration data for dairy raw milk (p=0.95): TS, dry matter content; CP, total protein; NCN, non-casein nitrogen; NPN, non-protein nitrogen; TP, true protein; CN, casein; SP, whey protein [11].**

<table>
<thead>
<tr>
<th>Name</th>
<th>TS</th>
<th>CP</th>
<th>NCN</th>
<th>NPN</th>
<th>TP</th>
<th>CN</th>
<th>CN/TP</th>
<th>SP</th>
</tr>
</thead>
<tbody>
<tr>
<td>SM</td>
<td>9.23</td>
<td>3.37</td>
<td>0.76</td>
<td>0.18</td>
<td>3.18</td>
<td>2.61</td>
<td>81.86</td>
<td>0.58</td>
</tr>
<tr>
<td>SM-permeat</td>
<td>6.23</td>
<td>0.54</td>
<td>0.51</td>
<td>0.18</td>
<td>0.36</td>
<td>0.027</td>
<td>7.42</td>
<td>0.33</td>
</tr>
<tr>
<td>SM-retentate</td>
<td>13.97</td>
<td>7.94</td>
<td>1.27</td>
<td>0.18</td>
<td>7.76</td>
<td>6.67</td>
<td>86.01</td>
<td>1.08</td>
</tr>
<tr>
<td>CFSM</td>
<td>6.47</td>
<td>0.76</td>
<td>0.74</td>
<td>0.18</td>
<td>0.57</td>
<td>0.02</td>
<td>3.48</td>
<td>0.55</td>
</tr>
<tr>
<td>CFSM-permeat</td>
<td>6.31</td>
<td>0.70</td>
<td>0.68</td>
<td>0.18</td>
<td>0.52</td>
<td>0.014</td>
<td>2.72</td>
<td>0.50</td>
</tr>
<tr>
<td>CFSM-retentate</td>
<td>6.43</td>
<td>0.82</td>
<td>0.79</td>
<td>0.18</td>
<td>0.64</td>
<td>0.32</td>
<td>4.96</td>
<td>0.60</td>
</tr>
</tbody>
</table>

From the analysis of the data presented, a conclusion was made about the predominant effect of casein on the pore blocking of the polymeric membrane during OM microfiltration separation [11]. In this case, based on the fact that the average conditional size of casein micelles is about 0.2 μm, and this indicator for serum proteins varies about 0.0036 μm [39], a sieve model of the polymer membrane surface blocking process (0.3 μm) is assumed: during the initial period of microfiltration, the smallest particles penetrate into the pores and partially attach to their walls, thereby reducing the average conditional diameter of these pores, which leads to increased selectivity of the membrane for casein particles and an increase in their concentration in the near-membrane zone. Over time, due to the low tangential flow rate of the separated system, a layer of casein micelle deposits forms on the membrane surface, which determines the actual permeability of the membrane to permeate. Since SM and CFSM differed only in casein content, in support of this conclusion the following data of the experimental determination of pure water flow (kg/m² hour) through the microfiltration membrane are given [11]: - initial value, 36.64; after CFSM separation, 35.12; after SM separation, 15.19; after washing and regeneration, 32.9.

However, in our opinion, from the data analysis (Table 2), first, it follows that during SM microfiltration almost all casein is retained on the polymer membrane, while only about 57% of serum proteins pass into the permeate, and this suggests their predominant participation in pore blocking. But, on the other hand, in CFSM partitioning, already about 91% of the SP and 70% of the remaining casein in the original polydisperse system passes through the membrane. However, such important, in our opinion, questions as the influence of the mineral complex of milk, in particular calcium phosphate, on the formation of a layer of deposits contaminating the membrane surface remain open [9], the pretreatment of raw materials, such as pasteurization regimes of the separated system on the kinetics of the microfiltration separation process [40]. It is obvious that from these positions the conclusion about the degree of influence of casein micelles on Jp value at OM microfiltration, made based on the analysis of the results of the experimental study [11], needs additional verification in part:

- justification of the choice of the average conditional diameter of pores of the polymeric membrane in the apparatus of the roll type;
- to study the effect of TMP and t parameters of the separated system on the permeability and selectivity of the selected membrane;
- determining the influence of diafiltration and concentration factor on the formation of a layer of deposits on the surfaces of the polymeric membrane.

6 Commercial use of micellar casein and whey proteins isolated by microfiltration of skim milk

It should be noted that despite the complexity of solving the problem of increasing the efficiency of the SM microfiltration process, commercial interest in the protein components of this type of raw materials in modern food production continues to grow. This is due to the unique functional as well as nutritional properties of both micellar casein concentrates (MCC) and whey proteins (WP). In most European countries and in the United States, there are no state standard requirements for MCC and WP; the American Dairy Products Institute (ADPI) only recommends regulating the ratio of MCC and WP content in microfiltered dairy products [41, 42]. And while in the USA WP is used mainly for protein enrichment of food products, in the EU microfiltered milk is used for standardization of milk in cheese production, thus increasing the yield of the finished product of loss of its functional properties and quality [19, 43]. And despite the need to adjust the traditional technology, microfiltration used to increase the content of solids and casein in cheese milk has shown itself as a competitive method in cheese production [44–47]. The increase of casein content in cheese milk from 3.2 to 10.9 % allows the pasteurization process to be carried out at a higher temperature (110-140 °C) for 10 seconds without noticeable changes in the kinetics of clot formation, which seems to be caused by changes in the mechanism of interaction of κ-casein and β-lactoglobulin [46]. But, on the other hand, such an adjustment of the classical cheese production technology leads to a change in organoleptic characteristics of the resulting product. For example, an attempt was made to produce Cheddar cheese from rehydrated powder (spray drying) MCC and containing only about 5% whey proteins. The resulting product with a low (about 6%) fat content not only had a bitter taste, but it lacked the characteristic or typical flavors of this type of cheese [47]. At the same time satisfactory results were shown by experimental production with the use of MCC analogues of mozzarella cheese and «clean-label» products of processed cheese [48, 49]. Another promising area of MSS application can be considered the production of protein bars, which have some organoleptic parameters higher than those of their analogues made of sodium caseinate, hydrolysates or whey protein isolates [50, 51]. The use of MCC is also of interest for making so-called Greek yoghurt [52], various drinks whose formulation is based on MCC with a reduced whey protein content of 95%, for example, in vanilla flavor (15 and 25 g of protein per 240 ml serving) [53]. Micellar casein concentrates are mostly in demand in dairy products due to their ability to bind large amounts of calcium, mild taste with a creamy hue, pure white color, etc. [54–56]. Milk beverage containing almost only MCC and low mass fraction of milk fat has the same whiteness as drinks with high fat content, is used as the main ingredient in the preparation of cream for coffee [57]. Of particular interest as a protein ingredient is a liquid, highly concentrated (protein content ≥18%) MCC (HC-MCC), which retains its flowability at temperatures above 22°C. Mixing it with skim milk and cream produces recombined concentrated milk (RCM) with high casein content and low whey protein content, which can be used in cheese production, but the temperature at which the clot is formed depends on a number of factors, such as pH of the medium, protein, calcium, salt, etc. [45, 46, 54]. In addition, during the baromembrane separation of SM, including diafiltration, the organoleptic characteristics of the resulting semi-finished products may vary significantly - from sweet and fragrant to «cardboard» [58, 59].
In cheese (cottage cheese) production, the separation of the clot from the fermented milk produces a stream containing, in ADPI terminology, «whey proteins» [51]. In publications, including purely scientific ones, many different and sometimes similar terms are used to name the liquid phase of raw milk containing proteins. Therefore, for the sake of certainty we consider it reasonable to use the terms «WPC» - milk whey protein concentrates (containing from 34 to 89% protein) and «WPI» - isolates (from 90% protein). But protein concentrates obtained because of thickening of skimmed milk microfiltration permeate - "CSP", which in principle does not contradict the recommendations of ADPI, as one of the authoritative scientific organizations in the dairy food industry and having a large number of publications in this area.

Since WP does not contain residues of starter cultures or enzymes and bleaching agents used in dairy products [60, 61], its use is especially relevant for the preparation of infant formulations. In addition, the composition of WP can be changed depending on the organization of the basic separation process, the membranes used, and the temperature during microfiltration. According to some researchers, the casein that passes through the MF membrane at low temperatures (about 4°C) is β-casein, which is in a dissolved state and is a monomer with a molecular weight of 25 kDa [62, 63]. However, in other works the results of OM microfiltration at 6°C using a ceramic (0.1 µm) membrane indicate the passage into the permeate of no more than 1% of β-casein, while for a polymeric (0.08 µm) membrane in a roll-type apparatus microfiltration at 7°C gives a value of 22% [64, 65]. Apparently, the selectivity of membranes with respect to β-casein (possibly other caseins as well) is determined both by the type of membrane used for OM separation and by the conditions of the process, including pretreatment of the separated system. In general, the use of WP, like the use of WPC and WPI, includes use both in the form of semi-finished products and in the formulation of ready-to-eat beverages, yoghurt, sour cream, confectionery, various baked goods, etc. However, the high level of protein purity and the absence of cheese by-products make WP primarily a versatile ingredient to produce ready-to-drink protein drinks and especially infant formulas.

It should be noted that studies of organoleptic characteristics, taste properties of MCC, WP, WPC and WPI produced with the use of membrane processes are still limited and require special experimental studies. Nevertheless, it can be considered that the necessary high-temperature processes, such as ultra-pasteurization, often cause the appearance of unpleasant sulfur flavors in milk beverages, the source of which are whey proteins [66]. But the exact values of their composition and concentration that cause the sulfur taste, as well as the purity, for example, of the MCC required to produce the same protein drinks without foreign flavors, have not yet been determined. This suggests that despite the fact that in our country and abroad adopted standard methods for determining the total protein content in the dry MCC, they do not provide information on the purity of the casein fraction, and it is this indicator, in our opinion, may be one of the key determinants of taste and functionality of the finished product. To correctly determine the amount of whey protein, i.e., the actual purity of MCC obtained after spray drying, HPLC (High performance liquid chromatography) analysis will probably be required, since after heat treatment of dairy raw materials due to partial denaturation of whey proteins and their binding to casein, the Kjeldahl arbitrage method may give a significant error for the purposes of the study [65].

7 Conclusion

Thus, based on the analysis of publications devoted to improving the efficiency of the membrane process of casein and whey proteins extraction from SM, the following conclusions can be made:
- Optimization of conditions for implementation of the general technological scheme of SM microfiltration separation should be carried out by sequentially finding solutions to particular problems in the area of pre-treatment of the treated raw materials and its process of subsequent baromembrane separation;
- For the microfiltration separation of SM almost equally possible to use both ceramic and polymeric membranes, the choice should be determined on the basis of economic indicators and technological requirements of the production of target products;
- Ceteris paribus for polymeric and ceramic membranes the main operating parameters of the SM microfiltration process are the value of TMP, V and t of the separated system in the channel of the baromembrane apparatus, whose values are determined by the performance characteristics of membranes and the type of device;
- For the development of recommendations for the practical application of the process of microfiltration separation (including diafiltration) SM requires the study to select by experiment the average nominal diameter of the membrane pores and its operating performance characteristics;
- Even though most of the experimental studies are performed using standard methods to determine the basic physico-chemical characteristics and functional indices of raw materials and semi-finished products, the data presented by the authors should be regarded as indicative, since the used milk raw materials, equipment and membranes, methods of organization and holding the process usually have their own characteristics which significantly affect the obtained results.

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**References**

3. Ö Coşkun, N Raak, M Corredig, Food Hydrocolloids, 137 (2023)


37. A. Ruiz-García, I. Nuez, Processes, 8, 6 (2020)


49. A. Kommineni, V. Sunkesula, C. Marella, L. E. Metzger, Foods, 11, 10 (2022)


53. K. G. Vogel, Protein Amount and Milk Protein Ingredient Effects on Sensory and Physicochemical Properties of Ready-To-Drink Protein Beverages. (2019)


