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The Research of Whey Permeate Mineral Profile at Different Stages of Membrane Filtration

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ABSTRACT

Whey permeate powder is widely used in technologies of various line groups of food products, but the main limiting factor of its application is its high ash content. This research aimed to establish the efficiency of ash reduction and change of mineral profile at various stages of production for obtaining demineralized whey permeate powder suitable for further usage in technologies of lactose. The experiments were carried out following the referee method and the common methods used in research practice. The objects of research were cheese whey and its concentrate and permeate obtained in the process of ultrafiltration (UF), nanofiltration (NF), electrodialysis (ED), vacuum-evaporating and spray drying. UF made it possible to remove partially Ca^{2+}, total phosphorus, and Mg^{2+} from cheese whey, NF was effective in removing part of K^{+}, Ca^{2+}, Fe^{2+}, Mg^{2+}, Cu^{2+}, Cl^{-} and total phosphorus from UF-permeate. Using polymer membranes made it possible to obtain the NF-concentrate containing mainly lactose and increase the efficiency of ED due to their high permeability relative to water, as well as their ability to eliminate proteins and partially some ions of mineral salts. The mass fraction of ash in the finished product decreased by 93.0% compared with cheese whey, as well as Na^{+} and K^{+} by 89-94%, and Ca^{2+} and Mg^{2+} by 60-75%; the total phosphorus – by 78%; chlorides – by 70%. The obtained results allow to justify the technological operation sequence to produce a product suitable for further usage as a raw material for highly purified lactose.

Keywords: ash content, electrodialysis, nanofiltration, ultrafiltration, whey

INTRODUCTION

In the past year the cheese world market increased by 2.0% and became 25.3 mln. tons accordingly, milk whey amount also increased, its unique chemical composition (50% of milk dry solids, including 95% of lactose, 80% of minerals, 20% of proteins and 10% of milk fat **[1]**) allows to produce new ingredients on its basis, both using membrane methods of separation (microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO)) due to pressure gradient and osmotic membrane. This fractionates solution components by size and molecular structure **[2]**, **[3]**. Membrane methods are used for obtaining from milk whey or milk of high molecular concentrate of proteins, fat and lactose (retained by membrane) and low molecular fraction containing lactose and ash, in this case, the moderate temperature conditions allow the components to remain as native as possible and preserve their functional-technological properties **[4]**, **[5]**. The raw milk permeate contains lactose, mineral salts, and other low molecular compound solutions (Table 1).

The world's production and consumption of whey protein concentrates is constantly growing. According to the analytic center Fortune Business Insights data in 2022-2029, at an average annual rate of 7.4%, it will reach $18.12 bln. **[6]**. However, the permeate market accompanying the production of milk protein concentrates is developing not so actively despite its unique functional-technological properties: in 2022 it was $836.5 mln, which meant an annual increase by 4.5% **[6]**.

Table 1 Chemical composition of whey permeates.

Parameter description	Value
Mass fraction of moisture in terms of dry solids, not more than, %	2-5
Mass fraction of lactose in terms of dry solids, not more than, %	76-92
Mass fraction of fat in terms of dry solids, not more than, %	0-1.5
Mass fraction of protein in terms of dry solids, not more than, %	1.2-4.0
Mass fraction of ash in terms of dry solids, not more than, %	
Demineralized permeate	
DD 25%	7.0
DD 50%	4.0
DD 70%	2.5
DD 90%	1.0
Don-demineralized permeate	9.0

The typical whey powder is widely used both as an independent ingredient in technologies of various product line groups of food products (dairy, confectionary, baking, grocery production, milk replacer, feed for farm animals etc.) and as a raw material for lactose production, including pharmacopoeia. The production of whey permeate powder is rapidly developing in our country, but there are no technologies for obtaining highly refined lactose. One of the main limiting factors for applying whey permeate for food and feed purposes is the mass fraction of minerals **[7]**. The number of food technologies excludes the application of ingredients with high ash content. The mineral salt content in cheese whey varies from 0.3 to 0.8%, up to 15.0% in terms of dry matter. The quantitative ratio of anions (5831 g/l) and cations (3323 g/l) of whey is the same as that of milk. Na, K, Ca, Mg, Cl, P predominate among minerals **[8]**. Sodium, potassium and chloride are electrolytes that belong to the diet's excessive macronutrients. Their high concentration in tissues and blood of the body disrupts water balance, osmotic pressure, acid-base balance (pH) and results in the development of diseases of kidneys, heart, gastrointestinal tract, liver, etc., so limiting their content in food products is worth limiting.

In accordance with the standard for dry permeates of raw milk (CXS 331-2017), the maximum ash content in whey permeate powder should not exceed 12.0%. Membrane methods allow fractionation of the raw materials and refining of the finished product from minerals, which prevent its application in food technologies. The permeate powder production includes the following operations: ultrafiltration, nanofiltration, and electrodialysis, which are widely used methods of demineralization, allowing the decrease in ash content at various technological stages **[9]**.

Ultrafiltration membranes with pore sizes from 0.01 to 0.1 μm retain fat and almost all the whey proteins. In the process of ultrafiltration, ions and minerals presented in the concentrate are connected with proteins (calcium, magnesium, phosphates and citrates), and free ones are fully transferred to the permeate (sodium, potassium, chloride).

Nanofiltration membrane with pore size from 0.001 to 0,01 μm makes it possible to retain substances with molecular weight from 100 to 300 Da, in this respect, minerals, some organic acids, non-protein nitrogen and a small amount of lactose (0.07%) pass into the nanofiltration permeate. The permeate production according to the known technology (Table 1) using ultra- and nanofiltration allows to obtain the finished dry whey permeate with partial demineralization, in which about 25% of salts are removed, and the mass fraction of ash in terms of dry solids is not more than 7% **[10]**.

Electrodialysis may reduce the minerals content due to ions separation by transportation through the osmotic membranes of cation and anion exchange under the influence of direct current, which results in a high degree of demineralization **[11]**. Unlike nanofiltration, electrodialysis does not require high hydrostatic load of the inlet solution to induce the mass exchange processes. The electric field provokes ion migration, the charged particles released from the soluble salts are easily removed from solution, and the uncharged ones, such as sugars and lactose, remain in the solution. The barriers that carry out ion transportation are ion-exchange membranes of various types and selective characteristics.

The research aims to establish the efficiency of ash reduction and change of mineral profile at various stages of production for obtaining demineralized whey permeate powder suitable for further use in technologies of lactose.

The following tasks have been set up to achieve this goal:

- to study the dynamics of ash and mineral profile of cheese whey permeate changes at various stages of production;
- to determine the degree of sample demineralization and the efficiency of sequential application of ultrafiltration, nanofiltration (with polymer membranes) and electrodialysis methods.

Scientific Hypothesis

In accordance with the standard for dry permeates of raw milk (CXS 331-2017), the maximum ash content in dry whey permeate should not exceed 12.0%. Membrane methods allow not only the fractionation of the raw materials but also refining of the finished product from minerals which refrain its application in food technologies. The combination of membrane methods of processing whey in a certain sequence using polymer membranes can provide a high degree of its purification from minerals for further application of highly refined whey permeate in technologies of lactose.

MATERIAL AND METHODOLOGY

Samples

The objectives of research included the whey permeates obtained after ultrafiltration, nanofiltration, electrodialysis and spray drying. The raw material for dry permeate production was cheese whey, produced at the PJSC DP branch office "Voronezhskii" "Kalacheevskii Cheese Factory".

Chemicals

All chemicals purchased by Stock Company "Lenreactiv" (Russia) were of analytical grade quality.

Instruments

The ultrafiltration unit of UF-1 type (DMP Ltd supplier, Stavropol, Russia) with polymer membranes Alfa Laval GR73PE 6338/30 (polyether sulphone with molecular weight cut-off (MWCO) 10kDa, Alfa Laval Corporate AB manufacturer, Lund, Sweden), the nanofiltration unit of NF-1 type of SD-Filtration brand (SiccaDania A/S, Denmark) with polymer membranes DOW FilmTecT Hypershell 245-8038 (MWCO 300Da, DuPont de Nemours, Inc. manufacturer, Wilmington, USA), the electrodialysis unit of ED2*EWDU6*EDR-II/250 type (LLC MEGA ProfiLine, Russia) using ion-selective membranes (RALEX CMH-PES and RALEX AMH-PES, MEGA a.s. manufacturer, Prague, Czech Republic), the vacuum-evaporating unit TH-TVR4 (LLC Kroneswerk Steinecker, Germany) and drying unit VRD5 (VZDUCHOTORG, spol. s r.o., Slovakia) were used for sample preparation.

Laboratory Methods

The experimental research was carried out following the referee method and the common methods used in research scientific practice (Table 2). Physical and chemical parameters of whey permeate were determined in scientific testing laboratories of FSFEI HE "All-Russian Research Institute of Dairy Industry", the Federal Reserve "State Regional Center for Standardization, Metrology and Testing in Moscow and Moscow region", the Federal Reserve "State Regional Center for Standardization, Metrology and Testing in Saint Petersburg and Leningrad region", FSFEI HE "Voronezh State University of Engineering Technologies".

The mass fraction of dry solids was analyzed following GOST 29246-91 **[12]**, **[13]** using oven drying; mass fraction of total protein was determined according to GOST 34454-2018 **[14]** with help of Kjeldahl method; mass fraction of lactose was analyzed following GOST 33958-2016 **[15]** using polarimetry; mass fraction of ash was determined according to GOST R 56833-2015 **[16]** with help of dry combustion method; chlorides content was analyzed following GOST R 54045-2010 **[17]** using capillary electrophoresis; calcium content was determined according to GOST R 55331-2012 **[18]** with help of the atomic absorption spectrometry; total phosphorus content was analyzed following GOST 31980-2012 **[19]** using capillary electrophoresis; sodium content was determined according to GOST EN 15505-2013 **[20]** with help of the atomic absorption spectrometry; potassium content was analyzed following ISO 8070:2007 **[21]** using atomic absorption spectrometry; iron content was determined according to GOST EN 14084-2014 **[22]** with help of the atomic absorption spectrometry; magnesium content was analyzed following MG 4.1.3606-20 **[23]** using atomic absorption spectrometry; copper content was determined according to GOST EN 14084-2014 **[22]** with help of atomic absorption spectrometry.

Description of the Experiment

Sample preparation: The whey was previously purified from fat and casein fume using a vibratory sieve, then pasteurized at a temperature t = (75 ±2) °C for 5 min, then cooled to t = (10-15) °C and sent to the ultrafiltration unit of UF-1 type with polymer membranes. The permeate received was sent to the nanofiltration unit of NF-1 type of SD-Filtration brand at a temperature t = (10 ±2) °C and the process pressure up to 2.5 MPa; in this case it was thickened up to dry solids content of 27.5%. The electrodialysis was carried out at the electrodialysis unit of ED2*EWDU6*EDR-II/250 type at a temperature t = (15 ±2) °C using ion-selective membranes. Stage I was conducted with negatively charged membrane for 4 h. Stage II was neutralization process up to 7.0 pH for 2 h. Stage III was conducted with charged positively membrane for 25 min until the electrical conductivity of 0.8 $mS.cm^{-1}$. Further, the permeate was thickened up to (54-55%) at vacuum-evaporating unit TH-TVR4 (P = 0.09 MPa, inlet temperature of 70-75 °C, outlet – 40 ±5 °C) and sent to crystallization at 33-35 °C for 3-4 hours and then to 10-15 °C for 10-12 hours. Subsequent drying was carried out at the unit VRD5 at an inlet temperature to the drying tower of 170-200 °C, at outlet – (70-100 °C). Then the whey permeate powder was cooled to 30 ±5 °C.

Number of samples analyzed: 7 samples were analyzed.

Number of repeated analyses: The experimental studies of each sample were carried out 3 - 5 times.

Number of experiment replication: The number of repetitions of each experiment to determine one value was three times.

Design of the experiment: The methodological research strategy realized in this study is represented in Figure 1.

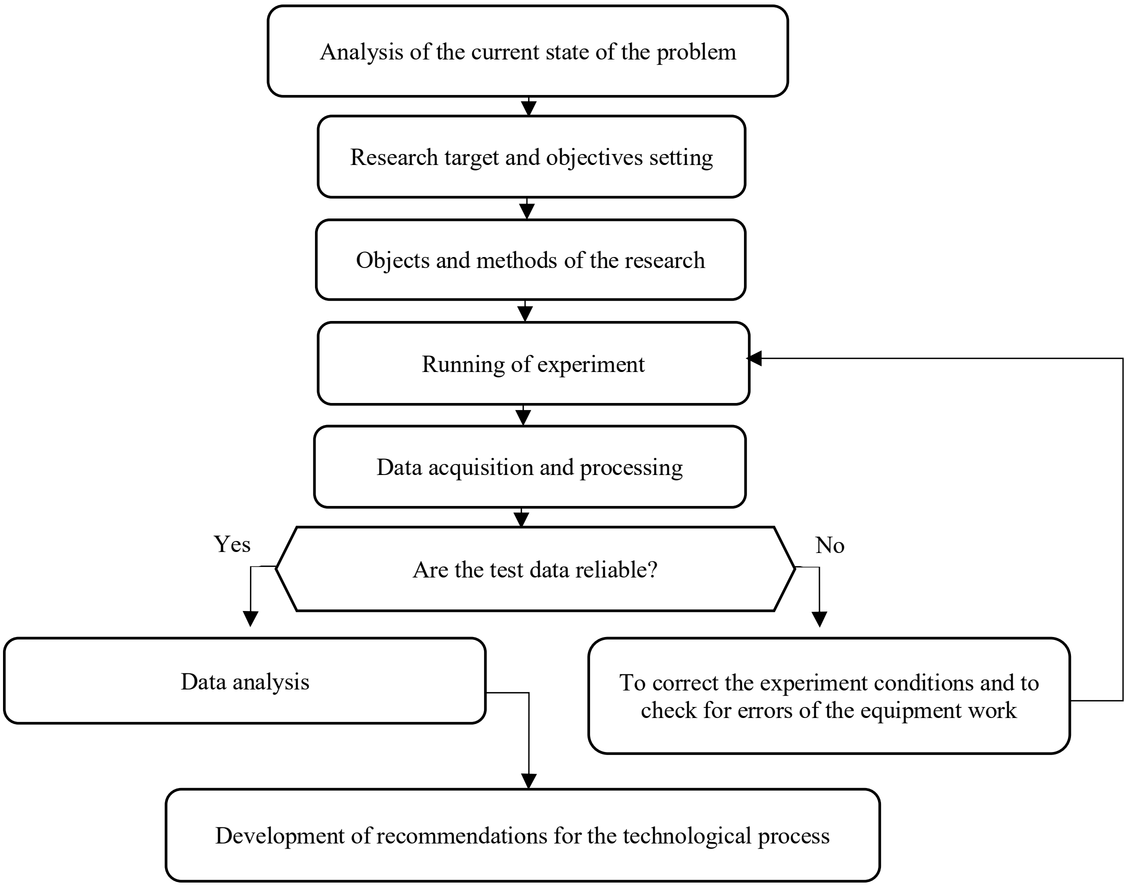

Figure 1 Methodological diagram of conducting the research.

Statistical Analysis

Calculations were carried out by methods of mathematical statistics with the help of Microsoft Office application for home and study 2021 for Mac and Statistica (USA). (Microsoft Corporation, WA, USA). The normal distribution of continuous variables was determined using Shapiro-Wilk test. Data were expressed as mean ± standard deviation and median (minimum value~maximum value) for normally and non-normally distributed data, respectively. The P-value, which was less than or equal to 0.05, was used to determine provided the findings were significant. The limitations of experimental studies included errors and uncertainties of the analysis methods used, which affected the represented results. The results are presented considering the errors found using the least squares method.

RESULTS AND DISCUSSION

Technologies for the production of whey powder **[24]** and its derivatives **[25]** (Figure 2) require the study of mineral composition at UF-filtration, NF-filtration, and electrodialysis stages **[2]**. The chemical composition and mineral profile change of the tested samples at various stages of cheese whey processing were established in the experiments (Table 2). The use of ultrafiltration allowed to concentrate **[9]** and partially purify from minerals the total protein of cheese whey **[1]**, in this case, the major part of lactose and salt passed into the UF-permeate, predominantly in the ion-molecular distribution **[26]**.

Nanofiltration effectively removes part of mineral salts from UF-permeate and increases lactose mass fraction in NF-concentrate samples **[27]**. The optimal parameters of the process (P = 2 MPa and t = 15 °C) ensured a higher degree of purification from chlorides without significant loss of milk sugar **[10]**. The part of lactose, calcium (30%), phosphorus (20%), sodium (10%), potassium (25%), chlorides (50%), iron (80%), magnesium (70%), and copper (85%) pass into NF-permeate **[28]**, which promotes its application as the raw material for isotonic drinks production **[29]**.

The electrodialysis treatment of NF-concentrate was carried out at a temperature (15 ±2 °C) **[30]** with the subsequent control of electrical conductivity and pH in the process **[31]** (Figure 3). The number of cations in NF-concentrate decreased during stage I of electrodialysis that led to low values of pH. In this case the protein in the NF-concentrate is unstable and may precipitate out **[32]**, which negatively affects the drying and physical-chemical characteristics of the finished product **[33]**. Therefore, the mixture of solutions KOH and NaOH (1:1) was added for further adjustment of this parameter to pH value of 6.2. The process is considered to be finished when the conductivity reaches 0.8 $mS \cdot cm^{-1}$ for the product **[34]** with a demineralization degree of 90%.

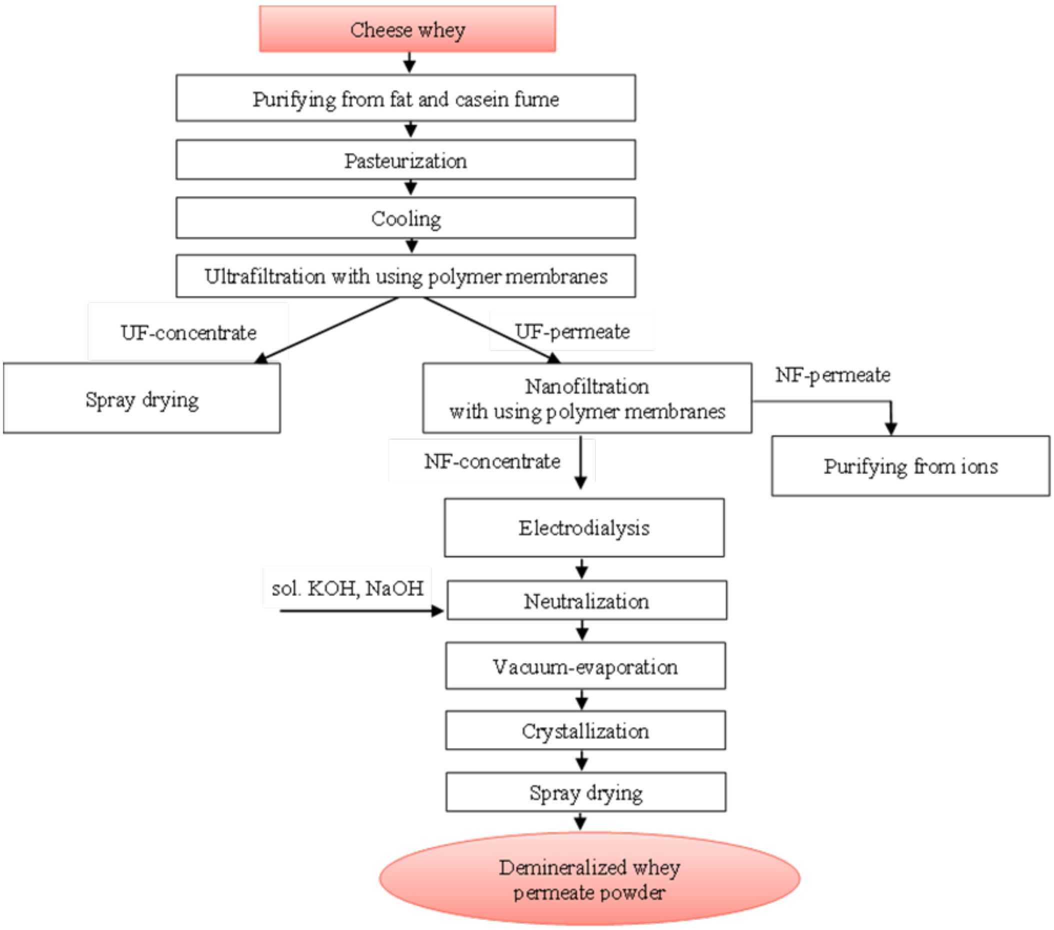

Figure 2 Cheese whey processing scheme.

There was an increase of UF-permeate total ash compared to cheese whey (more than 70%), due to free ion transfer and they were not associated with other components. It decreased by 25.4% for NF-concentrate and the mineral profile changed toward the increase in the concentration of Na^+, K^+, Cl^-. The electrodialysis application for the further demineralization of NF-concentrate of cheese whey made it possible to achieve the removal of Na^+ and K^+ by 89-94%, and of Ca^{2+}, Mg^{2+} by 60-75%; of total phosphorus – by 78%; of chlorides – by 70%.

It is well known that the efficiency of monovalent ion removal (K^+, Na^+ and Cl^-) is higher than that of multivalent ones (Ca^{2+}, Mg^{2+}, SO_4^{2-} and PO_4^{3-}) **[35]**. Several scientists **[36]**, **[37]**, **[38]** established the following deionization scheme for cations: $K^+ > Na^+ > Ca^{2+} > Mg^{2+}$; for anions: $Cl^- > SO_4^{2-} > PO_4^{3-} > LA^- > CA^{3-}$. It is accounted for the smaller hydrodynamic radius [38] and the higher diffusion coefficient of monovalent ions compared to multivalent ones **[39]**. Moreover, the mineral and organic profiles of the initial raw material **[40]** and the permeate technology **[11]** also influence the efficiency of demineralization **[41]**.

Table 2 Chemical composition and mineral profile change of the tested samples.

Parameter description	Cheese whey	Ultrafiltration using polymer membranes		Nanofiltration using polymer membranes		Electrodialysis	Spray drying (whey permeate powder with the DD 90%)
		UF-concentrate	UF-permeate	NF-concentrate	NF-permeate		
Mass fraction of dry solids, %	6.01 ±0.05	27.8 ±0.07	4.21 ±0.03	21.56 ±0.07	0.97 ±0.02	21.40 ±0.09	97.73 ±0.10
Mass fraction of lactose, %,	4.43 ±0.36	7.54 ±0.40	3.48 ±0.33	19.21 ±0.53	0.35 ±0.13	20.93 ±0.51	90.60 ±0.47
including in dry matter, %	73.71 ±0.21	27.12 ±0.24	82.66 ±0.18	89.10 ±0.30	36.08 ±0.08	97.80 ±0.30	92.70 ±0.29
Mass fraction of protein, %,	0.84 ±0.03	19.54 ±0.16	0.16 ±0.02	0.27 ±0.03	-	0.26 ±0.02	2.31 ±0.05
including in dry matter, %	13.98 ±0.04	70.29 ±0.12	3.80 ±0.03	1.25 ±0.05	-	1.21 ±0.06	2.36 ±0.08
Mass fraction of ash, %,	0.42 ±0.02	0.62 ±0.03	0.51 ±0.03	1.95 ±0.04	0.59 ±0.03	0.12 ±0.02	0.55 ±0.03
including in dry matter, %	6.99 ±0.04	2.23 ±0.05	12.11 ±0.03	9.04 ±0.06	60.82 ±0.03	0.56 ±0.06	0.56 ±0.07
Chloride content, mg/100 g,	0.87 ±0.03	1.32 ±0.05	0.86 ±0.04	1.98 ±0.05	2.16 ±0.04	0.58 ±0.02	3.57 ±0.06
including in dry matter, %	0.014 ±2.15 % relative	0.005 ±2.02 % relative	0.020 ±2.68 % relative	0.009 ±1.43 % relative	0.223 ±1.96 % relative	0.003 ±1.94 % relative	0.004 ±0.89 % relative
Calcium content, mg/100 g,	21.32 ±0.15	156.18 ±0.41	48.61 ±0.19	189.79 ±0.44	86.24 ±0.23	17.58 ±0.12	81.7 ±0.21
including in dry matter, %	0.35 ±0.77% relative	0.56 ±0.26% relative	1.15 ±0.55% relative	0.88 ±0.28% relative	8.89 ±1.17% relative	0.082 ±0.55% relative	0.084 ±0.18% relative
Total phosphorus content, mg/100 g,	64.36 ±0.05	138.54 ±0.10	44.39 ±0.03	173.53 ±0.11	76.26 ±0.06	49.19 ±0.05	215.5 ±0.10
including in dry matter, %	1.07 ±0.46% relative	0.50 ±0.16% relative	1.05 ±0.39% relative	0.80 ±0.19% relative	7.86 ±1.07% relative	0.230 ±0.26% relative	0.221 ±0.08% relative

Table 2 Cont.

Parameter description	**Cheese whey**	**Ultrafiltration using polymer membranes**		**Nanofiltration using polymer membranes**		**Electrodialysis**	**Spray drying (whey permeate powder with the DD 90%)**
		UF-concentrate	**UF-permeate**	**NF-concentrate**	**NF-permeate**		
Sodium content, mg/100 g,	54.21 ±0.02	141.58 ±0.08	106.96 ±0.06	539.63 ±0.76	183.73 ±0.15	16.35 ±0.003	88.60 ±0.04
including in dry matter, %	0.90 ±0.44% relative	0.51 ±0.16% relative	2.54 ±0.38% relative	2.50 ±0.23% relative	18.94 ±1.07% relative	0.076 ±0.22% relative	0.09 ±0.08% relative
Potassium content, mg/100 g,	193.4 ±0.10	177.1 ±0.09	198.5 ±0.12	794.80 ±0.95	205.28 ±0.16	36.84 ±0.007	159.2 ±0.06
including in dry matter, %	3.22 ±0.44% relative	0.64 ±0.15% relative	4.71 ±0.39% relative	3.69 ±0.22% relative	21.16 ±1.07% relative	0.172 ±0.22% relative	0.16 ±0.07% relative
Iron content, mg/100 g,	0.46 ±0.03	2.3 ±0.05	0.42 ±0.02	0.37 ±0.02	2.26 ±0.11	0.13 ±0.01	less than 1.0
including in dry matter, %	0.0007 ±3.68% relative	0.0008 ±1.21% relative	0.0010 ±2.74% relative	0.0002 ±2.87% relative	0.0233 ±3.47% relative	0.00006 ±4.06% relative	
Magnesium content, mg/100 g,	28.7 ±0.28	6.3 ±0.05	3.71 ±0.05	6.87 ±0.06	9.90 ±0.15	3.9 ±0.04	168.3 ±0.48
including in dry matter, %	0.047 ±0.91% relative	0.002 ±0.52% relative	0.009 ±1.03% relative	0.0032 ±0.60% relative	0.1021 ±1.79% relative	0.018 ±0.73% relative	0.0172 ±0.20% relative
Copper content, mg/100 g,	0.294 ±0.03	0.28 ±0.02	0.276 ±0.02	0.14 ±0.007	0.23 ±0.01	0.05 ±0.004	0.17 ±0.009
including in dry matter, %	0.0005 ±5.52% relative	0.0001 ±3.70% relative	0.0007 ±3.98% relative	0.0001 ±2.66% relative	0.0024 ±3.21% relative	0.00002 ±4.21% relative	0.00002 ±2.70% relative

The conducted research proved that monovalent ions were removed faster than multivalent ones due to their lower mobility. Moreover, considering their ability to form complexes with proteins **[8]**, the preliminary concentration of whey protein via UF or NF polymer membranes significantly affected the speed and process of demineralization by electrodialysis **[42]**. The filter area of the NF-membrane allow to remove most of the multivalent ions from NF-concentrate before ED. The high permeability of polymer membranes relative to water as well as their ability to eliminate proteins and partially some ions of mineral salts made it possible to obtain the NF-concentrate containing mainly lactose and increase the efficiency of electrodialysis due to the fact that the ED is not enough effective for multivalent ions removing. The total content of the inorganic ions was reduced by more than 93.0%. In the vacuum evaporation, crystallization, and drying process, there was no significant change in the mineral composition.

The obtained results proved the high demineralization degree of the samples and the efficiency of sequential application of UF, NF (with polymer membranes) and electrodialysis methods. The additional technological operations of cheese whey processing allowed to obtain the finished product with a demineralization degree of 90% and bring the mineral profile of dry whey permeate closer to the requirements to obtain pure lactose **[43]**.

The limitations such as concentration polarization during membrane filtrations and possible errors on a measurement could interfere with the validity and interpretation of the obtained data. Increasing of the mass fraction of dry solids in front of the membrane led to reducing of it permeate flux and efficiency.

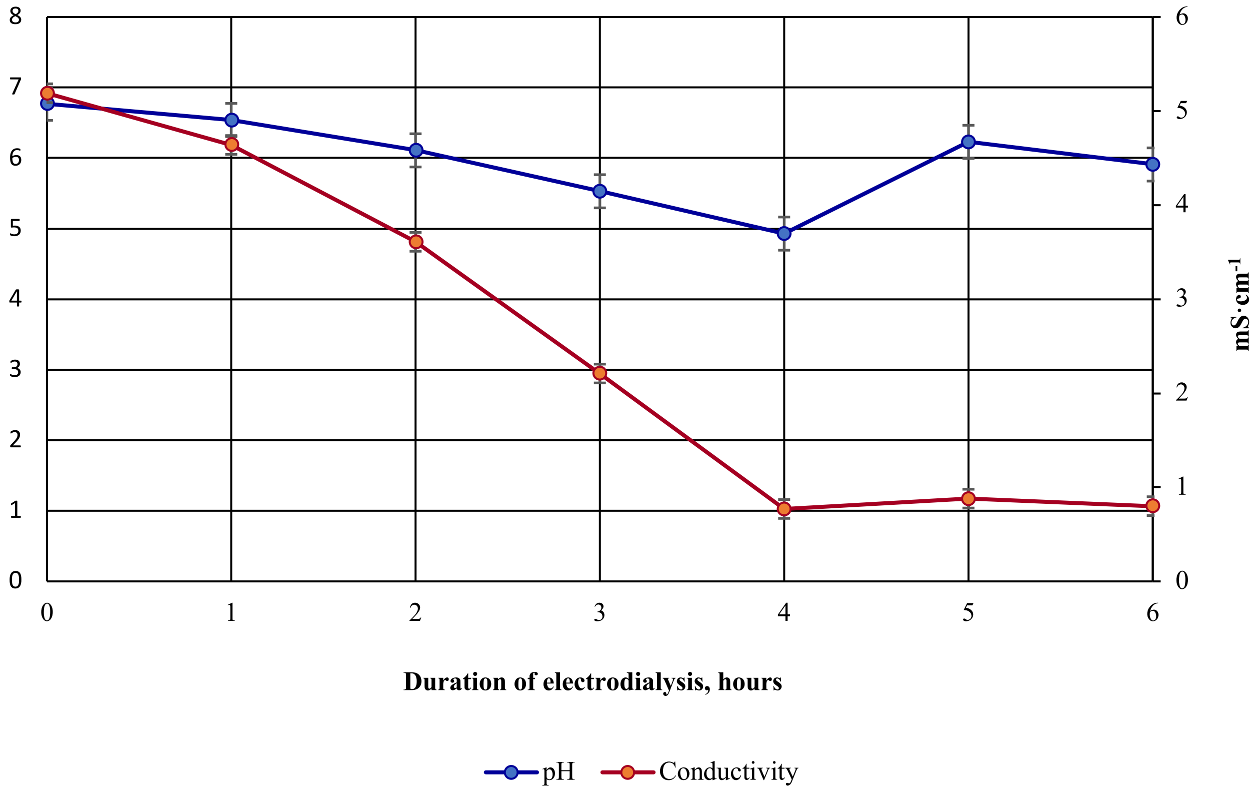

Figure 3 Changing pH and conductivity of NF-concentrate during electrodialysis.

CONCLUSION

The advantage of this study is running experiments in production conditions using industrial equipment. It was found that the UF with polymer membranes made it possible to remove partially Ca^{2+}, total phosphorus, and Mg^{2+} from cheese whey; the NF with polymer membranes was effective in removing part of K^{+}, Ca^{2+}, Fe^{2+}, Mg^{2+}, Cu^{2+}, Cl^{-} and total phosphorus from UF-permeate; the ED allowed to remove residual monovalent ions, and the most of Ca^{2+} and Cl^{-} from NF-concentrate. The theoretical significance of this research is determining the change in the content of different components of cheese whey, including the main macro- and microelements, during technological processing using membrane equipment with only polymer membranes. Practical importance of this research is the justification of the technological operation sequence to obtain a product suitable for further usage as a raw material for highly purified lactose. Further research will be focused on the application of the obtained demineralized whey permeate for lactose manufacture.

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