



Analysis of anion exchange membrane fouling mechanism caused by anion polyacrylamide in electro dialysis



Haicheng Guo^a, Lan Xiao^a, Shuili Yu^{a,*}, Haijun Yang^b, Jun Hu^b, Guicai Liu^a, Yulin Tang^a

^a State Key Laboratory of Pollution Control and Resource Reuse, College of Environmental Science & Engineering, Tongji University, Shanghai 200092, PR China

^b Key Laboratory of Interfacial Physics and Technology, Shanghai Institute of Applied Physics, Chinese Academy of Sciences, P.O. Box 800-204, Shanghai 201800, PR China

HIGHLIGHTS

- Mechanism of AEM fouling by APAM during ED is thoroughly analyzed.
- Voltage–time curves during ED processes are investigated.
- A gel layer is formed on diluate side of the AEM after fouling.
- AFM is utilized to investigate the interaction forces.
- Electrostatic interaction dominates the interactions between membranes and APAM.

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ABSTRACT

Tertiary oil extraction technologies, especially polymer flooding, have been successfully used to enhance oil recovery, but the enormous amount of produced oily wastewater is urgent to be treated and reused. Electro dialysis (ED) is an important process in treating oily wastewater for reinjection. The aim of this work was to reveal the fouling mechanism of an anion exchange membrane (AEM) caused by anion polyacrylamide (APAM) in electro dialysis. Fouling experiments were carried out with different APAM concentrations at different current densities. Contact angle, electrical resistance and ion exchange capacity for the AEMs were measured to verify the fouling processes. The highest fouling phenomenon was observed with a higher APAM concentration at the current density close to the limiting value, which was indicated by evident increase of hydrophobicity and electrical resistance of the AEM. Morphology analysis by scanning electron microscopy (SEM) showed that a gel layer was formed on the diluate side of the fouled AEM and the ATR–FTIR spectra verified the presence of APAM on this side. Force–distance curves measured by atomic force microscopy (AFM) clearly confirmed that electrostatic interaction dominated the interactions between APAM molecules and ion exchange membranes.

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1. Introduction

With the diminution of the easily recoverable oil and an increase in the demand for oil and its derived products in the world, the conventional methods of oil exploitation become unable to fit for the increase in demand. Meanwhile, much oil which has lost their drive after primary and secondary recovery methods still remains in the reservoirs [1,2]. Currently, enhanced oil recovery (EOR) techniques such as miscible displacement and chemical flooding, have proven to be effective to recover the remaining oil left in the reservoirs [3]. Among different kinds of the EOR techniques, polymer flooding has been successfully applied, especially in China [4,5]. The purpose of the water soluble polymers in this application is to enhance the rheological properties of the displacing fluid. Partially hydrolyzed anion polyacrylamide (APAM), one of the

most commonly used polymers in polymer flooding, can efficiently increase the oil recovery rate once injected into the flooding water.

However, with the largest polymer flooding project in the world, Daqing oil field generates about 3×10^8 tonnes oily wastewater per year [6], which contains APAM, oil, suspended solid and high concentration salts. Those effluents, if not appropriately handled, will cause a great deal of damage to the environment. Considering the deficient water resources and a great demand for fresh water to prepare the polymer solution, it becomes reasonable to reuse the enormous quantity of oily wastewater. To reach the reinjection criteria, a series of treatments such as coagulation, sedimentation and ultrafiltration (UF) are employed. With these pretreatment processes, most of the APAM, oil and suspended solid in the oily wastewater are removed. However, the high salt concentration in treated water after UF can reduce the thickening capability of the ionic water soluble polymer which will affect the oil recovery rate [7]. Therefore, the mineralization of the treated water must be lowered to a permissible level before reinjection. To solve this

* Corresponding author. Tel./fax: +86 21 65982708.
E-mail address: ysl@tongji.edu.cn (S. Yu).

problem, electro dialysis has been applied and it proves to be proper and effective.

Nevertheless, fouling remains a main obstacle in all membrane separation processes. The residual APAM in treated water may foul the ion exchange membranes in ED, thus limiting its application. Analysis of mechanisms for membrane fouling may give an insight to control fouling processes. There are some literatures focusing on the fouling by APAM on UF membranes [8–10], as well as the fouling on ion exchange membranes by other foulants [11–15]. Anion exchange membranes (AEMs) are apt to be fouled by negatively charged organics under electric field. Membrane fouling during ED depends on the type of the functional groups, the structure of membrane polymeric matrix and the properties of foulants. Fouling of inorganic salts on membranes generally occurs in the form of direct surface crystallization and/or precipitation [16,17], which is reversible. Whereas, organic foulants with fixed charged groups and aromatic rings [18] can cause irreversible fouling due to the combined bonding between benzene ring structures of the organic material and the ion exchange resin polymer. Park et al. [19] have investigated AEM fouling of bovine serum albumin (BSA) by the electrical impedance spectroscopic study and found that the negatively charged loose fouling layer changed to the dense deposited BSA on the surface of the membrane at a higher current density. Lee et al. [20,21] have pointed out that zeta potentials of the foulant and membranes play a key role in predicting the fouling tendencies. Unfortunately, fouling of APAM on ion exchange membranes was scarcely studied and the mechanism of ion exchange membrane fouling was rarely analyzed thoroughly in many previous researches.

This research investigated the fouling mechanism of heterogeneous anion exchange membranes caused by APAM in ED processes. Voltage–time curves for the fouling processes during ED were thoroughly investigated to provide information on ongoing fouling phenomena. Meanwhile, various evaluation parameters such as limiting current density (LCD), hydrophobicity, electrical resistance, and ion exchange capacity (IEC) were determined to characterize the changes of the membranes after fouling. SEM images and ATR–FTIR spectra were also examined to verify the fouling phenomena. Specially, force–distance curves were measured using atomic force microscopy (AFM) to give direct insight into the interaction forces between the foulant and membranes. This work can be a provision of the theoretic support for membrane fouling control in treating oily wastewater by ED.

2. Experimental

2.1. Characterization of anion polyacrylamide

APAM is a hydrophilic compound with a very high solubility in water [9]. It can tolerate the high mechanical forces during polymer flooding process [22]. The APAM used in this study is industrial grade (Wenxian, China) and its main properties are summarized in Table 1. The average molecular weight and molecular weight distribution of APAM were determined by Gel Chromatography Lc-10ADVP (Shimadzu, Japan). The

Table 1
Physicochemical properties of APAM.

Parameters	Values
Source	Wenxian, China
Chemical name	Anion polyacrylamide
Chemical structure	
Molecular weight (g/mol)	500,000
Purity (%)	≥99.8

As reported by manufacturer.

concentration of APAM used for Gel Chromatography was 10 mg/L. The zeta potentials of APAM as a function of pH were determined by electrophoretic mobility using Zetasizer Nano ZS90 (Malvern, UK) in 0.01 M KCl. From determined electrophoretic mobility, the zeta potential could be calculated using the Smoluchowski equation [12]. The hydrodynamic diameter of APAM at different concentrations was also determined by Zetasizer Nano ZS90.

2.2. Characterization of anion exchange membranes

Heterogeneous cation exchange membranes (CEMs) 3361 and anion exchange membranes 3362 (Hedun, China) were purchased and used in this study. These membranes are composites fabricated from powder of ion exchange resins and polyethylene, and they are reinforced with nylon [23]. The functional groups of anion and cation exchange membranes are quaternary ammonium and sulfonic groups, respectively. The detailed properties of the two kinds of ion exchange membranes were listed in Table 2. Properties of the AEM characterized in this study were the hydrophobicity, electrical resistance and ion exchange capacity. The contact angles of deionized water on the surface of dried membranes were measured by JC2000D contact angle meter (Zhongchen, China) using a sessile drop technique. The electrical resistances in 0.5 M NaCl solution at 25 °C were measured by LCR Meter AT810A (Applent, China) set at 1 kHz and expressed as $\Omega \text{ cm}^2$ [24]. The ion exchange capacities of the AEMs were determined using the Mohr method [25]. The membranes were immersed in 0.5 M Na_2SO_4 solution for 24 h in order to convert the AEMs in chloride form into sulfate form completely. Then the number of moles of chloride ions released from the membranes was estimated from the amount of 0.1 M AgNO_3 consumed by precipitation titration. The IEC was expressed as meq./g-dried membrane. IR spectra were obtained by the method ATR–FTIR spectroscopy using Nicolet 5700 (Nicolet, USA). A spectrum, collected as the average of 32 scans with a resolution of 4 cm^{-1} , was recorded from 4000 to 400 cm^{-1} . All spectra were collected with Omnic 8.0 software. The morphologies of the AEMs before and after fouling were examined by a scanning electron microscope XL30FEG (Philips, Netherland). The cross sections of membranes were prepared by freeze fracturing the samples in liquid nitrogen, to produce a clean brittle fracture. Membranes for ATR–FTIR and SEM were dried at 38 °C for 24 h before measurements.

2.3. Electro dialysis processes in the presence and absence of APAM

2.3.1. Six-compartment electro dialysis stack

A six-compartment stack made of Plexiglas was employed to carry out the electro dialysis experiments. The arrangement of the stack and its function for the ion exchange membranes are schematically shown in Fig. 1. The effective membrane area of the electro dialysis cell was 42 cm^2 . For the electrode solution, 0.8 L of 0.04 M Na_2SO_4 was used. The second and fifth compartments were filled with 0.5 L of 0.04 M Na_2SO_4 solution. The third and fourth compartments were filled with 0.5 L of feed solutions (0.05 M NaCl solution or 0.05 M NaCl solution containing different concentrations of APAM). The solutions were pumped through the compartments continuously from reservoir vessels using peristaltic pump BT100-L (LongerPump, China). The electrode solution, the solution in compartments 2 and 5, and the feed solution in

Table 2
Properties of ion exchange membranes used in this study.

Properties	AEM	CEM
Thickness (mm)	0.42 ± 0.04	0.42 ± 0.04
Water content (%)	30 ~ 45	35 ~ 50
Electrical resistance ($\Omega \cdot \text{cm}^2$)	≤ 12	≤ 11
Exchange capacity ($\text{mol} \cdot \text{kg}^{-1}$)	≥ 1.8	≥ 2.0
Transport number	≥ 0.89	≥ 0.90

As reported by manufacturer.

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