

A new technique for removing a gel layer from the membrane surface during the ultrafiltration concentration of pectin extract has been considered. An experimental setup has been designed and a procedure for processing the results of studying the process of the concentration of pectin extracts has been devised, using a technique of removing a gel layer from the membrane surface. The paper reports the results of studying the application of a vibration stirring technique to eliminate the gel layer and its effect on membrane performance. Mathematical models have been built and the modes to perform the process of the ultrafiltration of pectin extract by using vibration stirring have been determined.

The graphical dependences of the quantitative and qualitative characteristics of pectin concentrate (the concentration of pectin substances and dry substances in the concentrate and permeate) have been given that depend on the input parameters of the temperature and pressure of the ultrafiltration concentration process. An analysis of the given characteristics has made it possible to establish the rational input parameters for the process of concentrating pectin extracts. The rational operating parameters of the process of concentrating pectin extracts when using a new technique for eliminating the gel layer are the filtration pressure of 0.4–0.5 MPa, a temperature of 35...45 °C, a duration of 1.5–2.0 hours, and a vibration stirring speed of 1.5–1.7 m/s.

This study was performed with the aim of intensifying the membrane concentration of pectin extracts, improving the technical level of the concentration process, and implementing the developed technique under industrial conditions. Based on the research results, the expediency of using a new technique for removing the gel layer has been established. Further implementation of these results in the food and processing industry would make it possible to apply them in the production of a wide range of pectin products

Keywords: pectin extract, membrane treatment, polarization layer, ultrafiltration concentration, vibration stirring

DEVELOPING A TECHNIQUE FOR THE REMOVING OF A GEL LAYER IN THE PROCESS OF MEMBRANE TREATMENT OF PECTIN EXTRACT

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1. Introduction

The deterioration of the environmental situation in many countries, as well as the associated pollution of the environ-

ment and food products with toxic substances, necessitates an increase in the production of pectin and pectin-based products as natural detoxifiers. Pectin is recognized by the World Health Organization as a valuable food product that

can form complexes and remove heavy metals and long-living isotopes of cesium, strontium, yttrium, etc. from the human body [1].

At the same time, the high efficiency of the use of membrane processes in various industrial technologies, as well as their environmental friendliness, contributed to the rapid growth of membrane technology, as well as the associated increase in funding in all developed countries [2]. The membrane methods for the separation of liquid mixtures are increasingly used in the world [4].

At present, the creation of new promising food products aimed at improving the health of the population increases the demand for pectin as a food additive. Therefore, it is a relevant task to devise and examine new technological and technical solutions, so that the use of pectin, first, would expand the range of pectin-containing products and, second, could reduce the cost of its acquisition [5].

2. Literature review and problem statement

Work [6] investigates the technological parameters of the ultrafiltration (UF) concentration of beet pectin extracts and the quality indicators of the obtained pectin concentrate received with the participation of a stirring process. But the issues of intensifying the process through the selection of the technological parameters for the process temperature and pressure have not been fully resolved, due to a wide variety of membranes, in particular, polymeric, which can be used under different conditions of ultrafiltration with varying degrees of resistance against the technological environment [7].

While the process of UF treatment is widely used in the processing industry during the production of plant concentrates, the technical support of the UF process for treating pectin-containing raw materials has some special features [8].

At the same time, the difficulty in the development of ultrafiltration methods for pectin extract processing is the low performance of UF membranes, which is due to the physical and chemical properties of pectin substances [9]. In this case, there is practically no development of membrane equipment of small-scale productivity, which is associated with the insufficient number of technical solutions for intensifying the process of UF concentration [10].

The shortcoming of the process of membrane methods for concentrating pectin extracts is the formation of a gel layer at the selective surface of a polymeric membrane. This makes the processes of pectin extract treatment long and economically ineffective [11, 12]. To eliminate the formation of a gel layer, the design of the developed membrane module includes devices for the turbulization of the flow of separated pectin extracts, in particular, the hydro-mechanical processes [13].

The efficiency of the process of UF concentration of pectin substances is largely affected by the concentration of dry substances in the solution being processed [14]. With an increase in the mass fraction of dry substances in the pectin extract, the rate of UF concentration decreases. In addition, under conditions of the prolonged concentration of pectin extracts with a high dry matter content, the conditions for the emergence of a gel layer, which affects the efficiency of the process, are significantly improved [15].

Although literary sources describe a significant number of methods and devices with a hydro-mechanical effect to eliminate the formation of a gel layer at the surface of the membranes, their potential is far from being exhausted [16]. Therefore, it appears promising to envisage and study the new measures to combat the polarization gel layer at the surface of a polymeric membrane. In particular, it is promising to use the method of vibrational stirring of the flow of the medium being separated, which would form an effective turbulization of the process of UF concentration of pectin extracts [17]. All the above confirms the feasibility of experimental research and analysis in this field to improve the quantitative and qualitative indicators of the pectin concentrate and provide the consumer with high-quality pectin products, in particular, pectin concentrates.

3. The aim and objectives of the study

The work aims to develop a process for the UF concentration of pectin extracts using a vibration stirring technique to prevent the formation of a gel layer at the surface of a semi-permeable membrane.

To achieve the specified goal, the following tasks must be solved:

- to develop a procedure for studying the process of UF concentration of pectin extracts using a new technique for eliminating the gel layer;
- to determine the factors affecting the process of UF concentration of pectin extracts using the technique of vibration stirring;
- to determine the rational parameters of the ultrafiltration concentration of the pectin extract for the developed technique for eliminating the gel layer.

4. Materials and methods to study the UF concentration of pectin extract using vibration stirring

4.1. Scheme of the experimental setup with a vibration turbulator and its operation principle

The study into the choice of the rational parameters for the UF concentration of pectin extracts was performed at the scientific laboratory “Nanotechnology of Food Products” of the Kharkiv State University of Nutrition and Trade (Ukraine) [18]. To improve the process of the ultrafiltration treatment of pectin extract, a technique for eliminating the gel layer was proposed, which involves both the physical and hydro-mechanical processes.

To accelerate the process of the UF concentration of pectin extract, a technique for eliminating the gel layer was proposed implying the vibration stirring of processed food liquids directly near the surface of the semi-permeable UF membranes. In this case, the acceleration of the ultrafiltration process is achieved due to the combined effect exerted on the gel by the turbulization of flows of the treated liquid and the hydraulic impact of the liquid against the surface of a UF membrane [19, 20].

Fig. 1 shows a schematic diagram of the ultrafiltration experimental setup based on a membrane module with a vibration turbulator.

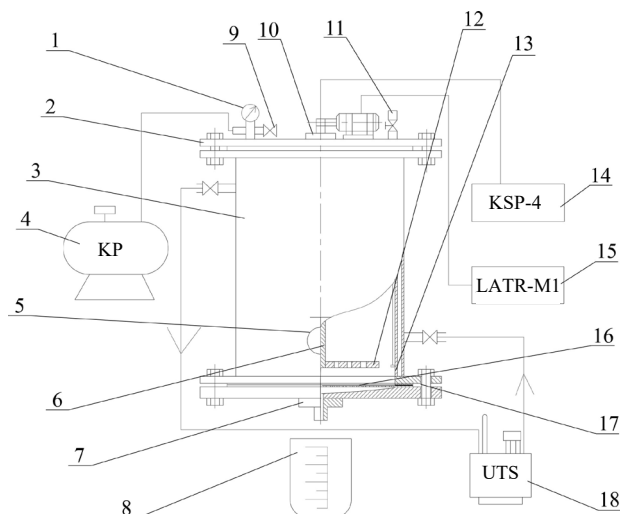


Fig. 1. Structural diagram of the experimental ultrafiltration setup with a vibration plate: 1 – pressure gauge; 2 – upper cover; 3 – the body of a membrane apparatus; 4 – compressor; 5 – a valve for diverting an ultrafiltration concentrate; 6 – vibration turbulator; 7 – lower cover; 8 – a container for permeate collection; 9 – a valve for pressure supply and discharge; 10 – sealant; 11 – electric drive; 12 – a valve to supply pure solvent; 13 – rubber sealant; 14 – thermocouple; 15 – potentiometer ksp-4; 16 – ultrafiltration membrane; 17 – a lining; 18 – ultrathermostat (UTS)

The ultrafiltration laboratory setup is based on filter module 1, made of stainless steel, with an internal volume of 5 liters. Flanges are welded to the lower and upper bases of the module, to which the upper 2 and lower 7 covers are attached by tie bolts. The tightness of the covers' fastening is provided by rubber gaskets 13. Semi-permeable ultrafiltration membrane 16 is fixed to the lower cover, which has radially drilled through holes to discharge the permeate. Pressure gauge 2 is installed in the top cover to control the process pressure, which ensures maintaining the required filtration pressure at complete tightness; thermocouple 18 is connected to the potentiometer 15 KSP-4. On the top cover, there is also a valve for supplying and releasing pressure 9, through which, with the help of compressor 4, the required pressure is formed inside the module. The top cover is also equipped with a valve (12) for supplying pure solvent during the diafiltration purification of the concentrate. To study the turbulization of the process, a vibration turbulator (6) is placed inside the module, the operation of which is supported by an electric drive (11). The membrane apparatus has a thermal jacket, the water temperature in which is regulated by an ultra-thermostat (18). In the lower part of the filter module, there is a valve (5) for draining the ultrafiltration liquid concentrate while the permeate is collected through a fitting in the lower cover to a container (8). The working surface of the setup's membranes is 0.024 m^2 .

The general view of the experimental ultrafiltration setup during the process of concentrating the pectin extract under laboratory conditions is shown in Fig. 2.

The UF process at this setup is as follows. The required amount of liquid is poured into the membrane apparatus, which is processed (the pectin extract). Using a compressor (4), the required pressure is created and the UF concentration

is conducted. To study the effect of turbulization on the UF concentration process, an electric drive (11) is turned on, which sets a vibration turbulator (6) into motion. During the UF concentration, permeate passes through the membrane and is collected by means of a funnel-shaped surface in the lower cover (7) to a measuring vessel (8). In the case of reaching the required concentration factor, the UF concentrate is taken from the filter apparatus using a valve (5).



Fig. 2. Experimental setup in operation

During the experiments, the required pressure in the over-membrane space of the UF module was created using a compressor and changed from 0.2 to 0.6 MPa. The motion speed of a PE flow in the intramembrane channel when using vibration stirring was $0.5 \dots 2.0 \text{ m/s}$. After 20 minutes, when the ultrafiltration rate became constant, the amount of filtrate that passed through the membrane over 10 minutes was measured. The PS amount in the concentrate was determined according to the volumetric method. The amount of dry matter was determined by refractometry [15].

To plot the dependence of the performance of UF membranes on the vibration stirring rate, all experiments were repeated five times. The research results were subjected to statistical treatment by the least-square method to determine the error in the data obtained. The experimental data were processed by statistical modeling methods using the Microsoft Excel 2007 spreadsheet processor. The differences were considered statistically significant at the confidence level of $A=0.95$.

4. 2. Modeling the process of the UF concentration of pectin extract

Under a vibration stirring mode, an electric motor connected to a mixer located inside the UF module was switched on. The necessary hydro-dynamic conditions at the surface of the semi-permeable membrane were created using the laboratory autotransformer LATR.

To determine the qualitative characteristics of the process of the UF concentration of pectin extract, mathematical modeling was proposed using the method of planning a factorial experiment [21]. In this case, according to the plan of the factorial experiment, 10 experiments were conducted, the results of which are given in chapter 5.

As a result of mathematical modeling, regression equations (1) to (3) were obtained, which allow a more detailed study of the factors affecting the process of the UF concen-

tration of pectin extract. At the same time, the equations given below have made it possible to determine the rational parameters for obtaining the UF concentrates of pectin extract based on the performance values of UF membranes of the PAN type.

The mathematical modeling and construction of the response surface of the qualitative parameters of the UF process involved the problem-oriented mathematical software package Mathcad 15 using a PC.

At the same time, to study the process of the UF concentration of pectin extract, the following main input parameters of the process were selected: t is the temperature of the UF concentration, °C; P is the filtration pressure, MPa; τ is the processing time, s².

The equation for the PS concentration in pectin concentrate during the UF concentration when using a vibration stirring technique takes the following form:

$$C_{PS} = 3.718 + 0.028 \cdot t - 10.804 \cdot P - 1.251 \cdot \tau - 6.362 \cdot 10^{-4} \cdot t^2 + 9.063 \cdot P^2 + 0.145 \cdot \tau^2 + 0.085 \cdot t \cdot P + 7.143 \cdot 10^{-3} \cdot t \cdot \tau + 1.429 \cdot P \cdot \tau \tag{1}$$

The equation for the DM content in pectin concentrate during the UF concentration when using a vibration stirring technique takes the following form:

$$C_{d.m.c.} = 12.37 - 0.065 \cdot t - 13.589 \cdot P + 4.592 \cdot \tau - 6.004 \cdot 10^{-4} \cdot t^2 - 4.152 \cdot P^2 - 0.908 \cdot \tau^2 + 0.492 \cdot t \cdot P + 0.01 \cdot t \cdot \tau + 1.786 \cdot P \cdot \tau \tag{2}$$

The equation for the DM content in pectin permeate during the UF concentration when using a vibration stirring technique takes the following form:

$$C_{d.m.p.} = -1.082 + 0.092 \cdot t + 6.982 \cdot P + 2.267 \cdot \tau - 1.484 \cdot 10^{-3} \cdot t^2 - 23.973 \cdot P^2 - 0.634 \cdot \tau^2 + 0.303 \cdot t \cdot P + 0.016 \cdot t \cdot \tau + 1.571 \cdot P \cdot \tau \tag{3}$$

5. Results of studying the effect of a vibratory stirring technique on the process of the UF concentration of pectin extract

Performance is one of the main characteristics of UF membranes. One distinguishes the initial performance of the membranes, that is, the performance of new membranes in the initial period of their operation [22], and the actual performance, which characterizes the operation of the membranes under continuous operation [10].

The results of our experiments on the effect of the process parameters on the performance of the semi-permeable membranes PAN-50 and PAN-100 are given in Tables 1, 2.

An analysis of the data obtained shows that an increase in the pressure during the UF concentration of PE over 0.4–0.5 MPa is unreasonable since this does not lead to a significant increase in the productivity of both types of membranes. In addition, the use of a vibrating perforated disk makes it possible not only to increase significantly the performance of the UF membranes by preventing the formation of a gel layer at their surface but also to reduce the working pressure in the head channel of the UF module.

Table 1

The results of experiments on the UF concentration of pectin extract using the membrane PAN-100

$t, ^\circ\text{C}$	P, MPa	$\tau, \text{s} \cdot 10^{-2}$	$C_{PS}, \%$	$C_{d.m.c.}, \%$	$C_{d.m.p.}, \%$
60	0.2	4	2.5	6.5	2.8
60	0.6	0.5	3.0	6.6	3.0
60	0.2	0.5	2.1	4.2	1.8
60	0.6	4	5.4	9.8	4.7
20	0.6	2	1.9	6.1	1.7
20	0.4	4	2.0	6.5	1.9
20	0.4	0.5	1.6	4.5	1.6
20	0.2	2	1.5	6.6	2.1
40	0.2	4	2.3	6.8	2.3
40	0.4	2	2.1	7.4	3.4

Table 2

The results of experiments on the UF concentration of pectin extract using the UF membrane PAN-50

$t, ^\circ\text{C}$	P, MPa	$\tau, \text{s} \cdot 10^{-2}$	$C_{PS}, \%$	$C_{d.m.c.}, \%$	$C_{d.m.p.}, \%$
60	0.2	4	4.0	4.7	2.2
60	0.6	0.5	1.3	4.6	2.3
60	0.2	0.5	1.0	3.6	1.2
60	0.6	4	4.9	2.7	3.2
20	0.6	2	2.8	2.8	0.1
20	0.4	4	3.3	3.2	0.3
20	0.4	0.5	1.2	2.0	0.6
20	0.2	2	1.1	2.5	0.8
40	0.2	4	3.5	4.6	2.4
40	0.4	2	1.9	4.7	2.1

An increase of the PE temperature at their UF concentration above 45...55 °C is impractical since there is no significant increase in the productivity of semi-permeable membranes. In addition, it should be taken into consideration that too high temperatures lead to the undesirable biochemical transformations of pectin substances and a decrease in their functional properties.

Fig. 3 shows a graphical dependence of the effect of the rate of pulsating flows of the vibration PE stirring during their UF treatment on the performance of UF membranes of the PAN type. The graphical dependences show that a change in the performance for both PAN-50 and PAN-100 membranes is of the same nature. With an increase in the speed of pulsating flows up to $U=1.5-1.7$ m/s, an increase in the membrane productivity is observed, by 1.5–1.6 times.

The value of the qualitative PC characteristics (the PS concentration in PC and the content of dry matter (DM) in the concentrate and permeate), obtained under the mode with vibration stirring, depends on many technological parameters. But a significant difference in the characteristics is observed at different values of temperature and pressure of the filtration process (Fig. 4, 5).

The value of the DM content in PC changes with an increase in the temperature and pressure parameters of the UF concentration process. Thus, the maximum values of the DM content in the concentrate and permeate are: $C_{d.m.c.}=9.8\%$ and $C_{d.m.p.}=5.3\%$, at temperatures of 50...60 °C and a process pressure of 0.5–0.6 MPa; the minimum values: $C_{d.m.c.}=5.8\%$ and $C_{d.m.p.}=2.1\%$, at temperatures of 20...25 °C and a pressure of 0.2–0.3 MPa.

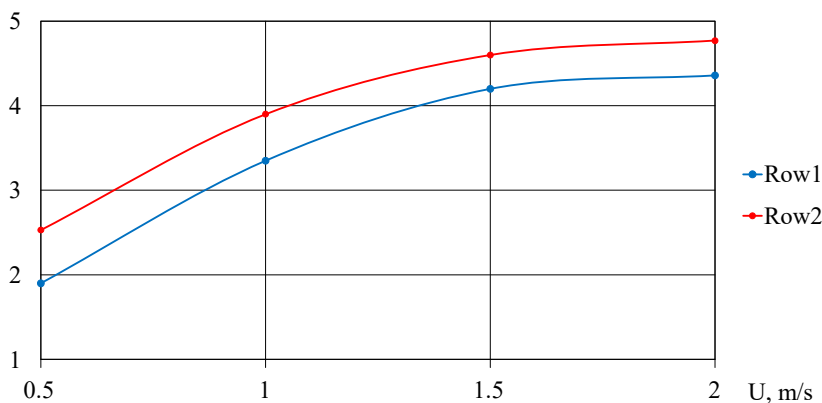


Fig. 3. Dependence of the performance of UF membranes of the PAN type on the rate of vibration stirring in the process of membrane separation of pectin extracts at a temperature of 50 °C and a pressure of 0.4 MPa: 1 – membrane PAN-50; 2 – membrane PAN-100

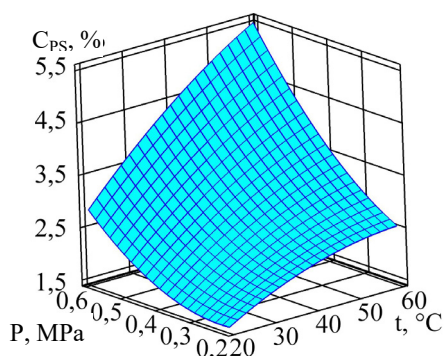


Fig. 4. Dependence of a PS concentration change on the temperature (*t*) and pressure (*P*) of the UF PE concentration process for PAN-100

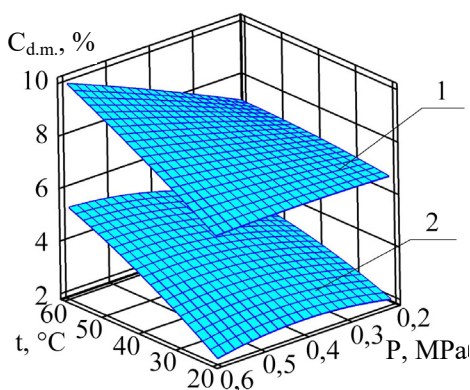


Fig. 5. Dependence of changes in the DM content on the temperature (*t*) and pressure (*P*) in the UF concentration process for PAN-100: 1 – in concentrate; 2 – in permeate

The data presented demonstrate that the change in the values of the PS concentration in pectin concentrates obtained by the UF concentration, and the content of DM in the concentrate and permeate for the PAN-100 membrane, confirms the complex nature of nonlinear dependences. Thus, the dependence of the PS concentration in PC has the values of maxima at the points of application of the maximum parameters and a descending character with a

decrease in the parameters of the UF concentration process. Thus, the maximum values of the PS concentration in the concentrate $C_{PS}=5.4\%$ are observed at a temperature of 60 °C and a process pressure of 0.5–0.6 MPa, the minimum value of $C_{PS}=1.6\%$ – at a temperature of 20 °C and a pressure of 0.2 MPa. At certain rational values of the temperature and pressure of ultrafiltration, the concentration of pectin substances in PC is: $C_{PS}=3.5-4.0\%$.

The decrease in the productivity of semi-permeable membranes with an increase in the duration of the process can be explained by the intensive formation of a gel layer of high molecular-weight substances at their surface, which significantly slows down the process of the UF concentration of PE. Under the mode with vibration stirring, the slow character of the decrease in the productivity of UF membranes is due to the effect of vibration turbulization on the thickness of the gel layer formed at their selective surface.

6. Discussion of results of determining the rational parameters of the UF pectin extract concentration for the developed technique of gel layer elimination

A comparative analysis of the calculated and experimental characteristics of the obtained PC showed a complete match between their values in terms of productivity and qualitative characteristics for both types of semi-permeable membranes.

Using the proposed mathematical model, the conditions for the UF concentration process were determined when using two types of semi-permeable membranes of the PAN type. The research results, presented in Tables 1, 2 and in Fig. 4, 5, have made it possible to determine the operating parameters for the process of the ultrafiltration concentration of pectin extracts using a vibration stirring element. Based on regression equations (1) to (3), it was found that the most rational technological modes are:

- the extract’s temperature $t=35...45\text{ }^\circ\text{C}$;
- the process durability $\tau=(1.5-2.0)\cdot 60^2\text{ s}$;
- the filtration pressure $P=0.4-0.5\text{ MPa}$;
- the vibration stirring rate $U=1.5-1.7\text{ m/s}$.

The results showed that the dependence of the performance of the PAN-100 semi-permeable membrane, the concentration of pectin substances (PS) in the concentrate, the content of dry matter (DM) in the permeate and concentrate under various technological modes are nonlinear. This is due to the complexity of the combined effect of the technological factors of the process of the UF PE concentration both on the productivity of the semi-permeable membrane and on the qualitative PC indicators. The approximation of the data by the regression equations has made it possible to reveal ambiguous dependences of the productivity of UF membranes, PS concentration, and DM content, on the pressure, temperature, and duration of the process of the UF PE concentration.

With the speed increase of pulsating vibration stirring flows above 1.5–1.7 m/s, the process stabilizes while the productivity of semi-permeable membranes of the PAN type increases by only by 1.3 times. It happens because, under the

action of the pressure of the pulsating flows, the compaction of both the sediment layer and the membrane material occurs. The effect of the perforated vibrating plate reaches a maximum performance value of $4.6 \text{ dm}^3/\text{m}^2\cdot\text{h}$ for PAN-100 and $4.2 \text{ dm}^3/\text{m}^2\cdot\text{h}$ for PAN-50, at which the density of the gel layer becomes constant.

Our study has limitations for the temperature and pressure parameters, as well as the use of a dead-end mode with a single type of polymeric UF membranes (PAN). The drawback of this study is the difficulty of applying the parameters of the ultrafiltration process for another type of raw materials, as well as incomplete removal of the formed polarizing gel layer due to its heavy compaction at the surface of the polymeric membrane.

The obtained results could be used when studying other operating parameters of the process of the UF concentration of pectin extracts, as well as for the development of a technical fleet of lines for making pectin products from various types of pectin-containing raw materials.

Our research is a continuation of the study into improving the processes of the membrane concentration of raw materials of animal and plant origin by using polymeric semi-permeable membranes and devising the methods for eliminating the gel layer at their surface. It is of scientific and practical interest and it must be continued and developed in the area of the application of turbulization methods for the process of the membrane processing of food high-molecular polydisperse systems.

7. Conclusions

1. To implement the technique for removing a gel layer from the membrane surface, a laboratory setup was proposed to study the process of the ultrafiltration concentration of pectin extract using the vibration stirring of raw materials. A mathematical model of the ultrafiltration process was built based on the method of planning a factorial experiment. The procedures for carrying out an experiment and for the statistical modeling of the obtained results have been presented.

2. The factors affecting the process of the membrane concentration of pectin extract using vibration stirring have been determined. It has been established that the combined effect of the parameters of the stirring rate, raw material's temperature and pressure, is ambiguous for the ultrafiltration process. This is due to the formation of not only a gel layer but also a dense polarizing layer at the membrane surface and the membrane shrinkage at high pressures and temperatures.

3. The results of our research and mathematical modeling have made it possible to determine the operating parameters for the process of the ultrafiltration concentration of pectin extracts using semi-permeable ultrafiltration membranes and applying a vibration stirring technique. These parameters are the pressure of $0.4\text{--}0.5 \text{ MPa}$, the temperature of skimmed milk of $35\text{--}45 \text{ }^\circ\text{C}$, the process duration of $(1.5\text{--}2.0)\cdot 60^2 \text{ s}$, the stirring speed of $1.5\text{--}1.7 \text{ m/s}$.

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