



## Influence of ion interaction on lead removal by a polyamide nanofiltration membrane



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### HIGHLIGHTS

- Increasing lead ion concentration resulted in a higher retention of this ion at a constant pressure.
- Increasing anion valences of the interfering ions resulted in a higher lead ion rejection and a lower permeate flux.
- Decreasing or increasing the pH of the lead ion solutions caused a higher lead ion rejection.
- Altering the pH of the lead ion solutions from their natural pH, decreased the permeate flux.
- The presence of monovalent cations didn't reduce lead ion rejection, significantly.

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### ABSTRACT

Retention of lead(II) ions on a polyamide nanofiltration membrane was investigated. Effects of different factors including operating pressure, lead ion concentration, anion nature, pH and composition of feed on the lead ion rejection were studied. The solutions used consisted of Pb(NO<sub>3</sub>)<sub>2</sub>, PbCl<sub>2</sub> and PbSO<sub>4</sub> in the single-salt solution system and Pb(NO<sub>3</sub>)<sub>2</sub>, Cu(NO<sub>3</sub>)<sub>2</sub>, Zn(NO<sub>3</sub>)<sub>2</sub>, Cd(NO<sub>3</sub>)<sub>2</sub>, NaNO<sub>3</sub> and NH<sub>4</sub>NO<sub>3</sub> in the binary-salt solution system. The influence of divalent and monovalent cations including cadmium, copper, zinc, sodium and ammonium on the rejection of lead ion was examined. The transmembrane pressure and lead ion concentration varied between 10 and 40 bar and 20 and 400 mg Pb<sup>2+</sup>/L, respectively. It was observed that increasing the pressure and initial feed concentration resulted in a higher lead ion rejection. By replacing NO<sub>3</sub><sup>-</sup> with Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup>, the rejection of lead ion increased about 2% and 9%, respectively. Applying anions with higher valences resulted in a higher lead ion rejection and lower permeate flux. Maximum permeate flux and minimum lead ion rejection was observed at pH 5.6. In the binary salt solutions, the rejection of lead did not change significantly in the presence of monovalent cations. However, the presence of divalent cations caused a substantial reduction in the lead ion rejection.

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### 1. Introduction

The presence of heavy metals in wastewaters causes many problems due to their persistence in the environment. There is also a high risk of heavy metal accumulation in the body tissues of living organisms due to their high solubility in the aquatic environments [1]. Industries that generate heavy metals such as Pb, Cd, Zn, Cu, Ni and Cr are considered as the most hazardous industries [2]. Among the heavy metals, lead is a very dangerous agent due to its side effects on the human body even at concentrations as low as 0.01–5 mg/L [3]. To date, numerous techniques have been considered for development of efficient and inexpensive treatment methods to improve the quality of effluents. Heavy metal removal can be achieved by conventional processes such as chemical

precipitation [4–7], solvent extraction [8], ion exchange [9–11], adsorption [12–14] and metal oxides [15]. Despite being efficient and cheap, these methods are suffering from substantial disadvantages such as incomplete removal of heavy metals, high energy consumption, slow removal rates and generation of toxic sludge [16]. On the path to tackle these difficulties and find less expensive and more efficient methods, membrane separation processes have attracted a high attention. Nanofiltration (NF) technology has been used to treat lead containing wastewaters. In this method, the rejection of lead ion can be high enough to achieve acceptable quality for the filtered water [17–20]. Nanofiltration is a membrane filtration process that stands between ultrafiltration and reverse osmosis in which the separation characteristics are based on the sieving effect and membrane surface charge, though the NF membranes have been modified in many commercial applications [21]. Nanofiltration membranes can remove divalent ions and low molecular weight organic materials [22]. Three phenomena contribute to the ion transport through

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NF membranes that include size exclusion, charge effect (based on Donnan exclusion and dielectric exclusion), and different diffusivity and solubility of existing ions [23]. It is known that various parameters such as feed solution concentration, composition and pH value are effective on the interactions between solutes of the ionic solution and solute–membrane interactions [24,25]. However, in the real industrial wastewaters, there is a mixture of several heavy metals in which the interfering cations can influence on the desired cation removal. Nevertheless, there are a few studies dealing with lead ion removal in the mixtures consisted of interfering cations [19,20,26]. To the best of our knowledge, there is only one study about the effect of anions nature on the lead ion rejection and permeate flux that is related to lead ion removal in the presence of a single salt [27].

In this work, the retention of lead ion of a single salt solution containing  $\text{Pb}(\text{NO}_3)_2$ ,  $\text{PbCl}_2$  or  $\text{PbSO}_4$  was investigated to study the influence of anion nature (monovalent and divalent) on the lead ion removal. Besides, the effects of different conditions i.e. pressure of 10–40 bar, pH of 3–7 and lead ion concentration of 20–400 mg/L were examined on the rejection of lead ions. Then, the binary-salt solutions consisted of lead ion and another cations of  $\text{Zn}(\text{NO}_3)_2$ ,  $\text{Cu}(\text{NO}_3)_2$ ,  $\text{Cd}(\text{NO}_3)_2$ ,  $\text{NaNO}_3$  or  $\text{NH}_4\text{NO}_3$  were examined in order to study the effect of interfering monovalent and divalent cations on the removal efficiency of lead ion by nanofiltration process.

## 2. Experimental

### 2.1. Membrane

A NE 4040-90 membrane manufactured by CSM, South Korea was used in this study. The used membrane was a thin-film polyamide composite membrane in spiral wound element configuration. The module was opened, and the flat sheet membrane was used in our experiments. As reported by the manufacturer, NaCl and  $\text{MgSO}_4$  (2000 ppm) rejection of the membrane was 85–95% and 97%, respectively.

### 2.2. Materials

Chemicals used in this work were all of analytical grade. All the metal solutions were prepared by dissolving enough mass of each material in a high purity demineralized water (conductivity  $< 1 \mu\text{S}/\text{cm}$  and pH  $6.3 \pm 0.1$ ). Lead nitrate  $\text{Pb}(\text{NO}_3)_2$ , lead chloride  $\text{PbCl}_2$ , lead sulfate  $\text{PbSO}_4$ , copper nitrate  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ , zinc nitrate  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , cadmium nitrate  $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ , sodium nitrate  $\text{NaNO}_3$ , ammonium nitrate  $\text{NH}_4\text{NO}_3$ , sodium hydroxide, nitric acid, hydrochloric acid and sulfuric acid were all obtained from Merck Chemicals, Germany.

### 2.3. Analytical methods

Elemental analysis of lead was conducted for the feed and permeate solutions by measuring the conductivity of these solutions at  $25^\circ\text{C}$  using a conductometer LF 330/340 (WTW, Germany), while the concentration of the cations in the feed and permeate of binary-salt solutions was determined using a Varian AA240FS Atomic Adsorption Spectrometer (US).

### 2.4. Membrane test unit

All experiments were performed with a cross-flow filtration unit equipped with a flat-sheet cell as presented schematically in Fig. 1. The effective filtration area of the filtration cell was  $36 \text{ cm}^2$ . The temperature of feed solution was maintained at the constant value of  $25 \pm 2^\circ\text{C}$  using a heat exchanger. The transmembrane pressure was varied between 10 and 40 bar by changing the feed pressure. The solution was pumped from the feed tank into the membrane cell by means of a volumetric pump. The membrane was immersed overnight in a mixture of ethanol/water solution with equal volumetric amount before using in any

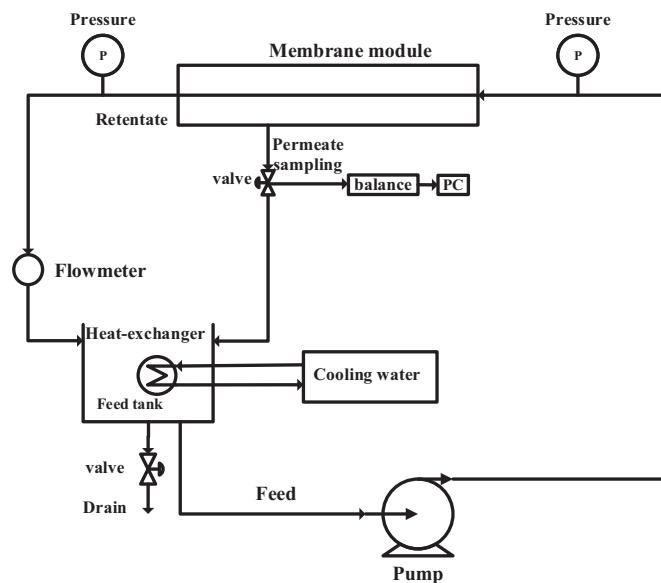


Fig. 1. Schematic diagram of nanofiltration experimental setup.

experimental work. Each membrane was washed by circulating demineralized water for at least one hour at the pressure of 30 bar followed by compacting for one hour at the maximum pressure used in this study (40 bar) to avoid any compression effect. Retentate and permeate were both returned to the feed tank. To measure the permeate flux, the amount of the collected solution passed the membrane was determined using an intelligent gravimetric analyzer connected to a computer. When the concentrations of permeate and feed reached a constant value, the operation reached the steady-state condition. To confirm this condition, the conductivity of both solutions was measured.

### 2.5. Electro-kinetic (zeta) potential measurement

The separation mechanism in nanofiltration membranes is usually described in terms of charge and sieve effect [28]. Therefore, knowledge about the surface charge is very useful in explaining the test results. Information about the charge of membrane surface provided by membrane manufacturer is rare. Determination of electrokinetic potential or zeta potential is a very appropriate method to measure the charge of membrane surface and determine the electrical properties of the membrane. Zeta potential of the membranes was determined by streaming potential method along the membrane surface using an EKA Electro Kinetic Analyzer instrument (Anton Paar, Austria). In the streaming potential method, movement of the electrolyte solution through a capillary system creates a streaming potential where its relation with the zeta potential of the capillaries is given by Smoluchowski–Helmholtz approach [29–31]:

$$\zeta = \frac{dU}{dp} \frac{\eta}{\varepsilon_0 \cdot \varepsilon} \frac{L}{Q \cdot R} \quad (1)$$

where  $\zeta$  is the zeta potential,  $dU/dp$  is the slope of streaming potential versus pressure,  $\eta$  is the electrolyte viscosity,  $\varepsilon_0$  is the permittivity,  $\varepsilon$  is the dielectric constant of the electrolyte,  $L$  is the length of the capillary system,  $Q$  is the cross-sectional area of the capillary system and  $R$  is the AC resistance of the cell using an electrolyte solution. According to Fairbrother and Mastin approach [32], for electrolyte concentrations greater than  $10^{-3} \text{ M}$ , the ratio  $L/Q \cdot R$  in Eq. (1) can be replaced by  $K_B$  that is the specific electrical conductivity of the electrolyte solution outside the capillary system.

Zeta potential measurements were conducted using a clamping cell. Schematic of the clamping cell and the geometric parameters defining a

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