

# ***Modern Technologies of Milk Whey Processing in the Conditions of New Techno-Economic Paradigm***

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**Abstract** - The transition to the next techno-economic paradigm under the conditions of new industrialization has demanded a change in the attitude of consumers to secondary raw materials and, in particular, approaches to the processing of milk whey. One of the breakthrough technologies of waste-free processing of whey is membrane technology. The study is currently significant insofar as it considers reprocessing of secondary raw materials represented by milk whey generated in bulk in the course of curd and cheese production. Waste-free method of whey reprocessing has been experimentally justified employing two stages of baromembrane technology: ultrafiltration at the first stage to recover macromolecular components followed by reverse osmosis concentration of lactose applying modern domestically produced membranes. The materials and methods for investigating the processes of ultrafiltration and reverse osmosis concentration of whey have been described. Proprietary solution has been suggested for milk whey ultrafiltration without its preliminary treatment using ceramic ultrafiltration elements (0.01). Optimum operating parameters for the processes of ultrafiltration and reverse osmosis concentration of whey have been experimentally established and verified in the workshop environment. A technological process for Waste-free whey processing has been developed. The results of the experimental investigations have been discussed and the relevant conclusions have been made. As a result ceramic membrane can be recommended as the most preferable technical solution for separating whey without any preliminary treatment during ultrafiltration process. Recommendations are given for reverse osmosis process conditions.

**Keywords** – new industrialization, milk processing, membrane technology, whey, ultrafiltration, reverse osmosis, ceramic membranes.

## I. INTRODUCTION

In the conditions of a transition to the next techno-economic paradigm in food industry today maximum utilization of raw materials, including secondary resources is a priority trend of development. Secondary resources are mostly represented by multi-component aqueous mineral-organic solutions that contain large amounts of useful commercially processable elements. A characteristic example here is whey that is generated in the course of manufacturing curd and cheese and that consists of a wide spectrum of proteins, lactose, vitamins, pectins and other components [1,2, 4-6, 8-10].

Low contents of those elements in original whey necessitate its concentration before its further processing in the course of the production process. Different aspects of potential waste-free technologies for recycling secondary resources in dairy industry have been studied by such leading Russian scientists as N.N. Lipatov, A.G. Khramtsov, L.A. Ostroumov, V.D. Kharitonov, V.A. Lyalin, A.F. Zyabrev, A.A. Svitsov, etc.

## II. LITERATURE REVIEW

Existing practices in Russia and all over the world prove that membrane technology has been recognized as a highly advisable method of whey processing that makes it possible to separate, to refine, to fraction and to concentrate similar media preserving the components in their native conditions under

gentle temperatures, without water phase transition and with minimum energy loss, as compared to other technological processes [3-6]. These baromembrane processes include microfiltration, ultrafiltration (UF), nanofiltration and reverse osmosis (RO). Despite obvious advantages, those processes have not been widely spread across food businesses, which can plausibly be explained by the insufficient experimental and theoretical foundations in the sphere of baromembrane processes employed for processing multiple food media, including whey [5-8]. Based on the analysis of literary sources, conclusions have been made that whey should be processed in two stages: applying ultrafiltration at the first stage to concentrate macromolecular components and applying reverse osmosis concentration of ultrafiltration permeate at the second stage to recover lactose [2,3,7-9,11].

The Objective of study was to establish the fundamental regularities of whey baromembrane processing applying domestically manufactured membranes, and, based on the obtained data, to determine the parameters of UF separation and RO concentration of whey.

### III. RESEARCH METHODOLOGY

The objects of the investigation are fresh curd whey and cheese unsalted whey with physical and chemical parameters shown in Table 1. The study made use of both conventional standardized and original methods of investigation.

TABLE 1. WHEY ANALYSIS (MEAN VALUES)

Parameter	Curd whey	Cheese whey
Total protein, %	0.9±0.15	0.7±0.15
Lactose, %	4.3±0.02	4.9±0.02
Fats, %	0.4±0.05	0.1±0.05
Mineral substance, %	0.7±0.05	0.6±0.05
Dry substance, %	6.2±0.22	6.4±0.22

Physical and chemical metrics have been determined based on the standardized methods: mass ratio of moisture, mass ratio of caseins, whey proteins, as well as the total content of proteins were identified applying refractometric analysis and the formol titration, whereas the Kjeldahl method was used as reference method; mass ratio of fat was identified applying the Gerber method; titratable acidity, mass ratio of mineral substance elements were found applying the method of atomic absorption spectrophotometry using U-2900 Hitachi instrument; mass ratio of lactose was determined by the Lorenz method.

UF process was investigated under laboratory conditions and in workshop environment. Laboratory investigations were carried out at a special plant shown in Fig 1. The plant consists of UF cell 1, pump 2, circulation tank 3 and permeate tank 4, pressure gage 5, flow meter 6, regulating valve 7, pipe coil 8, thermocouple 9, millivoltmeter 10, Dewar vessel 11, divider 12, valves 13 and 14. UF cells 1 are used for separating the whey under investigation. Pump 2, ONTz Type 1.5/20K – 0.75/2 with frequency converter of FRENIC-Eco F1S Type, is meant for feeding the whey under investigation into UF cells 1 and for creating pressure within the plant. Circulation tank 3 with volume 50 cubic decimeters is used for charging original whey and for circulating it within the “circulation tank – pump

– UF cell” loop. Permeate tank 4, represented by a measuring glass flask, serves for determining permeate consumption at the plant. Pressure gage 5, M0-5 Type, is used to control pressure at the plant. Flow meter 6, RS-5 Type, is applied to determine the whey consumption at the plant. Regulating valve 7, RU-160 Type, is meant to regulate pressure at the plant. Pipe coil 8 made of steel Grade 12Kh18N10T is used to regulate temperature of the whey under investigation. Thermocouple 9, chromel-alumel type, is applied to control the temperature mode of the UF process. Millivoltmeter 10, F-4214 Type, is used to control the electromotive force generated by thermocouple 9. Dewar vessel 11, represented by a waterproof tank made of plastic foam and containing ice within it is meant to take into account the effect produced by the temperature of the environment in the course of measuring the temperature of the process of separation. Divider 12 represented by a metallic membrane is used to prevent the whey from penetrating into the operational elements of the pressure gauge 5. Valves 13, 14 are used to connect UF cells 1 with the plant alternately.

UF cells 1 are the principal elements of the laboratory plant and they can operate in “tangential” mode. In the cylindrical cell, represented by a cylindrical 40 mm diameter and 890 mm long apparatus, there is an 800 mm long tube-type ceramic membrane element 4. The area of the membrane in the cell amounts to  $1.5 \cdot 10^{-2}$  sqm. In the flat-chamber cell, represented by a flat-chamber apparatus with 350 mm diameter lid, there is a 300 mm diameter plate polymer membrane. The area of the membrane in the cell makes  $7.0 \cdot 10^{-2}$  sqm. All metallic parts of the cells are made of stainless steel Grade 12Kh18N10T.

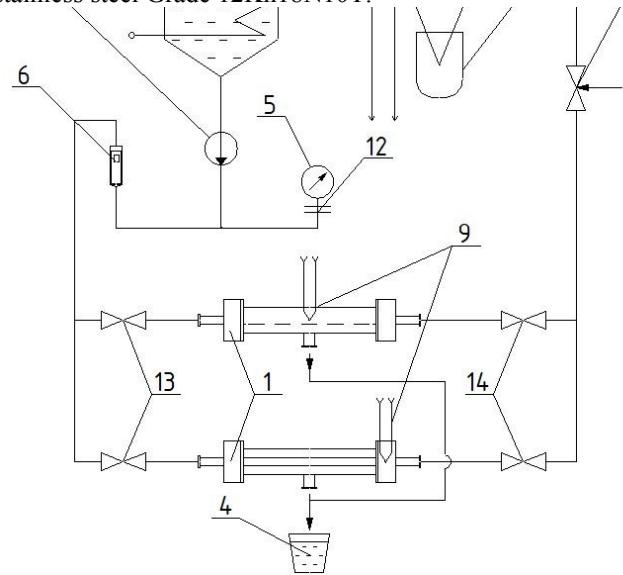


Fig. 1. Sketch of plant for UF process investigations

1 – UF cells (flat-chamber and cylindrical); 2 – pump; 3 – circulation tank; 4 – permeate tank; 5 – pressure gage; 6 – flow meter; 7 – regulating valve; 8 – pipe coil; 9 – thermocouple; 10 – millivoltmeter; 11 – Dewar vessel; 12 – divider; 13, 14 – valves.

The principle of the plant operation is founded on dividing the whey under investigation, in UF cell, into two flows: the flow that goes through the membrane (permeate) and the flow that remains above the membrane (concentrate). For this purpose, circulation tank 3 is charged with the whey to the amount of 25 to 40 cdm. When valve 7 is open, pump 2 is switched on, a specified flow rate is set, controlled by flow meter 6, and then, applying regulating valve 7 the required pressure is set in cell 1, that is controlled by pressure gauge 5. Temperature mode of the process is regulated by pipe coil 8 and is controlled by thermocouple 9 and millivoltmeter 10. Upon passing UF membrane, permeate is drained into measuring tank 4.

The experiment employs the following types of membranes: polysulfone amide plate membranes – UPM-20; 50M, cellulose acetate membranes – UAM-50P; 100P manufactured by Vladipor CJSC Scientific Technical Center, Vladimir, Russia, and ceramic membrane of CUFE series based on anatase titanium dioxide, plated with separating layer of  $\alpha$  aluminum oxide, modifications (0.01) and (0.02) manufactured by Scientific and Production Company "Ceramicfilter", Ltd, Moscow, Russia. These membranes feature molecular mass "cutoffs": 10; 30; 50; 100; 150 kilo Dalton.

In working conditions, the experiment was carried out at the pilot plant. In UF unit, there are 14 elements of CUFE-19 Type (ceramic ultrafiltration elements with pore sizes of 0.01 and 0.02 micrometer) manufactured by Ceramicfilter SPC, Ltd with total membrane area of 3.34 sqm. The work was done at the farm managed by A.V. Anikyev (the town of Polevskoy, the Sverdlovsk Region, Russia).

Within the framework of the study, two values were measured: permeability  $G$  and separation capacity  $\phi$  of the membranes in the process of separation under different parameters.

Permeability of the membranes was established according to the equation as follows:

$$G = V_p / (F_0 \cdot \tau), \quad (1)$$

where  $V_p$  – permeate volume, cbm;  $F_0$  – membrane surface area, sqm;  $\tau$  – time of the process, seconds [12-14].

Separation capacity of the membranes was evaluated in line with the following equation:

$$\phi = 1 - C_2/C_0, \quad (2)$$

where concentration  $C$  of macromolecular substances (MMS) was determined based on total protein content in whey ( $C_0$ ) or in permeate ( $C_2$ ) [12-14].

Reverse osmosis (RO) process was investigated in laboratory conditions and in workshop environment. The investigations have been carried out at a special plant (Fig. 2). The plant includes RO unit 1, pump 2, circulation tank 3 and permeate tank 4, pressure gauge 5, flow meter 6, regulating valve 7, pipe coil 8, thermocouple 9, millivoltmeter 10, Dewar vessel 11, divider 12.

RO unit 1 is used for separating the whey under investigation. Pump 2, Spek P45/120-80 Type with frequency converter FRENIC-Eco F1S Type, is meant for feeding the whey into RO unit 1 and for creating pressure at the plant. Circulation tank 3, volume 50 cbm, is used for charging original whey and for circulating it within the "circulation tank – pump – RO unit" loop. Permeate tank 4 represented by a

measuring glass flask serves for determining permeate consumption at the plant. Pressure gauge 5, M0 1227 Type, is used to control pressure at the plant. Vortex flow meter 6, EV 200Type, is applied to determine the whey consumption at the plant. Needle valve 7, VYC 147 Type, is meant to regulate pressure at the plant. Pipe coil 8, made of steel Grade 12Kh18N10T, is used to regulate temperature of the whey under investigation. Thermocouple 9 of chromel-alumel type is applied to control the temperature mode of RO process. Millivoltmeter 10, F-4214 Type, is used to control the electromotive force generated by thermocouple 9. Dewar vessel 11, represented by a waterproof tank made of plastic foam and containing ice within it, is meant to take into account the effect produced by the temperature of the environment in the course of measuring the temperature of the process of separation.

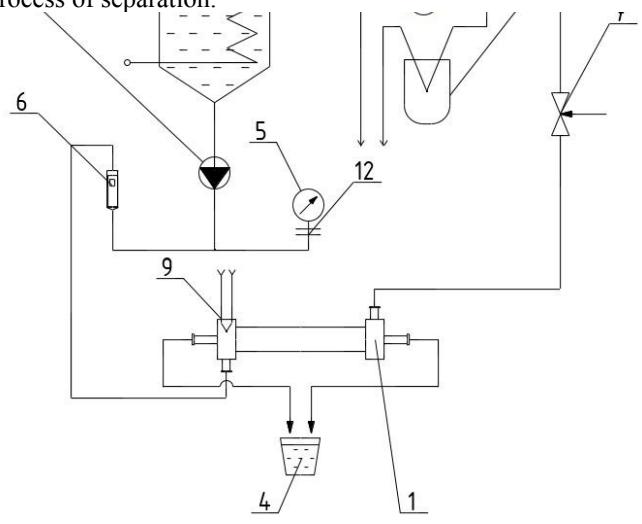


Fig. 2. Schematics of plant for investigating RO process

1 – membrane unit; 2 - pump; 3 – circulation tank; 4 – permeate tank; 5 – pressure gauge; 6 – flow meter; 7 – needle valve; 8 – pipe coil; 9 – thermocouple; 10 – millivoltmeter; 11 – Dewar vessel; 12 – divider.

Divider 12, represented by a metallic membrane, is used to prevent the whey from penetrating into the operational elements of the pressure gauge 5. The principal part of the plant is RO unit 1 equipped with ERO-B-45-300 roller element manufactured by Vladipor CJSC STC, Vladimir, Russia. The experiments employed two types of RO membranes: MGA-80P Type and MGA-100P Type.

The principle of the plant operation is founded on dividing the whey under investigation, in RO unit, into two flows: the flow that goes through the membrane (permeate) and the flow that remains above the membrane (concentrate). For this purpose, circulation tank 3 is charged with the whey to the amount of 25 to 40 cdm. When valve 7 is open, pump 2 is switched on, a specified flow rate is set, controlled by flow meter 6, and then, applying regulating valve 7, the required pressure is set in unit 1 that is controlled by pressure gauge 5. Temperature mode of the process is regulated by pipe coil 8 and is controlled by thermocouple 9 and millivoltmeter 10.

Upon passing RO membrane, permeate is drained into measuring tank 4.

Within the framework of the study, two values were measured: permeability  $G$  and separation capacity  $\phi$  of the membranes in the process of separation under different parameters. Membrane permeability was found according to equation (1), separation capacity was calculated in line with equation (2) where concentration  $C$  of the dissolved dry substances (DS) was determined based on lactose content in whey ( $C_0$ ) or in permeate ( $C_2$ ).

In workshop environment, the experiment was carried out at the pilot plant. In RO unit, there is one element of ERO-80-475 Type (with membrane of MGA-80P Type) manufactured by Vladipor STC. The work was done at the farm managed by A.V. Anikyev (the town of Polevskoy, the Sverdlovsk Region, Russia).

The results of the experiments have been processed applying the methods of mathematical statistics together with correlation and regression analysis at confidence coefficient of 95 (the significance level of 0.05). Functional dependency of the obtained experimental data upon the parameters under investigation has been determined by the least square adjustment method.

#### IV. RESULTS AND DISCUSSION

##### 3.1. Ultrafiltration Separation

Investigations of the effects produced by the basic parameters on UF membrane characteristics (permeability  $G$  and separation capacity  $\phi$ ) for protein phase are illustrated by Figures 3,4. The parameters of the concentrated whey are given in Table 2.

TABLE 2. PHYSICAL AND CHEMICAL ANALYSIS OF WHEY AFTER ULTRAFILTRATION (MEAN VALUES)

Parameter	Curd whey		Cheese whey	
	concentrate	permeate	concentrate	permeate
Total protein, %	8.45±0.15	trace amount	6.82±0.15	trace amount
Lactose, %	4.27±0.02	4.25±0.02	4.92±0.02	4.95±0.02
Fats, %	3.30±0.05	not detected	1.04±0.05	not detected
Mineral substance, %	0.70±0.05	0.65±0.05	0.67±0.05	0.61±0.05
Dry substance, %	16.72±0.22	4.91	13.45±0.22	5.57

Dependency  $G(u)$  has shown on Fig.3 that permeability of UF membranes becomes constant when the flow rate of the solution above the membrane amounts to  $u = 1.1\text{-}1.5 \text{ m/s}$ , which correlates with the Reynolds number of the flow in the tube-type membrane of CUFE (0.01) Type,  $Re = 4750\text{-}6500$ , and that of the flow in the flat channel  $Re = 4450\text{-}7400$ . Analysis of the dependencies  $G(P)$  and  $\phi(P)$  has shown that the characteristics of membranes CUFE (0.01) Type and UPM-50M Type are highly recommendable. Against this background, the following experiments were carried out with those membranes only. Analysis of the dependencies  $G(t)$  and  $\phi(t)$  has shown that permeability of UF membranes increases as the temperature gets higher, but it is limited by a certain interval  $t$ , depending on the type of UF membrane and on the concentration of the solution (Figure 6). As  $t$  increases, the separation capacity of UF membranes gets lower, which can be explained by partial entrainment of protein molecules in the

pores of the membrane occurring at higher speed of filtration because of hydrolysis [15].

Analysis of the dependencies  $G(C)$  and  $\phi(C)$  has shown that permeability of UF membranes tends to decrease as  $C$  gets higher, which is consistent with UF established regularities. It has been found that for cases with whey, the ratio of concentrate to permeate amounts to 1/10. The separation capacity of UF membranes remains practically the same as  $C$  increases, which is explained by lower permeability and constant hydrodynamic conditions above the membrane.

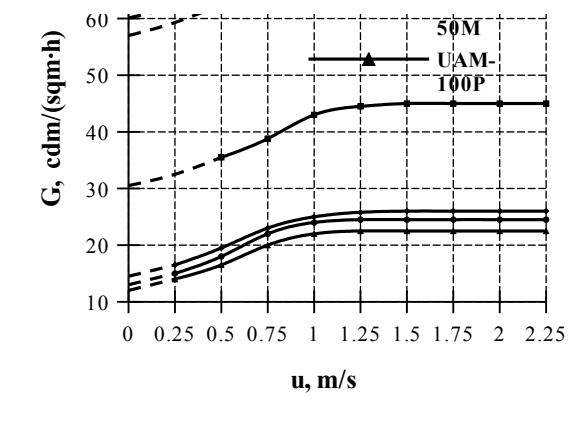


Fig. 3. Dependency of UF membrane permeability on the whey flow rate above the membrane at  $P = 0.3 \text{ MPa}$ ;  $t = 20^\circ\text{C}$ ;  $C = 0.8\%$  MMS

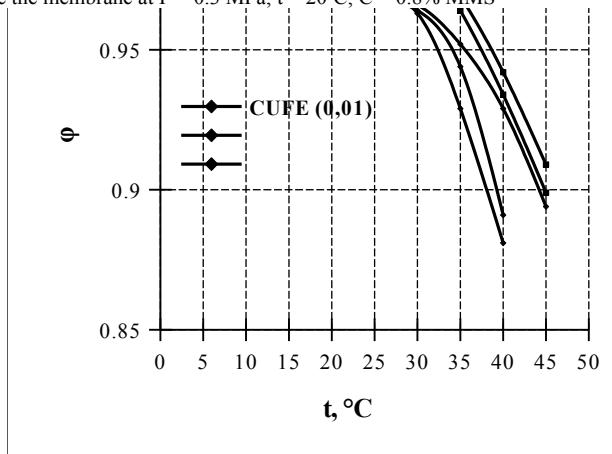


Fig. 4. Dependency of UF membrane separation capacity on temperature, at  $u = 1.5 \text{ m/s}$ ;  $P = 0.3 \text{ MPa}$ .

1 –  $C = 1.9\%$  MMS; 2 –  $C = 1.3\%$  MMS; 3 –  $C = 2.5\%$  MMS; 4 –  $C = 0.8\%$  MMS (macromolecular substance)

Thus, the results of investigating UF separation of whey enable the conclusions as follows:

- the flow rate of whey above the surface of the membrane should not be less than  $u = 1.5 \text{ m/s}$ ;

- operating pressure of UF process should be maintained within the range of  $P = 0.3$  MPa;

- UF process does not require temperatures higher than that of the environment, i.e. it is advisable that the process should be run at  $t = 20 \pm 5^\circ\text{C}$ ;

- it is feasible to apply UF process for concentration of macromolecular substance of  $C = 8\%$  MMS (ratio of MMS concentrate to permeate makes 1/10);

- ceramic membrane of CUFE (0.01) Type can be recommended as the most preferable technical solution for separating whey, as compared to other UF membranes.

### 3.2. Reverse Osmosis Concentration

The investigations were carried out using permeate obtained in the course of UF whey processing. For the purposes of RO concentration, the membranes of MGA-80P and -100P Type were applied.

The results of investigating the effects produced by the basic parameters (operating pressure, temperature, concentration and hydrodynamic conditions above the membrane) on the characteristics of RO membranes (separation capacity  $\varphi$  and permeability  $G$ ) are described in Figures 5,6.

The investigations showed that dependency  $G(P)$  for cases with whey differs from the straight-line correlation considerably (Fig. 5). These deviations are especially obvious at the initial and at the final sections of the dependency line. Nonlinearity of  $G(P)$  on the initial section at low values of  $P$  can be explained, based on the reverse osmosis theory, by the effects produced by the capillary-osmotic counter-flow that normally decrease as  $G$  grows. Moreover, the investigations proved that the effects of the capillary-osmotic flow become more explicit as the concentration of whey increases (Fig. 5, 3,4).

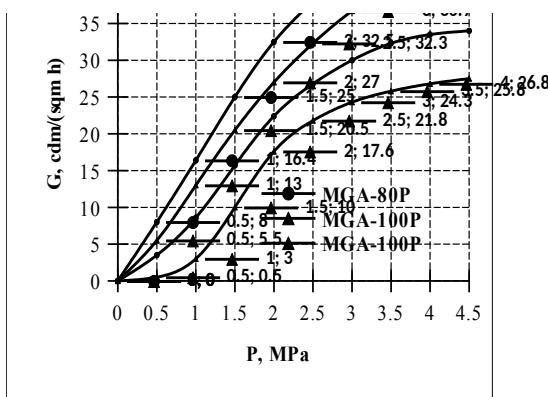
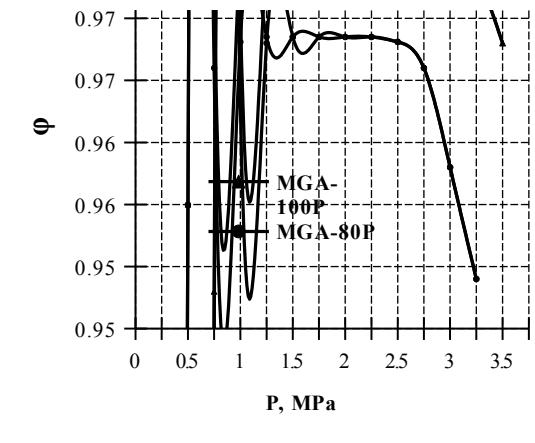


Fig. 5. Dependency of RO membrane permeability on operating pressure, at  $t = 20^\circ\text{C}$ ,  
1, 2 – 5% DS; 3, 4 – 10% DS (dry substance)



1,2 – 5% DS; 3,4 – 10% DS

Fig. 6. Dependency of RO membrane separation capacity on operating pressure, at  $t = 20^\circ\text{C}$

The mode when dependency  $G(P)$  is linear is established within a certain range of operating pressure (0.5 - 1.75 MPa for  $C = 5\%$  DS and 1.0 – 1.85 MPa for  $C = 10\%$  DS). As the pressure exceeds some certain value, a drastic drop in  $G(P)$  is observed (Fig. 5, 1-4), which represents a consequence of the effects produced by concentration polarization, caused by higher values of permeate flow and by their dominance over the diffusion intensity in boundary layer. When pressure differential is created at the membrane, one observes a drastic increase in the membrane separation capacity (Fig. 6). Thereat, the membrane separation capacity obviously depends on the effects produced on the process by capillary-osmotic flow. Drastic increase in separation capacity, depending on whey concentration, is observed at the section where the pressure changes in the range of 0.25 – 0.75 MPa, which corresponds, according to dependency  $G(P)$ , to the pressure range within which the effects of capillary-osmotic counter-flow are most explicit. Consequently, it is possible to conclude that separation capacity of the membrane increases as RO process becomes less affected by such factors as, first, diffusion in the pores of the membrane, and, second, the capillary-osmotic counter-flow. Increased membrane permeability  $G$  that suppresses the abovementioned factors can improve separation capacity.

Increased operating pressure makes separation capacity of RO membranes reach its maximum values  $\varphi$  MGA-100P = 0.977,  $\varphi$  MGA-80P = 0.968 (Fig. 6, horizontal sections). The investigations showed that here the pressure practically coincides with the pressure range that corresponds to the linear character of  $G(P)$  (Fig. 5). High values of separation capacity, at this section of dependency  $\varphi(P)$ , are presumably achieved due to the optimum values of permeate flow rate. Further increase in operating pressure, up to the values exceeding 2.5-3.0 MPa, leads to considerable decrease in separation capacity, which can be explained by obvious effects of concentration polarization and by partial solubility of lactose in the layer of "bound water".

The investigations of the effects produced by temperature of the whey on permeability  $G$  and on separation capacity  $\phi$  of the membrane were undertaken within a limited range of temperatures: 15 to 40°C, as higher temperatures result in irreversible changes to physical and chemical properties of whey. The experiments proved that permeability  $G$  increases considerably as the temperature gets higher. This is quite consistent with the results of the investigations and it can be explained by lower viscosity of permeate in the pores of the membrane and also by lower viscosity of the whey under investigation, which leads to smaller effects of concentration polarization as a result of higher diffusion quotient  $D_0$  and, consequently, as a result of better outflow of the dissolved substance from the surface of the membrane. However, increased temperature of the whey under investigation leads to considerable decrease in separation capacity  $\phi$  of the membrane. Such drastic decrease in  $\phi$  is by no means consistent with the results of the investigations, but it can be explained from the perspective of the surface force theory. It follows from this theory that the increase in temperature results in thinner polymolecular adsorption layers of water (bound water), which occurs due to the destruction of H-grid connections that are responsible for structural long-range interaction.

## V. CONCLUSIONS

The obtained results enable the basic conclusions as follows. It has been established that UF process can be feasibly applied for concentration of macromolecular substance of  $C = 8\%$  MMS under the parameters as follows:  $u \geq 1.5 \text{ m/s}$ ;  $P = 0.3 \text{ MPa}$ ;  $t = 20 \pm 5^\circ\text{C}$ . It has been proved that ceramic membrane of CUFE (0.01) Type can be recommended as the most preferable technical solution for separating whey without any preliminary treatment.

It has been also established that RO process should be run at a temperature of  $t = 20 \pm 5^\circ\text{C}$ ; operating pressure of RO process should be kept within the range of  $P = 2.0 - 2.4 \text{ MPa}$  for concentration of  $C = 5 - 15\%$  DS and  $P = 3.8 - 5.0 \text{ MPa}$  for concentration of  $C = 15 - 22\%$  DS; RO process can be effectively applied in cases where the concentration of dry dissolved substance (lactose) amounts to  $C = 20\%$  DS, employing membrane of MGA-80P Type for the purpose; thereat, the whey is demineralized by approximately 20%.

The undertaken analysis of the effects produced by the basic technological parameters on the characteristics of RO concentration of whey enables the conclusions as follows:

- operating pressure of RO process should be maintained within the range of  $P = 2.0 - 2.4 \text{ MPa}$  for concentration of  $C = 5 - 15\%$  DS and  $P = 3.8 - 5.0 \text{ MPa}$  for concentration of  $C = 15 - 22\%$  DS;

- RO process does not require temperatures higher than that of the environment, i.e. it is advisable that the process should be run at  $t = 20 \pm 5^\circ\text{C}$ ;

- RO process can be effectively applied to the cases where the concentration of dry dissolved substance (lactose) amounts to  $C = 20\%$  DS;

- RO process should be run applying membrane of MGA-80P Type; thereat, the whey is demineralized by approximately 20%.

Experiments of investigations of RO membranes separation capacity for dissolved mineral substance showed that the mineral substance that is contained in the whey in the state of molecular solution includes such salt chlorides as KCl, NaCl, CaCl<sub>2</sub> (94 – 96 % of all mineral substance in UF permeate). RO membrane separation capacity for all the salts under investigation remains constant irrespective of the increased concentration that makes it possible to conclude that it is advisable to use membranes of MGA – 80P Type in the process of RO concentration of whey. Thereat, the whey is demineralized approximately by 20%. The implementation of this membrane method of waste-free processing of whey at dairy plants will allow to enter the development of the food industry in the conditions of new techno-economical paradigm.

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