Desalination of fish meat extract by electrodialysis and characterization of membrane fouling

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Abstract−Fish meat extract (FME) desalination via electrodialysis (ED) was performed and the changes of important parameters such as membrane stack potential, brix, pH, and electrical conductivity were examined for a quantitative evaluation. The change of electrical conductivity in diluted or concentrated solutions showed that the mineral ions contained in the FME could be effectively removed. The characterization of membrane fouling indicated that the organic/inorganic components deposited, respectively, on the surfaces of anion/cation exchange membrane and resulted in the increase of membrane electrical resistance. Ion chromatograph (IC) analysis showed that the main mineral ions, such as Na^+ and Cl^+ ions contained in the FME, were removed about 82.3% at an average current efficiency of around 77.9% via ED. By considering the removal rate of mineral ions and current efficiency, the feasibility of FME desalination via ED was proved.

Key words: Fish Meat Extract, Electrodialysis, Desalination, Membrane Fouling

INTRODUCTION

Fish meat extract (FME), a byproduct of fish meat processing, contains a mass of nutrients such as sugar, protein, and special fish meat sapor, so it has been considered as a food additive. However, a high amount of mineral ions contained in FME limits its full utilization in food processing and cooking. As a result, only a few suppliers of FME currently existing have been reported, and a few patents [1,2] related to the manufacture of FME published, though there have been few, if any, reports about the desalination of FME found. It is necessary to explore the desalination of FME for its wider utilization in food industry.

Electrodialysis (ED), one type of membrane separation process, is an effective alternative method for demineralization using a direct current as the driving force to enable ion transport from one solution into another through ion-exchange membrane, which has been commonly applied in the desalination of seawater as well as brackish water and wastewater treatments [3-5]. The technology has regained its focus as a research topic because of being environmentally friendly [6], its convenience of operation and low energy consumption as compared with traditional desalination methods. Nowadays considerable research has also been performed in attempts to spread the ED application to fields such as food processing [7-9], deacidification of fruit juice [10-12], and fermentation broth desalination [13-15].

For instance, Cros et al. [16,17] reported the desalination of mussel cooking juices using ED. The study proves the feasibility of eliminating 85% of salts from a mussel cooking juice without altering significantly its aroma profile. The result reported by Wang et al. [18] showed that ED technology was feasible for desalination of oligosaccharides extract from the sweet slurry due to high desalination efficiency and oligosaccharides retention. And according to the report from Elisseeva et al. [19], the demineralization and separation of amino acids could be achieved by ED process. Recently Chindapan et al. [20] investigated the ED desalination of fish sauce for evaluating the ED performance and product quality. These studies indicated that ED technology has a significant potential for its application in food processing and fermentation industries, and it remains to be a very important work to explore the feasibility of ED application in the different fields.

The fouling of ion exchange membranes is one of the key factors to restrict the ED application in the different systems, especially in the complex solutions composed of many inorganic and organic components [21]. For the fermentation broth desalination, Ren et al. [22,23] investigated the effects of solution pretreatment and on-line cleaning of ionic membranes on membrane fouling during the $(NH_4)_2SO_4$ removal via ED from monosodium glutamate isoelectric supernatant. Bazinet and Araya-Farias [24] investigated the impact of calcium and carbonate concentrations in model solution on the composition of cation membrane fouling, and they found that carbonate had a higher buffer capacity to obtain a longer period of ED treatment and higher demineralization rate. Lee et al. [25,26] found that the negatively charged silica sol can result in membrane fouling in ED by adsorption on the surface of an anion exchange membrane, and the pulsing electric fields with different frequencies could reduce the fouling potentials. The results showed that the chemical composition and property of feed solutions can affect the formation of membrane fouling during the ED process, so it is necessary to characterize the membrane fouling occurring in the differ-

ent solutions, especially in the solutions containing the complex compositions.

The aim of this work is to investigate the feasibility of FME desalination via ED and the key parameters such as the membrane stack potential, brix, pH, and electrical conductivities were examined for a quantitative evaluation. The membrane fouling is characterized by membrane electrical resistance measurement and spectral analysis for exploring its formation mechanism and prevention methods. The ion concentrations of concentrated and diluted solutions are analyzed by ion chromatography (IC) to determine salt removal from the 7-fold diluted FME. Finally, the total energy consumption and average current efficiency of the diluted FME desalinization via ED are calculated.

MATERIALS AND METHODS

1. Fish Meat Extract

Fish meat extract (FME) used in experiments was generated based on a process involving the thawing of frozen fish, mostly tuna, which was provided by a company (Changwon, Korea). Because of its high viscosity and organic matter concentration, the original FME needed to be diluted and then could be desalted via ED. The characteristics of the 7-fold diluted FME (sample : water=1 : 6) shown in Table 1, in which the chemical oxygen demand (COD_{Cr}) and total nitrogen (TN) of the diluted sample were measured using an HS 3100 water analyzer (Humas, Korea).

2. Membrane Stack Configuration

The membrane stack configuration of a modified ED system is shown in Fig. 1. The compact membrane stack of the ED system used in the experiments is composed of two electrodes and diluted

compartments, and three concentrated compartments separated by alternately arrayed anion/cation exchange (AFX/CMX) membranes. Two sheets of bipolar membrane were placed on the sides of the membrane stack, i.e., near the anode and cathode, for inhibiting the transport of ions contained in electrode rinse solution into the adjacent concentrated compartments or otherwise. Thus the electrical conductivity change of the concentrated solution resulted only from the ion migration of the diluted compartments.

The characteristics of CMX, AFX, and the BP-1 membrane used in the experiments are shown in Table 2. Note that Pt/Ti and stainless steel were used as the anode and cathode, respectively, each having an active area of 4 cm×10 cm. In addition, the gasket thickness was about 1.0 mm, in which a reticular spacer was placed.

3. Electrodialysis Operation Conditions

With the same initial volume of 500 ml in the different compartments, peristaltic pumps were used to circulate 0.05 mol L^{-1} Na_2SO_4, 0.01 mol L^{-1} Na_2SO_4, and the 7-fold diluted FME in the electrode, concentrated compartment, and diluted compartment, respectively. The flow rates were adjusted by the peristaltic pumps to prevent
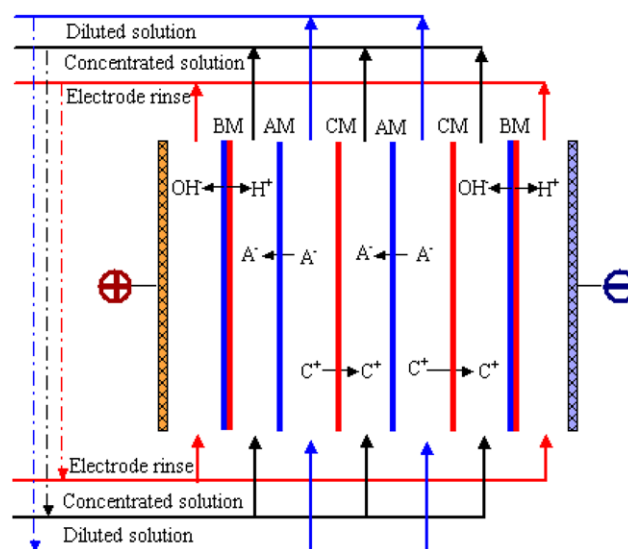

Fig. 1. Membrane stack configuration of a modified electrodialysis system. A-/C-: anions/cations; AM/CM: anion/cation exchange membrane; BM: bipolar membrane.

Table 1. Characteristics of the 7-fold diluted fish meat extract

Content	7-Fold diluted fish meat extract
Color	Deep yellow, turbid
pH	6.5
Conductivity (ms/cm)	26.4
Brix (%)	7.6
TN (mg/L)	5490
COD (mg/L)	40145

Table 2. Characteristics of the ion exchange membranes used in the experiments

Grade	Type	Electrical resistance (ohm·cm²)	Transport number (Total cations or anions)	Water content (%)	Ion exchange capacity (mg dg⁻¹)	Thickness (mm)	Burst strength (MPa)
CMX	Strongly acidic cation permeable, high mechanical strength,	2.5-3.5	>0.98	0.25-0.30	1.5-1.8	0.17-0.19	5-6
AFX	Strongly basic anion permeable, resistant against organic fouling	0.4-1.5	>0.98	0.40-0.55	2.0-3.5	0.15-0.20	2.0-3.5

Bipolar membrane	Type, group	Water splitting voltage (V) (1 mol l⁻¹ NaOH/1 mol l⁻¹ HCl, 100 mA cm², 30 ºC)		Burst strength (MPa)	Thickness (mm)	Water splitting efficiency (%)
BP-1 (Japan)	Composition of the cationic and anionic membranes	0.9-1.7		0.4-0.7	0.17-0.26	>0.98

solution volume change in different compartments due to the water pressure difference between the adjacent compartments. Before applying the electric potential, all solutions in the different compartments were circulated for around 30 min to inspect the conductivity changes of different solutions due to concentration diffusion.

Desalination of the 7-fold diluted FME via ED was performed at a constant current of 0.8 A (i.e., current density of 20 mA/cm²) at room temperature. All experiments were performed in batches, taking around 3 h for each run under the same operating conditions.

Samples 1 ml in size were withdrawn from the concentrated solution at intervals of 30 min in order to analyze the ion concentrations and examine the ion transport through the ion exchange membrane. The ion concentrations of 7-fold diluted FME before and after ED were analyzed using an ion chromatograph (IC) to calculate the salt removal, total energy consumption, and average current efficiency of the diluted solution during ED.

4. Analysis Methods

The brix values of the 7-fold diluted solutions before and after ED were measured with a PAL-1 digital refractometer (Atago Co. LTD, Japan), and the ion concentrations of the concentrated and diluted solutions were analyzed with a DX-120 ion chromatograph (Dionex Corporation, America). The 7-fold diluted FME before and after ED was diluted and cleaned using 0.2-μm syringe filters prior to ion chromatograph analysis.

Process parameters, including the membrane stack potential, pH, and electrical conductivity of the concentrated and diluted solutions, were measured by using a digital multimeter, an Orion pH meter and a Cole Palmer conductivity meter, respectively, during the ED process.

Membrane electrical resistance was measured at a frequency of 100 KHz using a clip cell connected to an NF 2353 LCZ meter (NF Electronic Instruments, Japan) in 0.5 M NaCl. The fresh and fouled surfaces of the AFX/CMX membranes were then examined with a field-emission scanning electron microscope (FE-SEM; Hitachi S-4700, Hitachi High Technologies, America) with an energy dispersive X-ray spectrometer (EDX). For characterization of the AFX/CMX membrane functional groups, ATR-FTIR spectroscopy (FT/IR-460 Plus, Jasco, Japan) was used at a measuring range between 650 and 4,000 1/cm[27].

RESULTS AND DISCUSSIONS

1. Fish Meat Extract Desalination via ED

With the ED system operating at a constant current of 0.8 A, the electrical conductivity changes of concentrated and diluted solutions presented in Fig. 2(a) indicate that the conductivity of the diluted solution decreased from 26.4 mS/cm to 4.65 mS/cm and that of the concentrated solution increased from 2.52 mS/cm to 25.5 mS/cm. Moreover, the appearance of the 7-fold diluted fish meat extract (FME) displayed almost no change after ED for 3 h. The brix change of the diluted solution showed that only a little sugar was transported into the concentrated solutions through ion exchange membranes. Moreover, the sugar loss can be decreased by selecting the suitable ion exchange membranes, which was similar to the report [28]. Thus, it could be confirmed that the FME could be effectively desalted by ED, while mostly nutritional ingredients remained in the FME after desalination.
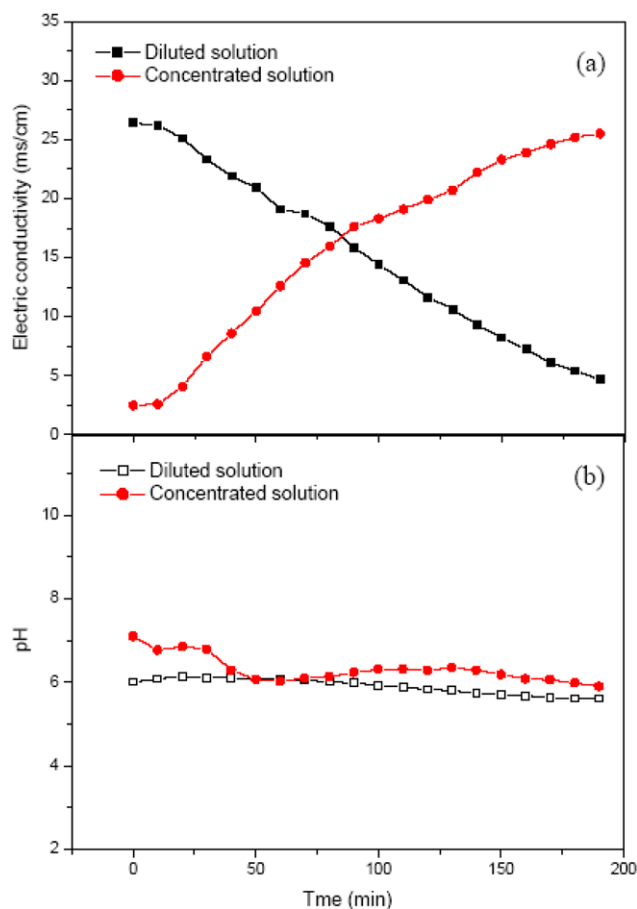

Fig. 2. Changes of (a) electrical conductivity and (b) pH in the diluted and concentrated solutions used in the electrodialysis system.

Fig. 2(b) shows that there was little change in the pH values of the diluted and concentrated solutions; moreover, the pH change of diluted solutions was lower than that of concentrated solutions because of the buffer action of organic components contained in the FME. It could hence be concluded that no water-splitting reaction
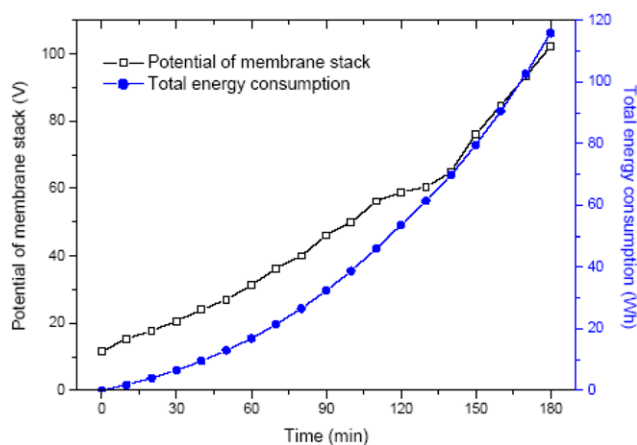

Fig. 3. Changes of the membrane stack potential and total energy consumption of the desalinated 7-fold diluted fish meat extract during electrodialysis.

occurred at the surfaces of AFX/CMX membranes due to concentration polarization. In other words, few H^+ or OH^- ions participated in the ion electromigration, which was favorable for maintaining high current efficiency during the fish meat extract desalination via ED.

The results in Fig. 3 show that the initial potential was about 11.57 V, which then gradually increased during ED at a constant current of 0.8 A. It was found that the increase rate of membrane stack potential slowed down around t=120 min in Fig. 3. It was presumed that a few of H^+ and OH^- ions generated for H_2O dissociation participated in the ion electromigration due to concentration polarization only when the mostly mineral ions were removed [29]. Then the rapid increase of membrane stack potential resulted from the change of solution composition and membrane fouling, and its maximum value reached 102.2 V for about 180 min at the end of experimentation. The total energy consumed (EC_{Total}) of the 7-fold dilution desalination via ED was calculated using [30]

$$EC_{Total} = \int_0^t UIdt \qquad (1)$$

where U is the potential (V), I is the current (A), and t is the time of operation (h). The EC_{Total} for different operational times is shown in Fig. 3. From the figure, the total energy consumed by the 7-fold diluted FME was around 0.116 kWh after ED for 3 h; moreover, the EC_{Total} increased slowly in the initial period of ED and then increased rapidly. It is presumed here that the increase of membrane stack potential resulted from the increase of membrane electrical resistance due to membrane fouling, and the electrical conductivity of diluted solutions decreased at a constant current of 0.8 A, which increased the energy consumption and decreased the current efficiency during the ED desalination of FME [31].

2. Characterization of Membrane Fouling

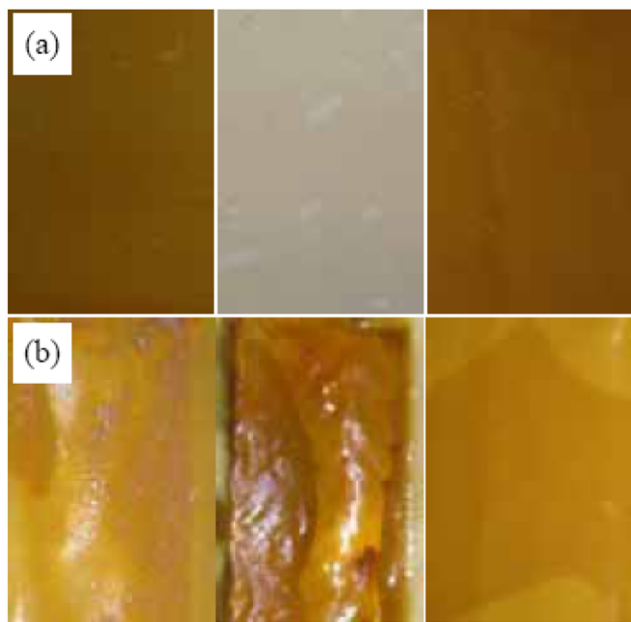Images of the three ion exchange membranes, CMX, AFX and

Fig. 4. Photographs of three ion exchange membranes (left→ right) CMX, AFX, and BP-1: (a) fresh membranes; (b) membranes after electrodialysis.

Table 3. Electrical resistances of ion exchange membranes before/ after electrodialysis

Ion exchange membrane	Membrane electrical resistance (ohm·cm²)	
	Fresh	After electrodialysis
AFX	0.72±0.02	1.09±0.07
CMX	3.38±0.03	4.47±0.1
BP-1	5.87±0.05	6.36±0.06

BP-1, before/after electrodialysis are shown in Fig. 4. Compared to the fresh membranes, membrane fouling can easily be observed on the AFX and CMX surfaces. Here, it is thought that the organic matter contained in FME was adsorbed onto the AFX surface, which was in contact with the diluted FME to form the brown-spotted and colloid deposits, but no mineral fouling appeared on the AEM surface in contact with the concentrate. The result was different from that in the solutions containing $CaCl_2$ using a concentrated solution maintained at a pH value of 7 or 12, reported by Ayala-Bribiesca et al. [32], which meant that the solution compositions could affect the features of membrane fouling.

There were white crystals deposited onto the CMX membrane that was in contact with the concentrated solution and adjacent to the anionic surface of BP-1, though no white crystals were observed on the CMX surface in contact with the concentrated solution and adjacent to the cationic surface of BP-1. OH^- and H^+ ions generated for bipolar membrane water-splitting [33] seemed to, respectively, facilitate and inhibit the formation of white crystals on the CMX surfaces. The results showed that the membrane fouling formation was related to ionic membrane properties and ion migration.

The electrical resistances of three ion exchange membranes before/ after ED are shown in Table 3, which indicates that all membrane resistances increased after ED. Here, membrane fouling was considered to be the main reason for the increase of the AFX/CMX membranes resistance. Compared with CMX, the AFX membrane had a quite low resistance increase due to its properties that inhibit organic fouling. For no obvious membrane fouling appearing on the BP-1 surface, the resistance increase of BP-1 might result from membrane deterioration [34,35] due to the higher applied potential, especially in the latter period of ED, which might be one of the main reasons for the increase in membrane stack potential in the ED system.

The SEM images of AFX/CMX membranes before/after ED are shown in Fig. 5. It was found that a fresh AFX membrane presented a clean surface with reticular lamination (Fig. 5(a)) and that the surface of the fouled AFX was scattered with many spotted foulants (magnified region shown on the right of Fig. 5(a′)), resulting from the adsorption of organic components contained in the FME. Figs. 5(b) and (b′) indicate that other than the fresh CMX surface with its tiny pores, the fouled CMX surface was covered with flaky and flocculated foulants (magnified region shown on the right of Fig. 5(b′)), due to the deposition of inorganic salt from the FME.

Membrane fouling on the AFX and CMX surfaces was further analyzed by EDX to examine the element compositions of the foulants. Figs. 6(a) and (a′) show that the amounts of C and Cl contained in the AFX (Cl-form) membrane had no obvious change after ED, in which the increase of O and the decrease of N might result
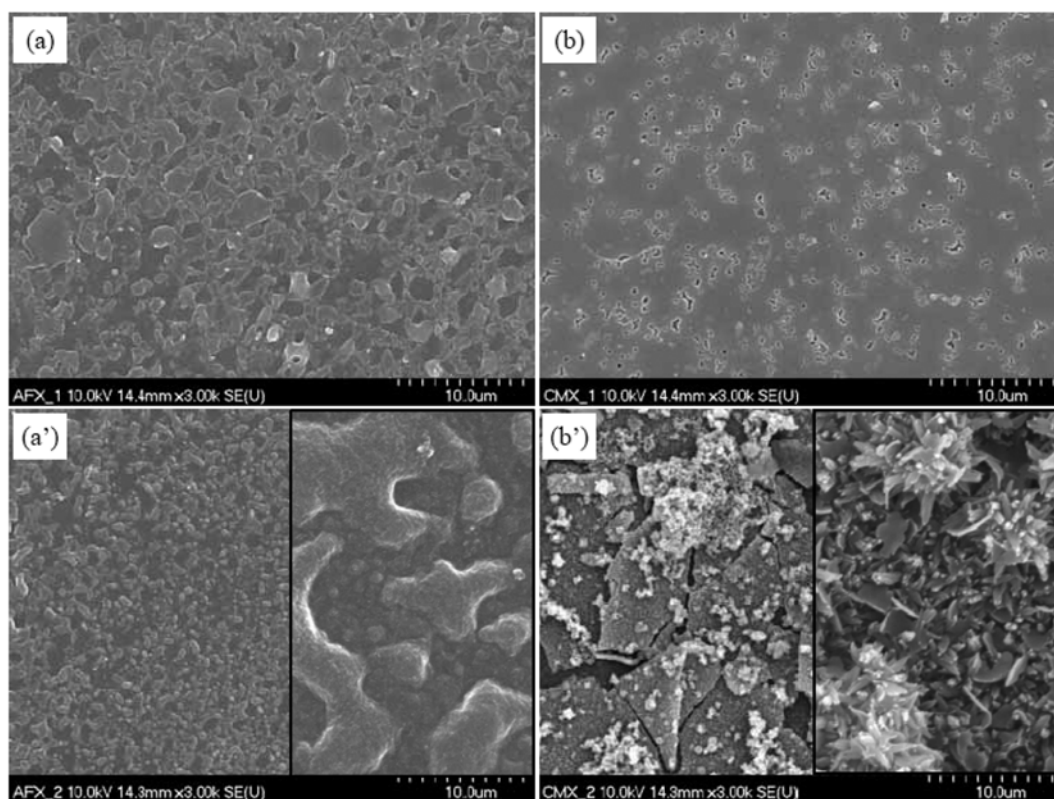
Fig. 5. SEM images of anion and cation exchange membranes before/after electrodialysis: (a, a′) fresh and fouled AFX; (b, b′) fresh and fouled CMX, in which the local magnified parts of the fouled AFX and CMX are shown on the right [(a′ or b′), respectively].

(a) Fresh AFX

Element	Weight %	Atomic %
C K	78.42	83.82
N K	4.54	4.16
O K	13.27	10.65
Cl K	3.77	1.37

(b) Fresh CMX

Element	Weight %	Atomic %
C K	74.05	83.06
O K	13.75	11.58
Na K	3.11	1.82
S K	2.01	0.84
Cl K	7.08	2.69

(a′) AFX after electrodialysis

Element	Weight %	Atomic %
C K	78.60	84.39
O K	17.67	14.24
S K	0.33	0.13
Cl K	3.40	1.24

(b′) CMX after electrodialysis

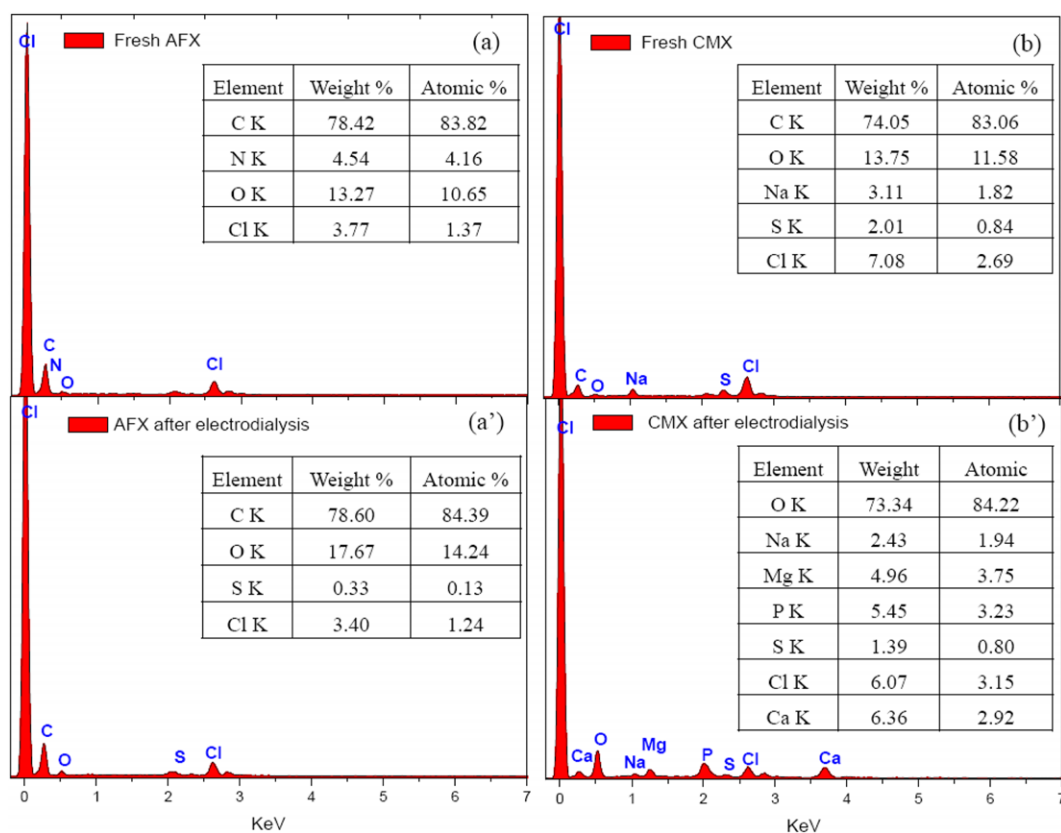| Element	Weight	Atomic
O K	73.34	84.22
Na K	2.43	1.94
Mg K	4.96	3.75
P K	5.45	3.23
S K	1.39	0.80
Cl K	6.07	3.15
Ca K	6.36	2.92

Fig. 6. EDX spectra and element compositions of anion and cation exchange membranes before/after electrodialysis: (a) fresh and fouled AFX; (b) fresh and fouled CMX.
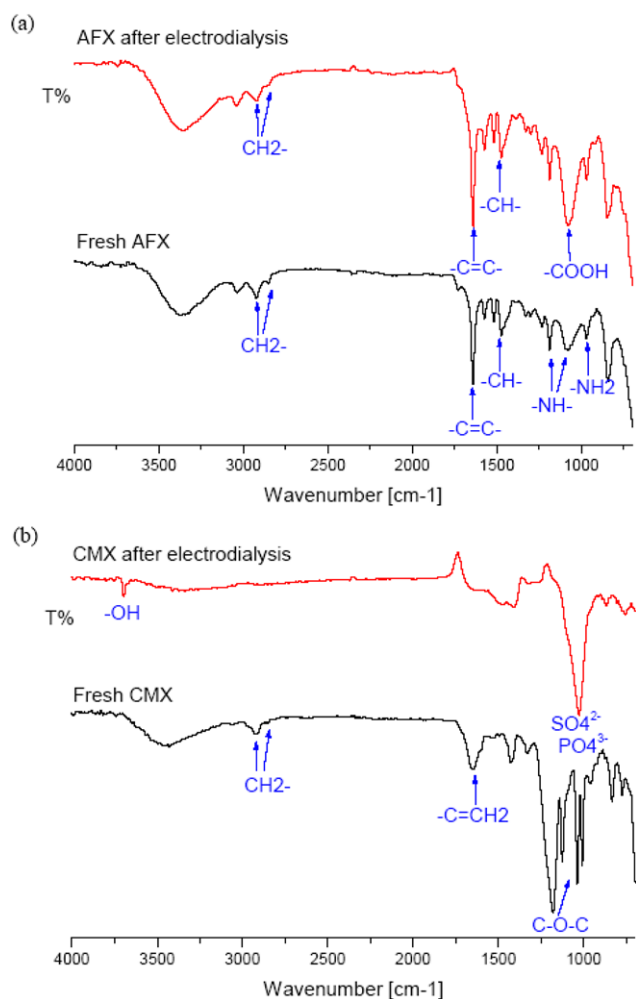
Fig. 7. ATR-FTIR analyses of AFX/CMX membranes before/after electrodialysis.

from the adsorption of organic components. For the charged components, such as proteins, peptides, et al., might enter into the membrane inner due to the driving force of electrical field [36]. In addition, the appearance of S might be due to the partial substitution of chloride (Cl^-) by sulfate (SO_4^{2-}) in the fouled AFX. It can be seen in Figs. 6(b) and (b′) that the elements C, O, Na, S, and Cl were contained in the surface of a fresh CMX (Na-form) membrane, whereas the appearance of Mg, Ca, and P in addition to the significant increase of O in the fouled CMX surface resulted likely from the salt deposition of Ca^{2+} and Mg^{2+} in the FME and the overlap of inorganic foulant could lead to the disappearance of carbon at the cationic membrane.

The transmittance spectra of the fresh and fouled AFX/CMX mem-

branes were measured in the wave number range between 650 and 4,000 1/cm using ATR-FTIR in order to investigate the functional groups of the membranes. For AFX, Fig. 7(a) shows that functional groups remained after ED, including $-CH_2-$, C=O and -NH- bonds; i.e., the functional groups were not significantly changed after the desalination process due to its property against organic fouling. Conversely, the foulant dramatically changed the functional groups in CMX after the desalination process. The new transmittance peak in Fig. 7(b) indicates that there was some sulfate deposited on the membrane surface, which might significantly affect the physical and chemical property of the CMX membrane [37]. The results from FTIR analyses were coincident with that of SEM/EDX analyses.

The characterization of membrane fouling showed that the membrane fouling resulted from the adsorption and deposition of organic matter and inorganic salts from the FME, respectively, on the surfaces of the AFX/CMX membranes during the process. This fouling is an important reason for restricting the ED application in desalinating FME; further research is needed to mitigate membrane fouling during the ED process.

3. Ion Concentration Analysis and Current Efficiency

The concentrations of ions contained in the diluted and concentrated solutions were analyzed via IC. The results in Table 4 show that the salts of the FME were composed of cations such as Na^+, NH_4^+, K^+, Mg^{2+}, and Ca^{2+}, and the anions Cl^-, PO_4^{3-} and SO_4^{2-} (organic ions are not shown), in which the main components of Na^+ and Cl^- ions could be effectively removed by ED. The salt removal of the 7-fold diluted FME was about 82.3% according to the concentration difference of all the ion concentrations before and after ED. In addition, the small amount of Mg^{2+}, Ca^{2+}, PO_4^{3-}, and SO_4^{2-} contained in the diluted FME could cause the serious CMX membrane fouling, thereby suggesting that membrane fouling of ED process might be mitigated if these components were selectively removed by some methods such as ion exchange [38].

Fig. 8 presents the concentration changes of ions contained in the concentrated solution with the initial 0.01 M Na_2SO_4, which shows that the main components including Na^+, K^+, and Cl^- in the 7-fold diluted FME were transported into concentrated compartments through ion exchange membranes, further confirming the feasibility of using ED to desalinate FME.

The average current efficiency (ACE) of desalinating the 7-fold diluted FME via ED was calculated using [30]

$$ACE = \frac{\sum zF\Delta n_i}{60N\int_0^t Idt} \times 100\% \tag{2}$$

where z is the ionic valence, F is the Faraday constant (96,500 C/mol), Δn is the amount of ion removed (mol), N is the number of membrane pairs, I is the current (A), t the operation time (s). Ac-

Table 4. Ion concentrations of 7-fold diluted fish meat extract before/after electrodialysis

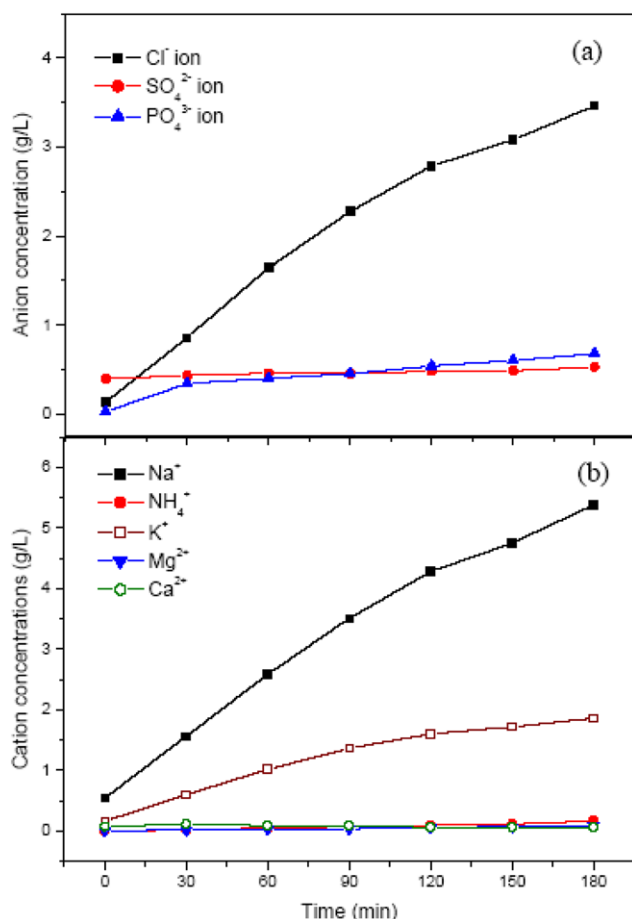| 7-Fold diluted fish meat extract	Ion concentration (g/L)							
	Cation					Anion		
	Na^+	NH_4^+	K^+	Mg^{2+}	Ca^{2+}	Cl^-	SO_4^{2-}	PO_4^{3-}
Before electrodialysis	5.61	0.97	1.93	0.19	0.10	3.83	0.21	0.98
After electrodialysis	0.70	0.46	0.18	0.06	0.06	0.35	0.12	0.51

Fig. 8. Concentration changes of anions and cations contained in the concentrated solution during electrodialysis.

resulted from the organic and inorganic foulants contained in the FME deposit, respectively, on the AFX/CMX membrane surfaces, which was proved further by SEM-EDX and FTIR analyses. Ion chromatograph analyses indicated the mineral ions contained in the FME were composed of the cations Na^+, NH_4^+, K^+, Mg^{2+} and Ca^{2+}, and the anions Cl^-, PO_4^{3-} and SO_4^{2-}, which could be effectively removed by ED with the salt removal rate of around 82.3%. The total energy consumption was about 0.116 kWh for 3 h of the ED process with an average current efficiency of around 77.9% in this work. The feasibility of FME desalination via ED was proved and the further studies should be aimed at mitigating membrane fouling and optimizing operation conditions to increase current efficiency and decrease the loss of nutritional ingredients.

ACKNOWLEDGEMENT

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cording to the ion concentration change of the diluted solution, the average current efficiency of the diluted FME desalination via ED was around 77.9% at a constant current of 0.8 A for 3 h, in which over 80% of the mineral ions contained in the diluted solution were removed. The value of average current efficiency for FME desalination via conventional ED was markedly higher than that of the recovery of glutamic acid from isoelectric supernatant with bipolar membrane ED [30]. Further work needs to be performed by optimizing operation conditions to increase the salt removal rate and current efficiency.

The feasibility of the 7-fold diluted FME desalination via ED was proven from the batch experiments. However, more research related to the mitigation of membrane fouling still needs to be performed for facilitating the technique application. Further studies will also involve process scale-up, economic evaluation and process optimization.

CONCLUSION

The changes of the key parameters such as electrical conductivity, brix, and pH indicated that the 7-fold diluted fish meat extract (FME) could be effectively desalted via electrodialysis (ED) process, with mostly nutritional ingredients remaining during the FME desalination via ED. The increase of membrane electrical resistance

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