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The use of nanofiltration to regulate the properties of secondary products of processing animal raw materials

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Abstract. The problem of processing secondary material resources of the dairy industry, in particular buttermilk, is closely related to environmental issues. Nanofiltration is an energy-saving alternative to demineralization by ion exchange and electrodialysis. The purpose of the article is to study the membrane concentration process using nanofiltration flat membranes of the OPMN-P and OPMN-K brands for ultrafiltration of buttermilk. In order to achieve the set goal, two brands of nanofiltration membranes were tested during the experiment. The experimental study of the NF-UV process of oil pan permeate was carried out with the help of a laboratory installation, the parts of which included a container with a pressure gauge; thermostat; pump; manometers; counter (rotameter); membrane module; measuring capacity; valve; cylinder with CO_2. To identify the main filtration dependencies, two types of Vladipor OPMN nanofiltration membranes were studied. For each type, pressure in the range from 0.5 to 2.0 MPa and temperature from 20 to 40°C were used. The results show that both types of membranes effectively retain lactose at a pressure of 2.0 MPa, a temperature of 40°C, and a concentration factor of about 2. Changing the flow velocity over the membrane from 0.5 m/s to 1.5 m/s allows obtaining increase in specific productivity by 15% from the initial one. The effect of concentration polarization effects on the specific performance of nanofiltration membranes was established. The limitation of the concentration coefficient due to the increase in the osmotic pressure of the solution was determined.A conclusion was made regarding the possibility of large-scale implementation of nanofiltration of secondary material resources of milk processing in the study of membrane regeneration processes. The practical significance of the work lies in helping to solve environmental problems through the use of the nanofiltration method, which has a number of advantages over other processing mechanisms. In addition, such a process will make it possible to obtain new valuable liquids that can be used during the production of a number of dairy products, in particular lactose-free

Keywords: buttermilk, ultrafiltration, nanofiltration, lactose, membrane concentration

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INTRODUCTION

Effective processing of raw materials, in particular dairy, remains an important problem of the food industry to this day. In recent years, special attention has been paid to secondary material resources and waste. Among them, whey and buttermilk are of great importance [1-3]. This is related not only to economic indicators, but also to environmental problems. The chemical composition of these liquids defines them as an exceptionally favorable environment for the development of microorganisms. This fact causes an increase in indicators of wastewater pollution of dairy enterprises [4;5]. Ukrainian and international legislation in the field of environmental protection regularly increases regulatory requirements for the discharge of wastewater into natural reservoirs. This approach to the problem is an additional incentive for the relevant enterprises to find and implement modern technologies for the utilization of whey and buttermilk [6; 7].

Membrane technologies significantly help to solve this problem. The use of membrane processes in various combinations allows highly efficient processing of milk processing waste. This is explained by low energy consumption and the ability to preserve valuable biological properties of the main components of the chemical composition [7-9].

However, in this case, there is a problem of disposal of secondary streams formed as a result of membrane concentration or purification. This usually applies mostly to permeates. They are used worse than concentrates.

The dairy industry today remains the main one among the food industries in terms of the use of membrane equipment and technologies. First of all, this applies to ultrafiltration. Taking into account the large volumes of milk processing in this way, the importance of this problem also for ecology becomes clear. Additionally, along with ultrafiltration, reverse osmosis, microfiltration, and nanofiltration have recently been actively used.

Among the processes of utilization of whey and buttermilk, the removal of lactose and mineral substances is developed most intensively. This is explained by the significant need for these types of raw materials as a basis for lactose-free products. Many types of ice cream, frozen desserts, dry drinks, bakery products, food additives require whey and buttermilk with a reduced concentration of salts. Traditionally, demineralization is carried out by concentration and electrodialysis or ion exchange. In any case, the energy and equipment costs are quite significant. Recently, membrane technologies have become a profitable alternative to these processes [10-12]. Membranes are successfully used not only in the dairy industry, but also in other industries for the concentration and purification of liquids. It is believed that, in the future, membrane technologies will maintain the trend of rapid growth in utilization due to low energy consumption, relative simplicity of hardware design, convenience of automatic control and management, etc. [13-15]. The minimum impact on the natural value of the composition of raw materials is also important.

Nanofiltration (NF) is a unique membrane process. It allows you to separate low-molecular organic and inorganic compounds. The basis of NF is many, including electrical and Donnan effects. The nature of the NF process depends significantly on the properties of the membranes and the filtering object [16; 17].

The presented work is a logical continuation of early research [8]. The main attention is paid to the application of NF membranes for the treatment of UV oil permeate. This product is obtained by ultrafiltration of raw materials during concentration. Ultrafiltration permeate is used extremely inefficiently today. The volumes of accumulation of this valuable raw material are quite significant. The problem of disposal of the specified liquid is significant due to the significant risk of discharge of wastewater contaminated with permeate components. In this case, the burden on the environment is a valid argument when planning the implementation of dairy by-product processing technologies. Nanofiltration has significant advantages over other treatment methods. In addition to solving environmental problems, this process can allow obtaining new valuable liquids that will be widely used in the production of a number of special dairy products, for example, lactose-free.

LITERARY REVIEW

A number of publications [18-20] previously established a clear dependence of NF on the processing method, parameters, chemical composition of raw materials and properties of NF membranes. For example, the authors [18] investigated a significant effect in the combination of NF and diafiltration using NF membranes of the roll type DS-5DL (GE-Osmonics). In this case, the UV permeate of the serum was the object of research. Special attention was paid to the main dependencies during demineralization of lactose solution.

The paper [19] indicated the hydrophilicity of NF membranes as an important factor in the process. The authors of the work [20] established that with NF a gel layer is formed on the membrane surface and the concentration of dissolved substances, especially lactose, increases. It is indicated in the work that about 1% of lactose passes into the NF permeate.

The removal of mineral substances during the processing of various dairy products was studied by scientists [21]. They achieved 27...36% removal of the mineral component depending on the degree of concentration of UV permeates of milk and serum.

In the publication [22] NF was used to obtain solutions rich in lactose. For membranes of type NFT 50 and HR95PP, better results were obtained at a pressure value of 3 MPa, when 80% of water was removed from the serum.

Mineral substances and lactic acid served as an object of attention in NF in the work [23]. The researchers achieved a significant degree of acid retention. In addition to NF, diafiltration (DF) was used in this case. This technique made it possible to remove 30...71% of mineral salts from the original solution.

The production of many dairy products requires a low lactose content. Buttermilk-based products are no exception. Special attention to the nutrition of people with lactose intolerance is the main factor of intensive search in this direction due to the steady progress of this disease [24-26]. When removing lactose from buttermilk, a good effect is achieved by UV concentration and DF. Using water as a solvent for DF is a fairly simple solution. However, in this case, there are losses of balanced low molecular weight milk. A successful replacement of water in DF is NF permeate, which is obtained by appropriate UV treatment of the permeate of the oil pan [8].

Large-scale implementation of this phenomenon in the practice of processing secondary milk streams requires data confirming this possibility. It is necessary to know the performance of the studied membranes, which determines the economic indicators of the nanofiltration treatment process. The dependence of productivity on various factors of membrane treatment is of great importance. These indicators are of decisive importance when deciding the feasibility of implementing membrane technologies in the utilization of secondary material resources of the dairy industry.

That is why the study of the main dependences of NF-UV of oilseed oil permeate is the focus of this work. In addition, there is very little published data on this topic in the available sources.

MATERIALS AND METHODS

The permeate of the ultrafiltration of the buttermilk served as the object of research. Its composition is described in detail in [8] and presented in Table 1.

Table 1. *Chemical composition of products of membrane processing of oil pan*

Index	Fresh buttermilk	UF retentant FK=3	UF permeat FK=3
Mass fraction of protein, %	3.2	9.61	0.11
Mass fraction of fat, %	0.4	1.21	–
Mass fraction of lactose, %	4.5	4.54	4.48

Table 1, Continued

Index	Fresh buttermilk	UF retentant FK=3	UF permeat FK=3
The mass fraction of mineral substances, %	0.7	0.94	0.7

Source: *author's development*

Determination of constituent components in this work was carried out: mass fraction of lactose according to [27], content of mineral substances, mass fractions of fat and protein according to [28]. The obtained quality indicators clearly confirm the high value of the oil pan.

Nanofiltration membranes. In the experiments, two brands of nanofiltration membranes were tested, which are manufactured in series by the company "Vladipor" (Volodymyr, Russia). The characteristics of the membranes are given in table. 2. Membranes have high resistance to oxidants. The working pH range is 2...12. Membranes withstand a concentration of active chlorine of at least 1 mg/l. This allows for highly effective regeneration and disinfection. Organic compounds with a molecular weight of more than 100 D are retained highly efficiently.

Table 2. *Properties of "Vladipor" nanofiltration membranes*

Type	Researched conditions	Selectivity, %	Productivity, l/m2·h
OPMN-P	0.15% solution NaCL, 25°C, p=15 atm	30	140
OPMN-P	0.15% solution NaCL, 25°C, p=15 atm.	60...70	1400

Source: *author's development*

NANOFILTRATION TREATMENT

Experimental studies of the NF-UV process of oil pan permeate were carried out in laboratory conditions at the installation, the photo of which is shown in Fig. 1. The schematic diagram of the laboratory installation is shown in Fig. 2.

Figure 1. *Laboratory installation FT-01*

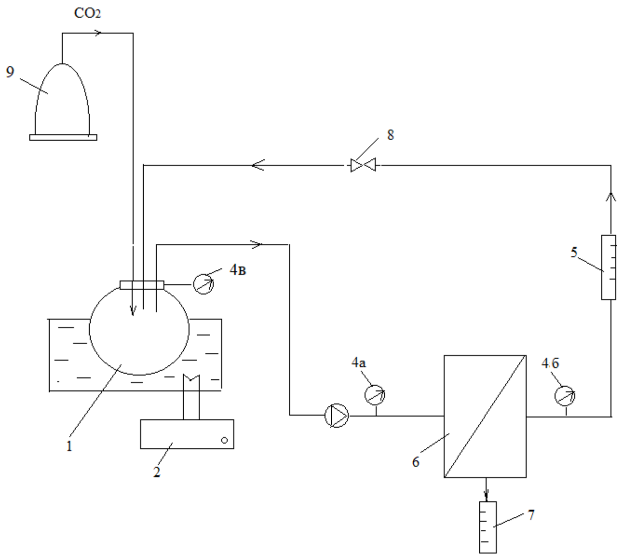

Figure 2. *Schematic diagram of the laboratory installation*

Notes: *1 – container with pressure gauge; 2 – thermostat; 3 – pump; 4 a, b, c – manometers; 5 – counter (rotameter); 6 – membrane module; 7 – measuring capacity; 8 – valve; 9 – cylinder with* CO_2

Source: *author's development*

The membrane module of the installation contains 6 elements of the same type, one of which is shown in the photo (Fig. 3). The design feature of the presented elements is the spiral channels, which allow the liquid flow to be transferred to the turbulent mode at relatively low flow velocities. This contributes to the mixing of the liquid layer in the space above the membrane surface. This design of the module is extremely important in the sense of preventing the formation of deposits on the membrane surface and eliminating the negative effect of concentration polarization. Membrane elements are made of high-quality structural plastic mass. They have high resistance to pressure and high temperatures and are resistant to corrosion under the influence of aggressive reagents. The incoming liquid flow is directed tangentially, which additionally contributes to the turbulent regime of the flow over the membrane surface.

Figure 3. *Membrane module of the installation*

The plant works in flow mode through membrane elements and a membrane module. This regime has a beneficial effect on the mass transfer of dissolved compounds across the membrane surface. In this way, the accumulation of compounds trapped by the membrane in a thin layer of liquid above the surface of the membrane is eliminated. That is why there are additional positive conditions for increasing the specific productivity of membranes. When the unit is operating, the permeate from container 1 of the closed UV type is fed into the membrane module 6 using a high-pressure gear pump 3. Additional pressure in the unit is created by supplying compressed CO_2 from the cylinder. This method of liquid processing allows to obtain a greater value of the driving force of the process, which is inhibited by the technical limitations of the pump of the installation and the power of its engine.

The NF permeate is taken into measuring tank 7 to measure the performance of the process. The working pressure is monitored at the inlet and outlet of the membrane module using manometers 4 a, b and is determined by the formula:

$$P = \frac{P_1 + P_2}{2}, \mathrm{MPa} \quad (1)$$

where P_1, P_2 are the fluid pressure at the inlet and outlet of the module, respectively, MPa. Concentrate flow rates are controlled by a meter.

A periodic mode of concentration was implemented in the installation. In this way, after UV concentration, the permeate was sent to the output tank 1 through the valve 8. The flow rate in the system was regulated by setting the valve in a certain position and closing the "by-pass" channel provided in the pump. The flow rate was calculated using the expression:

$$V = \frac{Q}{S}, \text{м/с} \quad (2)$$

where Q is the volume flow rate of the concentrate, m^3/s; S is the cross-sectional area of the intermembrane channel, m^2. The necessary filtration temperature was set with the help of a thermostat, which controlled the degree of heating of the electric heat exchanger.

After a certain time, the concentration factor was determined by the expression:

$$FK = \frac{V_B}{V_K}, L \quad (3)$$

where V_v, V_k are the volumes of the initial liquid and concentrate, liters.

The volume of the concentrate was determined by the equation:

$$V_{\mathrm{K}} = V_{\mathrm{B}} - V_p, L \quad (4)$$

where V_p is the permeate volume, l.

Selectivity was determined in NF permeate samples for each membrane:

$$\varphi = \left(1 - \frac{C_n}{C_k}\right) \bullet 100\% \quad (5)$$

where C_n, C_k are the concentrations of components in permeate and concentrate, respectively, g/l.

RESULTS AND DISCUSSION

Specific performance of membranes. The NF process was carried out at different operating parameters of pressure, temperature and flow rate. The obtained results are shown in Fig. 4 a, b, c and Fig. 5.

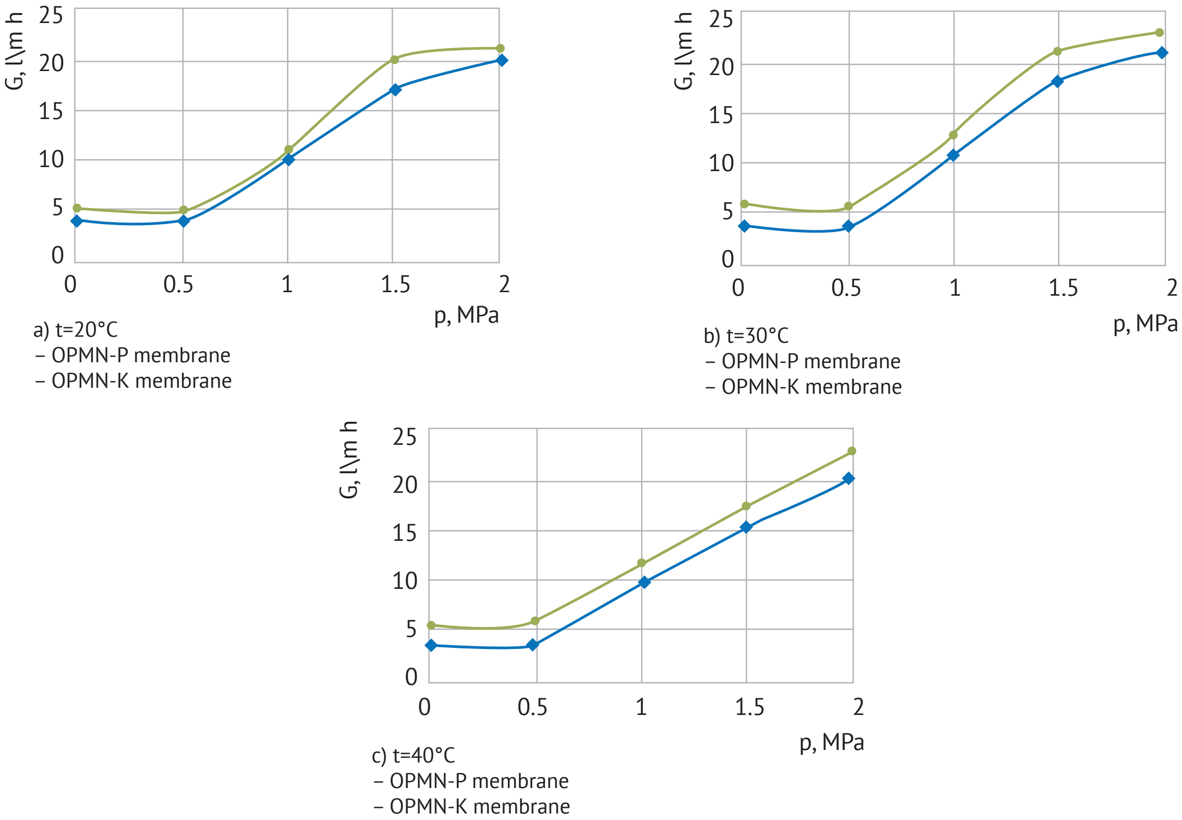

Figure 4. *Effect of temperature on membrane performance*

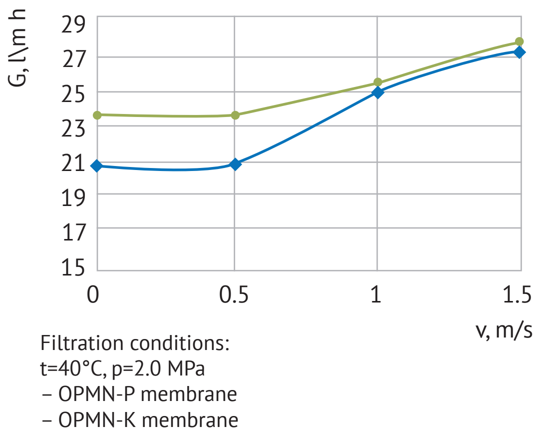

Figure 5. *Effect of flow rate on the specific performance of membranes*

In all cases, the same trend was investigated: with increasing pressure and temperature, the specific performance of the membranes increased. However, when the value of 1.5 MPa was exceeded, this growth became less pronounced. This phenomenon can be explained by the formation of a dense layer on the separating surface of the membranes. It is called mud polarization of the membrane. This phenomenon occurs due to exceeding a certain limit of solubility of the compound. Intensive transfer of the solvent (water) through the distribution layer of the membrane leads to an increase in the concentration of dissolved compounds and the formation of a precipitate in the form of a thin, uniform layer on the membrane. It mostly contains proteins that were not separated from the buttermilk in the UV process and got into the UV permeate. The phenomenon of concentration polarization also has a certain influence on the performance of membranes by nanopermeate. It occurs near the membrane surface due to the accumulation of molecules and ions. Molecules and ions of the same charge begin to repel each other, which prevents the transfer of solvent and dissolved compounds that pass through the membrane. In this zone, intensive transfer of solvent molecules through the membrane takes place. It is because of this that the concentration of proteins increases, their aggregation, and a thin layer of gel is formed. Together, these phenomena add up to additional resistance and impair mass transfer through the membrane. In each of the experiments, the indicators of the specific productivity of the membranes of the OPMN-P brand were significant in comparison with the OPMN-K brand.

The increase in the performance of the membranes when the oil pan is heated can be explained by the decrease in the viscosity of the solution. In this case, there are better conditions for liquid circulation, increasing flow speed and its turbulence. Accordingly, under such conditions, liquid layers are better mixed. This weakens the phenomenon of concentration polarization and the formation of a gel layer. In this case, the increase in the flow of the solvent through the membrane is investigated.

It is interesting that increasing the temperature of the oil pan from 30 to 40°C did not cause a significant increase in the performance of the membranes for any pressure. Temperatures higher than 40°C were not used in the experiments due to the threat of denaturation of the remaining proteins after UV treatment. Denatured proteins are known to form a dense layer on the membrane more intensively. This phenomenon can be explained by the irreversible loss of higher forms of spatial organization of the molecule by denatured proteins. As a result, the loss of protein solubility and the formation of sediment on the surface of the membrane. Therefore, for further experiments, a temperature of 40°C was chosen as the optimal temperature in terms of productivity and energy savings. In addition, there were manufacturer's temperature limitations for the membranes. For these conditions, the effects of the flow rate on the specific productivity were investigated. The results were obtained (Fig. 5), which prove that in the range of speeds of 0.5…1.5 m/s the increase in productivity was noticeable. The highest result of 15% was achieved at a flow rate over the membrane surface of 1.5 m/s. This tendency was later much less pronounced. It is possible that at higher speeds the increase in productivity would be more significant. The technical capabilities of the experiments did not allow to fully investigate this effect. Perhaps this would cause additional energy costs, which would have a negative impact on the overall economic indicators of the NF process.

The change in the specific performance of the membranes with the treatment time is an important indicator that is needed to estimate the total membrane filtration area of a certain volume of liquid. Without this indicator, it is impossible to determine the economic feasibility of introducing the nanofiltration process into the practice of processing secondary raw materials. The change in specific productivity is crucial when choosing a membrane plant and regeneration methods. The necessary results are presented in Fig. 6.

The dynamics of the process is reflected by the increase in the oil concentration factor. This indicator was calculated according to the decrease in the volume of the concentrate. It shows how much the volume of the original liquid has decreased compared to the initial value. In the range of FC changes from 0.5 to 2.0, the decrease in the specific productivity of both brands of membranes was quite significant (by 60% of the initial). It is interesting that at FC values greater than 1.0, the studied membranes showed almost the same performance. This can be explained by the significant increase in the osmotic pressure of the NF concentrate and the thickening of the membrane structure due to the prolonged exposure to high operating pressure. Osmotic pressure, as is known, is a function of the concentration of certain soluble compounds. In this case, this indicator is affected by dissolved compounds retained by the nanofiltration membrane (lactose and part of mineral salts). The compact structure of the membrane mechanically prevents the transfer of molecules through the membrane due to the reduction in the diameter of the holes. In this state, probably, the main role in the separation of the components of the oil pan is played by the secondary membrane, which was formed due to the phenomenon of gel polarization. It has slightly different properties from the main membrane, which adds some complexity to the process of transferring molecules and causes unpredictable results.

Such a phenomenon is known for flat membranes that are constructively placed on a support in "plate and frame" type installations. When using a different type of membrane, for example, a tubular or roll membrane, the indicated effect might be less important. Additional research in this direction has certain interest and prospects.

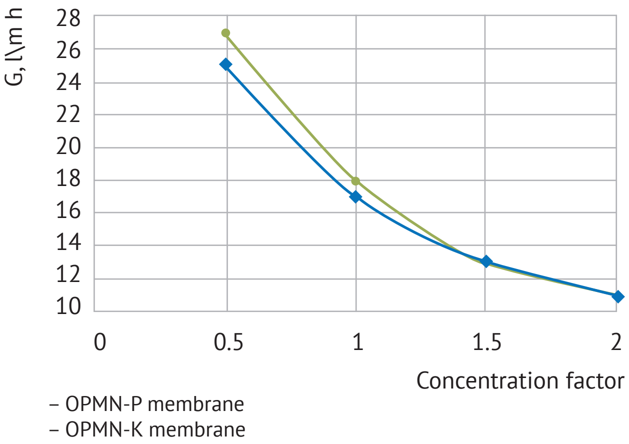

Figure 6. *Influence of the concentration factor on the specific performance of membranes*

MEMBRANE SELECTIVITY

There were certain changes in the selectivity of membranes in the process of NF treatment of buttermilk established in a series of subsequent experiments. Their results are shown in Fig. 7

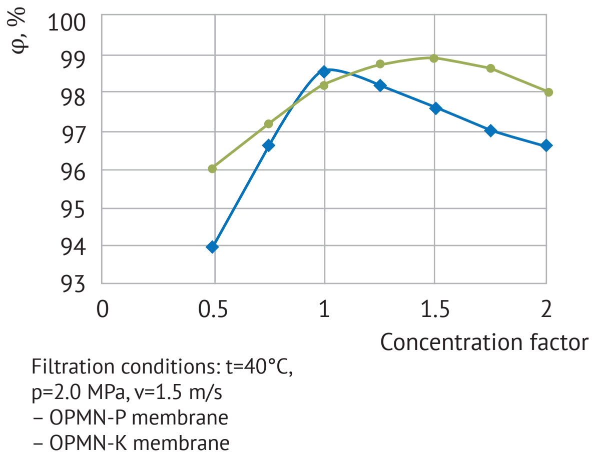

Figure 7. *Influence of the concentration factor on the selectivity of the membrane*

Similar effects of lactose retention were studied for both brands of membranes. At first, the selectivity increased, and then decreased. This phenomenon was more pronounced for the OPMN-K membrane. in our opinion, the obtained results can be explained from the standpoint of the theory of membrane filtration. With the growth of FC, the structure of the membranes is compacted over time, which causes a corresponding decrease in the diameter and volume of the pores. This gives, to a certain extent, an increase in the effect of lactose retention. In the future, some increase in the presence of lactose in the NF permeate due to the lack of an ideal structure of the membrane in relation to the diameter of the holes is investigated. This is explained by certain defects in the formation of membrane surfaces in industrial conditions. For example, it is impossible to achieve the same shape and diameter of the membrane pores over the entire surface of the polymer used for the manufacture of membranes. Deformation of the membrane under pressure also makes adjustments to the pore volume of the semipermeable membrane.

CONCLUSIONS

The NF process is an effective means of processing the UV permeate of buttermilk to obtain a NF permeate with a minimum lactose content.

In order to establish the main dependencies of NF processing of this valuable secondary product of milk processing, flat membranes of OPMN-P and OPMN-K brands were investigated in the concentration mode.

It was established that the specific productivity of membranes is significantly affected by temperature, pressure, flow rate and liquid concentration factor. The best results for the investigated process were determined in the conditions of laboratory experiments: pressure 2.0 MPa, temperature 40°C, flow speed about 1.5 m/s. the studied membranes revealed minor discrepancies in the main indicators characterizing the performance of the process. Taking into account the best indicators of the lactose retention effect, it is possible to recommend the OPMN-P membrane (φ=99%) for the practical purpose of removing lactose from the UV permeate of the buttermilk. The productivity of this membrane is 11…28 $l/m^2 \cdot h$. depending on the concentration factor.

Further research in this direction will be conducted using industrial roll elements for NF and comparative characteristics of different manufacturers of membranes and membrane elements.

The work also did not investigate the important problem of membrane regeneration. The choice of the method of regeneration and the establishment of the main parameters of this process is a separate important link of research in membrane technologies. Large-scale implementation of NF, of course, requires an assessment of this important factor, which significantly affects the economic efficiency of the process. For example, it affects the operating time of the membranes, the duration of the technological work cycle, etc.

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Застосування нанофільтрації для регулювання властивостей вторинних продуктів переробки тваринної сировини

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Одеська національна академія харчових технологій

Анотація. Проблема переробки вторинних матеріальних ресурсів молочної промисловості, зокрема маслянки, тісно пов’язана з екологічними питаннями. Нанофільтрація є енергоощадною альтернативою демінералізації методом іонного обміну та електродіаліза. Два типи нанофільтраційних мембран ОПМН фірми «Владіпор» досліджено з метою виявлення основних залежностей фільтрування. Питома продуктивність та селективність тестованих мембран є головними предметами досліджень. Є підстави для висновку щодо широкомасштабного впровадження нанофільтрації при утилізації маслянки

Ключові слова: пахта, ультрафільтрація, нанофільтрація, лактоза, мембранне концентрування