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Research Paper

Demineralization of Cheese Whey by Sequential Nanofiltration (NF) and Electrodialysis (ED) Processes

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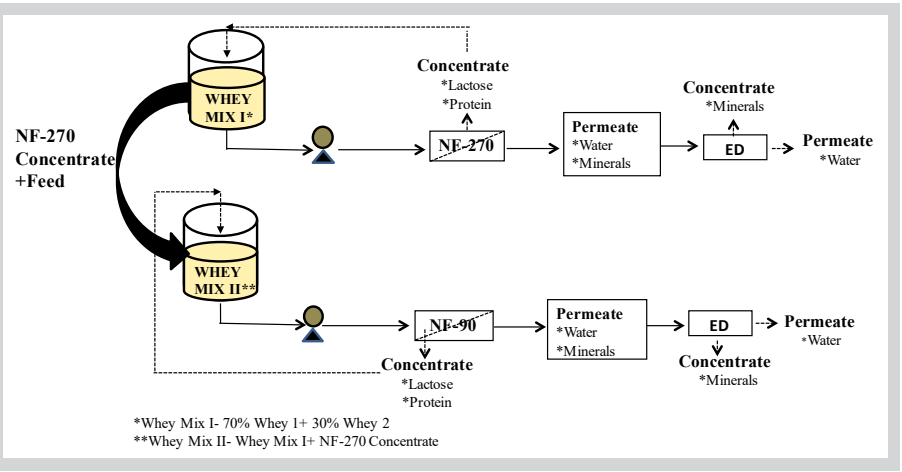

Highlights

- Sequential process including NF and ED for cheese whey demineralization.
- A 90% of rejections of protein and lactose with 65-87% of demineralization by NF.
- More than 90% of total mineral rejections from NF permeate of cheese whey by ED.

Abstract

In this study, demineralization of cheese whey by sequential nanofiltration (NF) and electrodialysis (ED) methods was investigated. For the NF step, two NF membranes labeled as NF-270 and NF-90 were tested. Approximately 90% of protein and lactose rejections were obtained with partial demineralization during NF step. For full demineralization of the NF permeate, NF followed by ED process. Two different ED systems (Tokuyama TS-1-10 with Neosepta membranes and Mega EDZ-10x4-0.8 with Ralex membranes) were used. The rejections of Na^+, K^+, Ca^{2+}, and Mg^{2+} ions from the NF permeate with Ralex membranes were calculated as 82.5%, 99.3%, 87.9%, and 92.9%, while the corresponding values were 80.2%, 99.0%, 95.6%, and 100% by Neosepta membranes, respectively.

1. Introduction

Cheese whey is a yellowish byproduct which is produced during the cheese making. This product contains all the serum phases of milk including lactose, proteins and minerals. Depending on the production method, cheese whey has different compositions. Cheese whey can be classified as the sweet whey and acid whey according to the pH values of the product obtained. The pH value of the sweet whey is around 5.6 while the acid whey has a pH value lower than 5 due to the the fact that different processes are employed in cheese production for both cases [1]. Almost all of the total solid content in cheese whey is composed of lactose. The remaining components are protein, minerals and fat [2].

Cheese whey contains organic components which are destroyed by microorganisms, thus COD and BOD values in cheese whey are quite high. Because of this, cheese whey is considered to be a serious pollutant byproduct for the environment. Nowadays, legal boundaries forbid the discharge of the cheese whey to the environment directly without any treatment. In addition, its valuable components were well understood recently. Also, developing technologies, especially membrane separation processes, give a chance to regain value added substances such as protein and lactose from cheese whey [3].

Cheese whey can be separated into its fractions by means of membrane

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separation methods. The substances recovered from cheese whey can be useful for various applications. Especially, cheese whey proteins can be used in food industry, for cosmetics production, food materials for animals, additive for pharmaceutical products, etc. [4]. For the separation of cheese whey into its fractions, microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO), electrodialysis (ED) and ion-exchange (IE) processes have been employed [5].

Divalent ions such as magnesium and calcium are good for healthy life style as they support the bones and regulate the biochemical pathways. However, the excess of divalent ions is unwanted, specifically in baby foods. On the other hand, monovalent ions such as sodium and potassium create the unwanted salty taste in different food products [6]. The rejection of monovalent and divalent ions from cheese whey provides convenience in the subsequent stage, i.e. the crystallization process [7].

For obtaining healthier products from cheese whey, the demineralization process can be a good practice. For this, ED, IE, NF and RO are the methods used for demineralization of the cheese whey. IE and ED systems are both good alternatives for demineralization of the cheese whey. However, the main disadvantage of the IE system is the regeneration of ion exchange resins for their cycle use. Thus, using some chemicals during the regeneration step is necessary and this should be frequently done. Therefore, the IE process will not be very cost-effective and effluent treatment following regeneration will be a problematic issue [8, 9].

NF process is generally used for the partial demineralization and the concentration of cheese whey. For producing cheese whey powder, the evaporation and drying processes are applied after the NF step. Moreover, the NF process helps reducing the cost of evaporation step [9]. For this reason, NF membranes can be investigated as a promising option for processing of the cheese whey.

Ecer et al. [10] applied evaporation and NF methods before ED application with Ralex membranes. According to the obtained results, NF application was more effective than evaporation. In their experiments, first NF method was used and then a higher conductivity removal was achieved by ED. Chandrapala et al. [11] studied on processing of acid whey pre-treated with MF and using different NF membranes to show the dependency of the membrane performance on both pH and temperature. Removal of divalent ions by NF was in the range of 24-50% while monovalent ions removal changed in a wide range of 67-91%.

Rice et al. [6] compared NF and ED systems for demineralization of the UF permeate of ~~the~~ cheese whey. According to the reported results in this work, NF membranes were especially helpful for removal of all ions with an efficiency of 30-70%. The applied NF membranes in that work reduced divalent ions more than monovalent ones. They suggested that the removal was in an order of $Mg^{2+}>Ca^{2+}>Na^{+}>K^{+}$. During the demineralization process, especially for removing the monovalent ions, NF application cannot meet the required performance. An undesirable salty taste was noticed in the final product of the NF process. On the other hand, removal of all ions by the ED method was achieved successfully while the average ion removal was 90%.

Suarez et al. [12] tested the diafiltration method to increase the ion removal from the cheese whey. With the first diafiltration step, about 37% of ion removal was achieved from cheese whey at 37ºC. The second diafiltration step did not exhibit a significant difference for ion removal. Elsewhere, Roman et al. [13] reported that diafiltration can increase the ion removal from the cheese whey. Authors performed a number of tests at 40ºC with XN45 NF membrane using the acid whey samples. The rejections of protein, lactose and TDS were over 90% in all experiments. On the other hand, full demineralization was not achieved with NF.

Simova et al. [14] studied on the effects of spacer thickness and electrical voltage applied on demineralization of cheese whey by ED process. They applied centrifugation method as the pre-treatment step of the cheese whey. When the electrical voltage increased during the ED operation, the demineralization time became shorter while the energy consumption increased. The spacer thickness did not have an important role for demineralization of the cheese whey by ED. Diblikova et al. [15, 16] focused on demineralization of the cheese whey by the successive MF and ED operations. They selected MF as the pretreatment step for preventing the microbial growth in cheese whey. In another work, Perez et al. [17] studied on demineralization of a synthetic UF permeate of cheese whey by ED. In both studies, it was considered that ED process can be a useful method for the demineralization of MF and UF permeates of cheese whey. According to the demineralization data obtained by ED method, the monovalent ions were removed faster than the divalent ions. It was reported that the divalent ions have lower mobilities than the monovalent ions. Moreover, they make complexes with proteins [17]. Thus, protein removal with UF or NF membranes before the ED process was considered to be helpful in the demineralization process by ED.

In the present study, sequential NF and ED systems were applied for the full demineralization of cheese whey. We studied the effectiveness of the sequential NF and ED processes by using real cheese whey samples obtained from dairy industry. For this purpose, two different NF membranes and two different ED systems were tested and compared to obtain the optimal conditions for demineralization of cheese whey. First, we wished to separate the protein and lactose using NF method by the partial demineralization. Then, we focused on the complete desalination of the NF permeate of the cheese whey by ED for water recovery and reuse.

2. Materials and methods

Cheese whey samples were obtained from Progida Co. which is a subsidiary of Gürsüt Co. in Tire Organized Industrial Zone, Izmir, Turkey. Two types of cheese whey were received from the company. The properties of the cheese whey samples and their mixture are summarized in Table 1. As could be observed in Table 1, the cheese whey-1 is the sweet whey and the cheese whey-2 is the acid whey. The pH and other properties of whey samples are different from each other. All experiments were done by mixing two different cheese whey samples.

SEPA CF II (GE-Osmonics), a laboratory scale, cross-flow flat membrane test system was used for NF experiments. The feed volume of the cheese whey was 20 L while the temperature was maintained at 18±2ºC during membrane tests. In all NF tests, NF-270 and NF-90 membranes (Dow FilmTec) were used by applying 20 bar of operating pressure for NF-270 membrane and 30 bar for NF-90 membrane. The concentrate flow rate was adjusted as 96 L/h during NF operation.

The characteristics of NF membranes were summarized in Table 2. A pre-treatment was not applied to cheese whey samples before the NF operation. During the NF experiments, the concentrate stream was circulated back to the feed tank and the permeate stream was collected for the ED experiments as the following step in ED operations.

Table 1
The compositions of cheese whey samples and cheese whey mixture

Cheese Whey 1						Cheese Whey 2					
pH	**Conductivity (mS/cm)**	**TDS (g/L)**	**Salinity (‰)**	**Protein (%)**	**Lactose (%)**	**pH**	**Conductivity (mS/cm)**	**TDS (g/L)**	**Salinity (‰)**	**Protein (%)**	**Lactose (%)**
5.27	10.60	5.81	6.01	0.44	3.81	4.47	7.19	3.81	3.96	0.75	5.51

Cheese Whey Mixture-I (70% CheeseWhey-1 + 30% CheeseWhey-2)					
pH	**Conductivity (mS/cm)**	**TDS (g/L)**	**Salinity (‰)**	**Protein (%)**	**Lactose (%)**
5.14	9.27	5.01	5.20	0.47	4.01

Cheese Whey Mixture-II (Cheese Whey Mixture-I + Concentrate of NF-270)					
pH	**Conductivity (mS/cm)**	**TDS (g/L)**	**Salinity (‰)**	**Protein (%)**	**Lactose (%)**
4.52	10.00	5.51	5.68	0.59	5.68

NF experiment was continued until 5 L of NF permeate was collected. In addition, samples were taken from feed tank, concentrate and permeate streams periodically during NF operation for measurements. Conductivity, salinity and pH values were recorded for each samples collected. After the NF test with NF-270 membrane was completed, the mixture of the feed and the concentrate streams was used as the feed for the test carried out by using NF-90 membrane in the next experiment.

The permeate streams of NF-270 and NF-90 membranes were used as the feed sample for the ED operation. Figure 1 shows the diagram of sequential NF and ED processes. In all ED tests, two different ED systems were employed. Mega EDZ-10x4-0.8 Model ED system has ten pairs of Ralex CMH-PES and AMH-PES ion exchange membranes. Tokuyama TS-1-10 Model ED system contains a stack of 10 pairs of Neosepta AMX and CMX ion exchange membranes. The characteristics of ion exchange membranes employed in both ED systems were summarized in Table 3. Electrode rinse and concentrate compartments in both ED systems contain Na_2SO_4 solution with an electrical conductivity of 500 µS/cm. During ED operations, a constant electrical voltage of 10 V was applied. The ED tests were continued until the electrical current dropped to 0.02 A.

2.1. Analyses

During the NF tests, total dissolved solid (TDS), pH, temperature, conductivity and salinity of the samples were measured by the Hach-Lange-HQD multimeter every 15 min. Also, flow rates of permeate and concentrate streams were recorded for the calculation of flux. Every hour, samples were taken from permeate, concentrate and feed streams for protein and lactose analyses. Protein analyses were made by Kjeldahl method [20] and HPLC was employed for determination of lactose [21] by using Agilent Technologies 1200 model HPLC device with RID detector. Cation analyses (Na^+, K^+, Ca^{2+}, Mg^{2+}) were performed by a Shimadzu AA-7000 model atomic absorption spectrophotometer. All rejections (R) were calculated using Equation 1.

$$R(\%)=[(C_o-C_p)/C_o)]*100 \quad (1)$$

Table 2
The specifications of NF membranes [18]

Membrane	NF-270	NF-90
Producer	DOW FILMTEC	DOW FILMTEC
Material	Polyamide Thin Film Composite	Polyamide Thin Film Composite
Max. Operating Temperature	45°C	45°C
Max. Operating Pressure	41 bar	41 bar
pH Range	3-10	2-11
Stabilized Salt Rejection (%)	> % 97	> % 97
Permeate Flow Rate (m^3/d)	47	7.6

Table 3
The specifications of ion exchange membranes in ED systems [19]

Membrane	Ralex CMH-PES	Ralex AMH-PES	Neosepta CMX	Neosepta AMX
Application Areas	ED EDI	ED EDI	Whey demineralization Organic Purification Inorganic Concentration Sucrose demineralization Underground Water Desalination	Whey demineralization Organic Purification Inorganic Concentration
Ionic Form	Na^+	Cl^-	Na^+	Cl^-
Electrical Resistance (Ω -cm^2)	<7.50	<8.00	2.00-3.50	2.00-3.50
Thickness (mm)	<0.70	<0.75	0.16-0.20	0.14-0.18

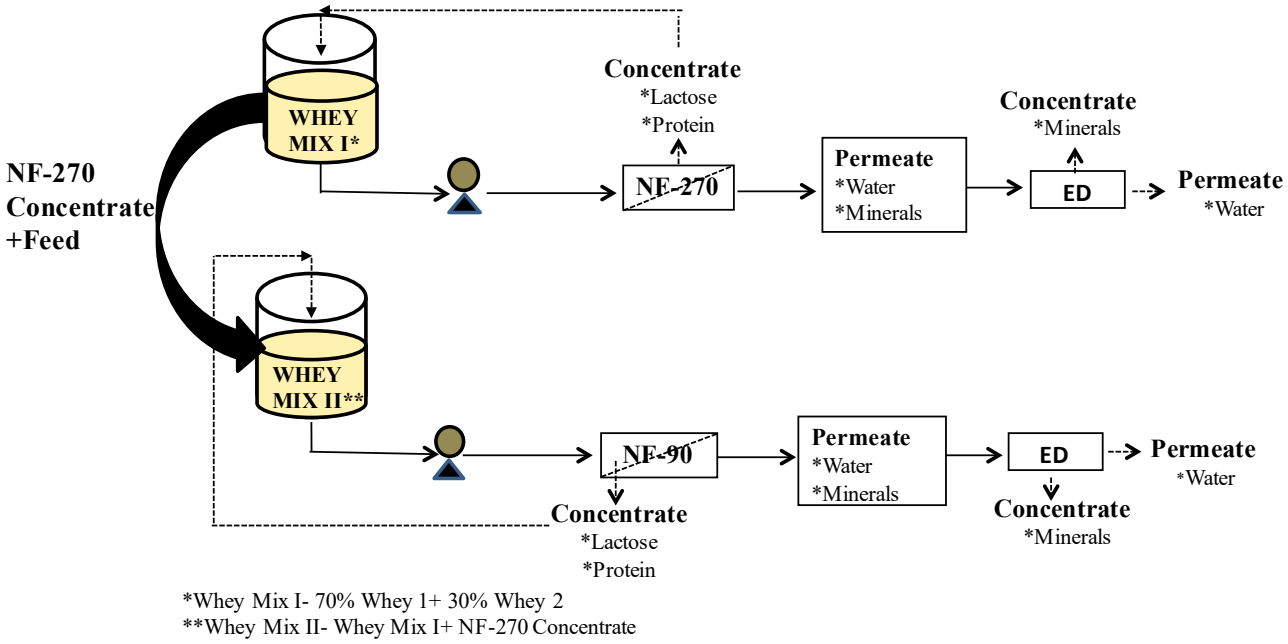

Fig. 1. Diagram of the sequential NF-ED processes applied for cheese whey demineralization

3. Results and discussion

As shown in Figure 1, the cheese whey mixture-I whose characteristics were given in Table 1 was used as the feed solution for the test run by using NF-270 membrane at a pressure of 20 bar. Concentrate stream containing lactose and protein was fed back to the feed tank during the process. The permeate stream was collected for the ED operation.

For the test carried out by the NF-90 membrane at 30 bar, the cheese whey mixture-II obtained by mixing the cheese whey mixture-I and the concentrate stream of the NF-270 membrane was used as the feed solution. The permeate of the NF-90 membrane was collected for the ED test while the concentrate stream was fed back into the feed tank during the operation.

3.1. NF Tests

The conductivity rejections from the cheese whey versus time plots for NF-90 and NF-270 membranes were shown in Figure 2. As can be seen in Figure 2, the qualities of permeates produced by two NF membranes in terms of conductivity were very different. The NF-90 membrane gave a high rejection for electrical conductivity from cheese whey. According to Table 2, the permeate flow rates of the NF membranes employed were also very different from each other. Therefore, different pressures were applied to balance the permeate flow rates. As shown in Figure 3, the average permeate flux obtained with NF-90 membrane was 16 LMH while respective value for NF-270 was 26 LMH.

Although 30 bar and 20 bar of operating pressures were applied for the NF-90 and NF-270 membranes, respectively, the permeate flux of the NF-90 membrane was still considerably lower than that of the NF-270 membrane. This was due to the fact that the pore size of the NF-90 membrane is smaller than that of the NF-270 membrane. The average pore size diameter of the NF-90 membrane is 0.55 nm while that of the NF-270 membrane is 0.71 nm [22]. Thus, the conductivity rejection with the NF-90 membrane was higher compared with NF-270 membrane.

The protein and lactose rejection values by two NF membranes were illustrated in Figures 4 and 5, respectively. The average protein rejections by NF-90 and NF-270 membranes were almost 90%±0.013. The average lactose rejections of two NF membranes were almost 100% ±3.61. Small differences between the results of both NF membranes were probably due to the analytical errors during the analysis. According to our calculations, the standard deviations of lactose and protein analyses were found as ±3.61 and ±0.013, respectively. For both membranes, the concentrations of lactose and protein in the feed solutions were very close. On the other hand, NF-90 membrane showed faster clogging than NF-270 membrane during NF operation.

Chandrapala et al. [11] worked with cheese whey pre-treated with MF for their NF tests. Experiments were carried out with various NF membranes including DK, DL and HL (GE-Osmonics) membranes at different temperatures (25-40°C) and pH values (3.0, 4.5, 7.3) at 21 bar of operating pressure. The optimum conditions for lactose and lactic acid separations were reported as pH 3 and 40°C of temperature, respectively. At those conditions, a 95% of lactose and 50% of lactic acid rejections were achieved with HL membrane. At all NF trials, the lactose rejection was higher than 93% and only 17-22% of protein passed through the membrane pores.

Suarez et al. [12] treated UF permeate of cheese whey and milk with NF membrane (Osmonics DK2540C). For both UF permeates, protein and lactose rejections were almost 100%. In our case, we were also able to achieve protein and lactose rejections in the range of 90-100% by NF membranes.

Table 4 shows the ion rejections from cheese whey by NF membranes. The ion rejections obtained by ~~the~~ NF-270 membrane were generally lower than the ion rejections given by NF-90 membrane. Especially the rejection of K^+ ions by ~~the~~ NF-270 membrane was quite low (15.7%). But the rejections of Na^+ ion by NF-270 and NF-90 membranes were close to each other. In terms of removals of divalent ions, removal of Ca^{2+} ions was achieved more successfully with NF-90 while removal of Mg^{2+} ions was similar with both membranes.

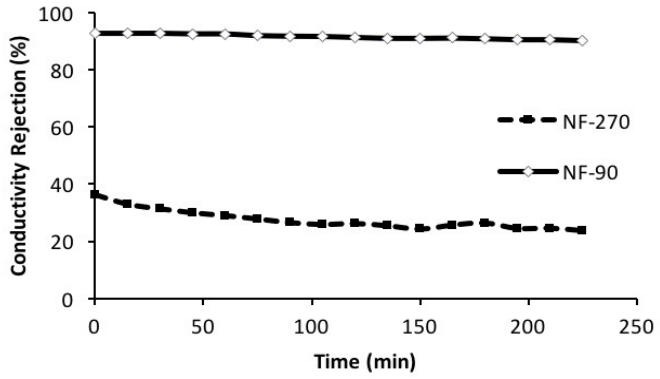

Fig. 2. Conductivity rejections from cheese whey vs. time plots by NF membranes

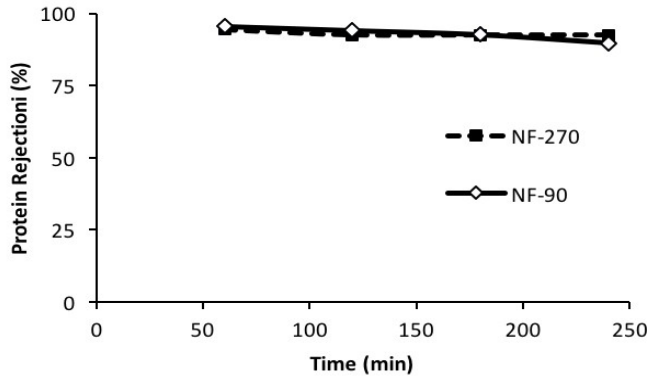

Fig. 4. Protein rejection vs. time by NF membranes

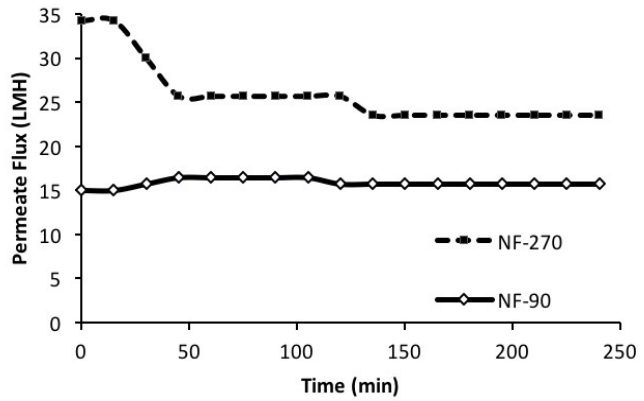

Fig. 3. Permeate flux vs. time plots by NF membranes

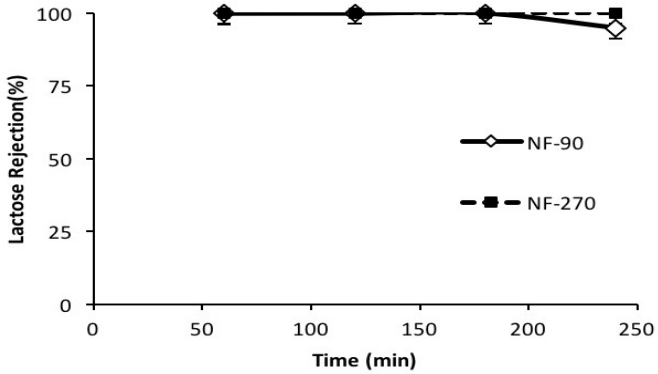

Fig. 5. Lactose rejection vs. time by NF membranes

Table 4

The ion rejections by NF membranes

Ions	Ion rejections (%)	
	NF-270	NF-90
Na^+	89.3	86.0
K^+	15.7	85.5
Ca^{2+}	55.9	82.0
Mg^{2+}	97.4	93.0

Roman et al. [13] reported that the rejections of monovalent ions from acid cheese whey were around 70-90% with NF membrane (XN45) by diafiltration at 40ºC. They reported that protein and lactose removals did not differ significantly with diafiltration and in all cases, at least 90% of removal was achieved. Especially with NF membranes, the main reason for obtaining different demineralization efficiencies was considered to be due to the formation of the limiting layer originating from the protein and the salt accumulated on the membrane surface by concentration polarization [17].

Cuartas-Uribe et al. [23] worked with cheese whey pre-treated by UF at 16-18ºC using NF membrane (DS-5-DL, GE Osmonics). They applied two different operating pressures (10 and 20 bar) and also diafiltration. According to their results, 89% of lactose removal was obtained at 20 bar. Lactose removal was 85% at 10 bar when the volume reduction factor was 2. Also at 20 bar of applied pressure, the ion removal was higher than that obtained at 10 bar.

Suarez et al. [12] tried diafiltration for removing ions. They reported that average mineral salt removal was in the range of 27-36% depending on the volume reduction factor.

3.2. ED Tests

In the literature, it was reported that ED could be also employed for cheese whey demineralization [14-17, 24]. In this study, we have also investigated the applicability of ED process as polishing step following NF process. The main target here is to demineralize the NF permeate for water reuse applications.

Figure 6 shows the conductivity change versus time plots during ED tests performed with the NF-270 permeate using two different ED systems. As shown in Figure 6, the decrease in permeate conductivity of NF-270 membrane was faster with Mega EDZ-10x4-0.8 system than with TS-1-10 ED system. The maximum conductivity rejection with TS-1-10 ED system was 99.4% after 100 min. With Mega EDZ-10x4-0.8 system, 99.7% of conductivity rejection was achieved after 70 min.

Similar results were obtained during the tests performed with the permeate of NF-90 membrane. The maximum rejection of conductivity with the TS-1-10 ED system was 97.2% after 70 min. On the other hand, a 98.3% of conductivity rejection was achieved after 50 min with the Mega EDZ-10x4-0.8 system (see Figure 7).

Perez et al. [17] reported a 90% of conductivity removal after 89 min at 25ºC by ED. They observed the similar rejection after 64 min at 35ºC with the synthetic solution of UF permeate. In this study, ED applications were performed at room temperature. A special care was taken to ensure that the temperature of the solutions heated by the pumps did not exceed 25ºC. Higher conductivity removal at lower temperature was achieved in a shorter time with our ED applications using the Mega EDZ-10x4-0.8 system.

The specifications of membranes employed in this study were summarized in Table 3. As shown in Table 3, the main differences between the membranes in both ED systems are their thicknesses and electrical resistances. Ralex membranes employed in the Mega EDZ-10x4-0.8 system are much thicker than Neosepta membranes in the TS-1-10 ED system and they have also higher electrical resistance. Thus, the TS-1-10 ED system is expected to exhibit a faster kinetic for conductivity removal from the NF permeate of cheese whey. According to the results obtained, the Mega EDZ-10x4-0.8 system achieved slightly better conductivity removal than the Tokuyama TS-1-10 ED system. Another reason of such difference obtained might be due to the difference in the flow rates of diluate and concentrate streams. In the Mega EDZ-10x4-0.8 system, flow rates of diluate and concentrate streams were 76 L/h and 70 L/h, respectively. In the Tokuyama TS-1-10 ED system, diluate and concentrate flow rates (average) in two operations were 60 L/h and 72 L/h, respectively. It was not easy to keep the flow rates of both ED systems similar to each other since both ED systems have different configurations and pumps.

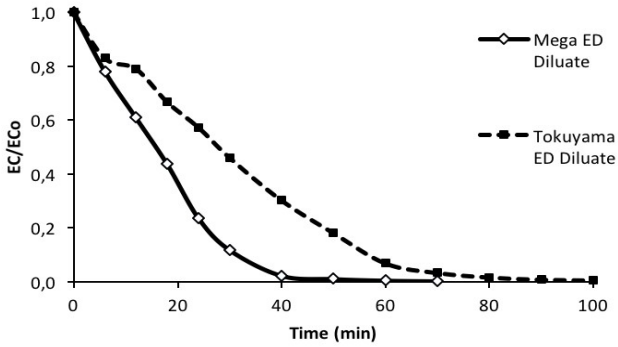

Fig. 6. EC/EC_o of diluate vs. time plots obtained by two ED systems (Feed: NF-270 permeate)

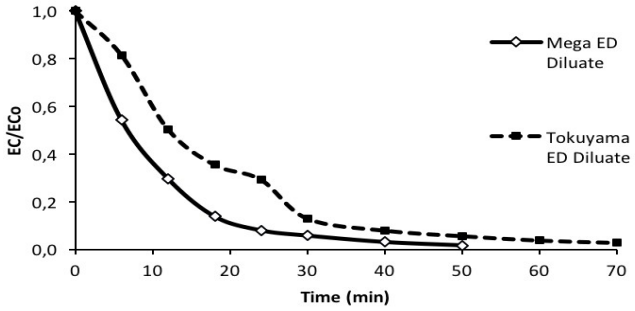

Fig. 7. EC/EC_o of diluate vs. time plots obtained by two ED systems (Feed: NF-90 permeate)

Diblikova et al. [16] tested the ED-Z mini system having Ralex membranes (AMH-PES and CMH-PES) at 20 V. During that study, the cheese whey was pre-treated with MF prior to the ED operation and it contained 1% of NaCl. According to the results they obtained, the salt content of the feed affected the duration of the ED process but removal of minerals did not change.

As shown in Figure 2, the permeate of NF-270 membrane has higher conductivity, thus it contains more salt. According to the ion rejections results given in Table 5, there was a slight difference in ion rejections obtained from NF-270 and NF-90 permeates used as the feed solutions during the ED operations. But, the demineralization period changed for two different NF permeates. For the NF-90 permeate, which has lower conductivity than the NF-270 permeate, the demineralization period was shorter. During ED operation, a 98.3% of conductivity removal was achieved after 50 min when NF-90 permeate was applied as feed solution for the Mega EDZ-10x4-0.8 system having Ralex membranes. On the other hand, for NF-270 permeate, a 99.7% of conductivity removal was obtained after 70 min with the Mega EDZ-10x4-0.8 system.

Diblikova et al. [15] obtained 83-100% of removal for monovalent ions (K^+ and Na^+) and 61-96% of removal for divalent ions (Mg^{2+} and Ca^{2+}) from cheese whey by ED operation at 20 V. We have also obtained high ion rejections even at 10 V of electrical voltage during ED operation following NF process for cheese whey. A 79-100% of removal monovalent ions and 92-100% of removal of divalent ions were achieved with TS-1-10 ED system at 10 V. With Mega EDZ-10x4-0.8 system, the removal was in the range of 77-100% for monovalent ions and 80-99% for divalent ions at 10 V.

Perez et al. [17] studied ED process using a stack of 10 cell pair SC-1 and SA-1 membranes for demineralization of synthetic UF retentate. The ion removals were 29-52%, 70-66%, 27-34%, and 27% for Na^+, K^+, Ca^{+2}, Mg^{+2}, respectively. In our study, the ions removals were high especially for divalent ions as depicted in Table 5.

According to results we have obtained, the ED process can be used for further polishing of NF permeates of the cheese whey. The performance of the ion exchange membranes employed in both ED systems were good for demineralization of NF permeate of the cheese whey. But Neosepta membranes, which have lower electrical resistance than Ralex membranes, gave a higher rejection for especially divalent ions (see Table 5).

Table 5
The rejection of monovalent and divalent ions from NF permeates of cheese whey by different ED systems

Ions	Ion rejection (%)		Ion rejection (%)	
	NF-270 permeate		NF-90 permeate	
	Mega (after 70 min)	TS-1-10 (after 100 min)	Mega (after 50 min)	TS-1-10 (after 70 min)
Na^+	88.1	79.4	77.0	81.1
K^+	99.6	99.7	98.9	98.4
Ca^{2+}	80.4	91.7	95.5	99.5
Mg^{2+}	86.6	100.0	99.1	100.0

4. Conclusions

Separation of both protein and lactose from the cheese whey can be achieved successfully with NF membranes. They can be concentrated with further evaporation and used for different purposes. If one wishes to separate protein from lactose, an UF step should be applied before the NF operation.

Following NF operation, ED method was successfully employed for demineralization of NF permeate stream for water recovery and reuse. A high efficiency for separation of divalent and monovalent ions was achieved by the ED method.

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Nomenclature list

LMH	Permeate flux (L/h/m^2)
R	Rejection (%)
C_p	Concentration in permeate at any time
C_o	Concentration in feed when t=0
t	time (min)

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