Determination of optimal values for the main parameters of ultrafiltration of curd whey enriched with natural polysaccharides

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Abstract. The current trend in the development of the human community is reduced to solving two main tasks: providing the population with full-fledged food and preserving the environment. Their solution is possible only on the basis of organizing the rational use of natural raw materials, which is hindered by insufficient study of such modern food production processes as ultrafiltration separation of liquid polydisperse systems. The aim of the study was to experimentally determine the optimal values of the driving pressure (DP) and the speed of circulation (V) of curd whey during its ultrafiltration through polymer membranes. The article presents the following research findings: the dependences of the permeability and selectivity of the UPM-20, UPM-50, Biomax 100 and Biomax 300 membranes during ultrafiltration of curd whey on the driving pressure and flow rate in the channel of the baromembrane apparatus are identified; the optimal values for DP=0.28÷0.36 MPa and V=0.25÷0.35 m/s of cassette-type devices for ultrafiltration of modified natural polysaccharides in curd whey are determined; the residual amount of total protein in permeate of curd whey is identified: with the use of UPM-20, UPM-50 membranes it amounts to 0.012÷0.013 %, with the use of Biomax 100 and Biomax 300 it amounts to 0.035÷0.088 %; the feasibility of the use of Pellicon 2 Biomax membenes with a threshold delay of 100 kDa and 300 kDa to obtain a highly purified permeate of curd whey is justified.

1. Introduction

The use of membrane methods in the processing of secondary dairy raw materials makes it possible to organize waste-free production and eliminate environmental pollution. The application of such methods in the modern food industry is a promising direction that opens up opportunities for creating a large range of dairy products, beverages, feed and other resource- and energy-saving technologies [1–7].

The purpose of the study was to provide theoretical justification and experimental determination of the optimal parameters of the technological process for obtaining permeate after ultrafiltration.

2. Methods and materials

In the course of the experimental study, we used curd whey (Table 1), obtained at JSC “Stavropol Dairy Plant” during the production of main products in accordance with GOST 31534-2012 [8]. The main
The physical and chemical characteristics of the original curd whey and the curd whey enriched with plant extract (Jerusalem artichoke juice) are presented in Table 1.

| Indicator                                      | Curd whey | Original | Enriched |
|------------------------------------------------|-----------|----------|----------|
| The content of dry substances, %, not less than |           | 6.5      | 6.5      |
| Including:                                     |           |          |          |
| Lactose                                        | 5.1       | 5.1      |          |
| Protein                                        | 1.0       | 0.7      |          |
| Fat                                            | 0.1       | 0.1      |          |
| Mineral substances                             | 0.3       | 0.7      |          |
| Acidity, ° Т                                   | 45        | 42       |          |
| pH indicator                                   | 5.0       | 5.1      |          |
| Density, kg/m³                                  | 1023      | 1024     |          |
| Optical density of 10 % solution                | 0.26      | 0.2      |          |

The results of data analysis [9] obtained in the course of theoretical studies show that currently, the technology of ultrafiltration separation of curd whey uses polymer membranes of such brands as UAM 150, UAM 500, UPM 50, HyStream, with a threshold delay of 20-50 kDa (Table 2).

| Main parameters | Membrane type | UPM-20 | UPM-50 | HyStream | UAM-150 | UAM-500 |
|-----------------|---------------|--------|--------|----------|---------|---------|
| Pressure, MPa   |               | 0.1-0.4| 0.1-0.4| 0.2-0.5  | 0.2-0.45| 0.2-0.45|
| Threshold delay, kDa |            | 15-20 | 40-50  | 45-50    | 20-25   | 50-55   |
| Temperature, °C |               | 5-45   | 5-45   | 5-50     | 5-50    | 5-50    |
| pH of the cleaning medium |        | 2-10   | 2-10   | 2-12     | 2-12    | 2-12    |
| Service life, hour |            | to 3000| to 3000| to 4000  | to 4000| to 4000 |
| Price for 1 m², Rub. |         | from 500| from 500| from 500 | from 350| from 350|
| Acquisition of filter elements                  | basic    | order  | order   | basic   | basic   |

Permeate obtained as a result of separation of curd whey using the above-mentioned membranes is of high quality with a residual protein content of less than 0.02 % and dry substances of no more than 5.1 % (Table 3).

However, for further nanofiltration concentration, it is sufficient to conduct preliminary ultrafiltration using membranes with a delay threshold greater than 50 kDa. The rationale for this can be the following: the lowest cost of membranes, reducing the cost of washing membrane equipment, increasing the service life of membrane modules, improving the efficiency of existing equipment samples due to reducing the time for the stages of fractionation and washing, a sufficient degree of purification of permeate from protein components.
Table 3. Physical and chemical parameters of permeate of milk whey obtained using membranes with a threshold delays of 50kDa, (p=0.95).

| Indicator name                          | Permeate of curd whey |
|-----------------------------------------|-----------------------|
| Weight fraction of dry substances, including: | 5.1±0.1              |
| Protein, %                              | 0.01±0.001            |
| Lactose, %                              | 4.4±0.1               |
| Mineral substances, %                   | 0.6±0.05              |
| Fat, %                                  | 0.1±0.1               |
| Active acidity, pH                      | 4.5±0.1               |
| Titrable acidity, °T                    | 65±2.0                |
| Density, kg/m³                          | 1022±2.0              |

Taking into account the standardized delay thresholds for baromembrane separation, Millipore Pellicon 2 Biomax cassette type membranes with a delay threshold of 100 and 300 kDa with the characteristics shown in table 4 were selected for ultrafiltration of curd whey enriched with plant extract in order to obtain permeate.

Table 4. Main characteristics of Pellicon 2 Biomax polymer membranes.

| Main parameters                      | Membrane type |
|--------------------------------------|---------------|
|                                      | Biomax 100    | Biomax 300   |
| Pressure, MPa                        | 0.4-0.5       | 0.4-0.5      |
| Threshold delay, kDa                 | 95-100        | 295-300      |
| Temperature, °C                       | 4-50          | 4-50         |
| pH of cleaning medium                | 1-14          | 1-14         |
| Service life, hour                   | to 5000       | to 6000      |
| Price for 1 m², Rub.                 | from 500      | from 500     |
| Acquisition of filter elements       | order         | order        |

Based on the data presented in table 4, we can conclude that the parameters such as service life, pH of the cleaning medium, and temperature of the Pellicon 2 Biomax membrane are superior to the UPM, UAM, and HyStream membranes, while the cost per 1 m² is the same. Since there is not enough information in the open press about the results of industrial application of these membranes for ultrafiltration of curd whey enriched with plant extract in order to obtain permeate for further nanofiltrational concentration, this makes it necessary to conduct our own research.

For practical confirmation of the effectiveness of polymer membranes with a high threshold delay, a comparative experiment of ultrafiltrational separation of curd whey enriched with plant extract was conducted on the UPM 20 membranes (threshold delay is 15-20 kDa), UPM 50 (threshold delay is 45-50 kDa), Biomax 100 (threshold delay is 95-100 kDa) and Biomax 300 (threshold delay is 295-300 kDa). Experimental studies were performed using the KrosFlo® Research II TFF laboratory tangential filtration unit. Its design features allow performing ultrafiltration of liquid polydisperse systems using a cassette with polymer membrane elements with a total active filtration area of 0.01 m², controlling the circulation speed at a constant pressure, conducting the process at different pressure values and a constant speed of movement of the shared system in the channel of the membrane apparatus. The effectiveness of the process of ultrafiltration of curd whey was evaluated by the degree of increase (in comparison with the whey purified by the traditional method) of membrane permeability while maintaining their selectivity for protein substances acceptable from the technological point of view.

3. Results and discussion
The analysis of the literature data [10, 11] and the results of our own experimental studies allowed us to
conclude that when obtaining high-quality permeate by ultrafiltration of curd whey enriched with plant extract, the key factors that can be used to determine the effectiveness of the influence on permeability and selectivity when using polymer membranes are the driving pressure \( P \) (MPa) and the flow rate \( V \) (m/s) in the device channel.

The results of experimental studies presented as graphical dependencies of the form \( Q=f(P) \) and \( \phi=f(P) \) are shown in figures 1 and 2.

It was found that at fixed values of the parameters \( T, t, v \) and \( C_{c.v} \), the permeability \( Q \) of all membranes increases significantly at a pressure \( P = 0.44-0.49 \) MPa. An increase in the driving pressure above 0.5 MPa does not lead to a significant increase in \( Q \), and the selectivity of the membranes is reduced to \( \phi = 95-96\% \). This can be explained by the fact that at this pressure, most likely, the deformation of the membrane structure can occur, which becomes one of the reasons for the intensive pore blocking process. This interpretation is supported by the fact that the cleaning time of membranes exposed to driving pressure above 0.5 MPa increased by an average of 18-22% in comparison with the duration of this process for membranes operating at \( P \leq 0.5 \) MPa. The average value of the permeability \( Q \) of the BioMax 100 and Biomax 300 membranes for a full cycle of ultrafiltration of curd whey enriched with plant extract was 15-20 kg/m²•h higher than this indicator for the UPM-20 and UPM-50 membranes. This is due to the fact that the threshold delay for UPM-20 and UPM-50 membranes is approximately 2-2.5 times lower than for Biomax 100 and Biomax 300. But since the porosity of the UPM-20 and UPM-50 membranes is higher, there is no significant difference in the permeability of all the membranes.

![Figure 1](image_url)

**Figure 1.** The dependence of \( Q \) membranes permeability (▲-UPM 20, ■ - UPM 50, ♦ - Biomax 100, ●- Biomax 300) on the driving pressure value \( \Delta p \) (\( t=10-12^\circ \text{C}, v=0.1-0.3 \) m/s, \( C_{c.v} = 8-8.2\% \)) for ultrafiltration of curd whey enriched with plant extract.

An indirect confirmation of this explanation can be the fact that the selectivity of all membranes at the beginning of the ultrafiltration process of curd whey enriched with plant extract differs by an average of 20-23%, and after 110-125 minutes this difference decreases to 12-14%. By the end of the ultrafiltration process, the difference in selectivity for both
membranes does not exceed 7%. This means that the Biomax 100 and Biomax 300 membrane brands, which have larger pores, are subject to more intensive blocking of the pore space by protein molecules in the initial period of operation, in comparison with the UPM-20 and UPM-50 membrane brands. In this case, the formation of “primary” layers occurs not only on the surface of the membrane, but also in the largest pores, which can get not only individual protein molecules, but also particles of mechanical impurities.

![Figure 2](image)

**Figure 2.** The dependence of \( \varphi \) membranes selectivity (▲ - UPM 20, ▼ - UPM 50, ♦ - Biomax 100, ● - Biomax 300) on driving pressure value \( \Delta p \) (\( t=10-12^\circ C \), \( v=0.1-0.3 \) m/s, \( Cc.v.=8-8.2\% \)) for ultrafiltration of curd whey enriched with plant extract.

The analysis of experimental data reflecting the dependence of the selectivity of the used types of membranes on the driving pressure in the channel of the membrane apparatus showed that for fixed values of the parameters \( T \), \( t \), \( V \) and \( Cc.v. \) the \( \varphi \) parameter of both membranes increases significantly with increasing pressure to \( P = 0.40-0.45 \) MPa. An increase in the driving pressure above 0.45 MPa leads to a significant increase in the selectivity of the membranes to \( \varphi = 98-99\% \). At the same time, the increase in permeability is only 5–7%, with an increase in energy consumption for carrying out both the process of ultrafiltration of curd whey enriched with plant extract, and subsequent cleaning of the membranes.

It should be taken into account that in the presence of a tangential flow of a shared liquid system, the permeability \( Q \) and selectivity \( \varphi \) of the membranes, in addition to the driving pressure, are significantly affected by the value of the circulation velocity \( v \) in the baromembrane apparatus circuit. Graphic dependencies of the type \( Q=f(v) \) \( \varphi=f(v) \) for ultrafiltration of curd whey enriched with plant extract are shown in figures 3 and 4.
Figure 3. The dependence of $Q$ membranes permeability (▲-UPM 20, ■-UPM 50, ♦-Biomax 100, ●-Biomax 300) on the flow rate $v$ of curd whey enriched with plant extract $p$ ($t=10-12^\circ C$, $p=0.3-0.4$ MPa, $C_{ca} = 8-8.2\%$) in the channel of the membrane apparatus.

Figure 4. The dependence of $\varphi$ membranes selectivity (▲-UPM 20, ■-UPM 50, ♦-Biomax 100, ●-Biomax 300) on the flow rate $v$ of curd whey enriched with plant extract $p$ ($t=10-12^\circ C$, $p=0.3-0.4$ MPa, $C_{ca} = 8-8.2\%$) in the channel of the membrane apparatus.

Analysis of the obtained dependences of the permeability and selectivity of the membranes on the flow rate of circulation in the membrane channel of the device, with fixed other parameters of the process, suggests that all types of membranes can be used for ultrafiltration of curd whey enriched with plant extract. It is possible that the choice of one of them will be determined primarily by the technology of further processing of permeate, that is, the requirements for the content of the residual amount of...
protein substances in it. An important technological aspect of choosing a particular type of membrane for ultrafiltration of curd whey enriched with plant extract may be their selectivity at different temperature parameters of the separation process.

Thus, for ultrafiltration of curd whey enriched with plant extract, the expected permeability of BioMax 100 and Biomax 300 membranes is higher by 5-8 kg/m2•h than that of UPM-20 and UPM-50, and the selectivity is lower on average by 1-2%, respectively.

An important technological factor in the baromembrane separation of liquid polydisperse systems is the physical and chemical parameters (table 5) and the presence of nitrogenous substances in permeate (table 6). In accordance with the standard CXS 331-2017 (Dry permeates from raw milk), the maximum nitrogen content in dry whey permeate should not exceed 1.1%.

Table 5. Physical and chemical parameters of permeates of curd whey enriched with plant extract obtained using ultrafiltration membranes with a threshold delay of 100 kDa and 300 kDa (p=0.95).

| Indicator name                                        | Permeate obtained using ultrafiltration membranes with a 100kDa threshold delay | Permeate obtained using ultrafiltration membranes with a 300kDa threshold delay |
|-------------------------------------------------------|---------------------------------------------------------------------------------|--------------------------------------------------------------------------------|
| Weight fraction of dry substances, including:         | 5.2±0.1                                                                         | 5.3±0.1                                                                         |
| Nitrogenous substances, %                             | 0.035±0.001                                                                     | 0.088±0.001                                                                     |
| Lactose, %                                            | 4.1±0.1                                                                         | 4.1±0.1                                                                         |
| Mineral substances, %                                 | 0.7±0.05                                                                        | 0.8±0.05                                                                        |
| Fat, %                                                | 0.1±0.1                                                                         | 0.1±0.1                                                                         |
| Active acidity, pH                                     | 4.5±0.1                                                                         | 4.5±0.1                                                                         |
| Titrable acidity, °T                                   | 65±2.0                                                                          | 65±2.0                                                                          |
| Density, kg/m³                                         | 1022±2.0                                                                        | 1022±2.0                                                                        |

Table 6. The results of the analysis of curd whey samples, permeate and retentate enriched with plant extract for the total amount of protein using the Kjeldahl method.

| Sample name               | UPM-20 | UPM-50 | Biomax 100 | Biomax 300 |
|---------------------------|--------|--------|------------|------------|
| Enriched curd whey        | 0.641  | 0.641  | 0.641      | 0.641      |
| Retentate                 | 10.1   | 9.8    | 8.9        | 7.6        |
| Permeate                  | 0.012  | 0.013  | 0.035      | 0.088      |

The analysis of permeate of curd whey samples enriched with plant extract for the total amount of protein by the Kjeldahl method showed that in the process of ultrafiltration using UPM-20 and UPM-50 membranes, about 0.012-0.013% of protein particles contained in the original whey remain on the BioMax 100 and Biomax 300 membranes: 0.035-0.088 %.

4. Conclusion

The results of the conducted research prove the dependence of the permeability and selectivity of UPM-20, UPM-50, Biomax 100, Biomax 300 membranes in the ultrafiltration of the curd whey enriched with plant extract on the driving pressure and flow rate in the channel of baromembrane apparatus. The optimum values for DP =0.28-0.36 MPa and V=0.25-0.35 m/s for cassette-type devices used for ultrafiltration of curd whey enriched with plant extract were identified; the residual amount of total protein in the permeate of curd whey enriched with plant extract was determined: when using UPM-20 and UPM-50 membranes it amounts to 0.012-0.013 %, when using Biomax 100 and Biomax 300 it amounts to 0.035-0.088 %; the feasibility of using Pellicon 2 Biomax membranes with threshold delay of 100 kDa and 300 kDa for obtaining highly purified permeate of curd whey enriched with plant extract.
was justified. Taking into account the technological requirements for obtaining ultrafiltrate of curd whey enriched with plant extract, it can be said that the use of Pellicon 2 Biomax membranes with threshold delay of 100 and 300 kDa is advisable to implement in the technology for obtaining highly purified permeate and subsequently in experimental studies of combined baromembrane purification of curd whey.

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