



Rejection of ammonium and nitrate from sodium chloride solutions by nanofiltration: Effect of dominant-salt concentration on the trace-ion rejection

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HIGHLIGHTS

- Rejection of NH_4^+ and NO_3^- ions by a nanofiltration (NF270) membrane was studied.
- Dominant NaCl concentration effect on the trace ions rejection was evaluated.
- The model was used to obtain membrane ion permeance from the experimental data.
- If dominant salt concentration increased, dominant and trace ions rejection decreased.
- When dominant NaCl concentration increased, membrane permeances to ions increased.

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ABSTRACT

In this work, the rejection of two ions associated with fertilizer pollution of ground water (NH_4^+ and NO_3^-) by a polyamide nanofiltration (NF270) membrane was studied. Sodium chloride was selected as the dominant salt and the effect of its concentration on the NH_4^+ and NO_3^- rejections was evaluated. Filtration experiments were carried out in a cross-flow setup with a rectangular spacer-filled feed channel. The solution-diffusion-electro-migration-film model (SDEFM), previously developed to model the pressure-driven trans-membrane transfer of electrolyte mixtures consisting of a single dominant salt and (any number of) trace ions, was used to obtain membrane ion permeance from the experimental data. In this way, the membrane permeances with respect to single ions (Na^+ , Cl^- , NH_4^+ , NO_3^-) were obtained for various concentrations of both dominant (0.05–0.3 M NaCl) and trace salts (0.001–0.003 M $\text{NH}_4\text{Cl}/\text{NaNO}_3$). It was observed that when the dominant salt concentration increased, the rejections of dominant salt and trace ions decreased, whereas the membrane permeances to ions increased. This information can be useful for developing design tools for nanofiltration of mixed electrolyte solutions in water-treatment applications.

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1. Introduction

Coastal zones are typically densely populated areas as they generally present the best conditions for productivity. However, these regions face many hydrological problems like flooding wave surge and drinking water scarcity due to the problem of salt water intrusion. In the case of areas with intensive agriculture the overdosing of mineral and organic fertilizers deteriorates groundwater quality affected potentially by NaCl via the sea-water intrusion in

combination with the incorporation of N-based ions (e.g. ammonium and nitrate). Groundwater treatments often use ion exchange or membrane-based processes (e.g. reverse osmosis, nanofiltration, electro dialysis) to reduce salinity.

Most commercially available nanofiltration (NF) membranes have a thin-film composite structure, consisting of an ultrathin top polyamide active layer (~50–200 nm), an intermediate polysulfone support (~50 μm) and a backing of nonwoven polyester fibers (~100 μm). Within the scope of the solution-diffusion-electromigration model (SDEFM) it is postulated that the ability of ions to diffuse through this active layer eventually controls the ion rejection. This transport may strongly depend on the ion properties

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(charge and size). Thus, comparing how ions with different properties and at different concentrations interact with the active layers can improve our understanding of the mechanisms controlling ions transport and rejection by NF membranes. NaCl rejection by NF membranes has been widely studied previously [1–5]. Less effort has been devoted to NO_3^- and NH_4^+ rejection studies. Su et al. [1] studied the rejection in mixtures of Na^+ , Ca^{2+} , Cl^- and NO_3^- for a concentration range 0.01–0.04 M (at pH 6.5). Paugam et al. [6] conducted experiments with NaNO_3 and NH_4NO_3 , using a spiral-wound NF membrane module. In the studied concentration range (from $0.8 \cdot 10^{-3}$ to $3 \cdot 10^{-3}$ M at pH 5.8) the removal of NO_3^- decreased when the feed salt concentration increased and this effect was attributed to the increased screening of membrane surface charge.

Despite the wide use of NF worldwide, even at industrial scale, there are still difficulties in establishing its predictive model. This is due to the complexity of mechanisms of solvent and solute transport through NF membranes. As reviewed recently by Wang et al. various NF models still coexist [7]. Basically, some models [8–11] use a nanopore assumption for the membrane structure to describe the behavior of ions inside the membrane, while others [12–15] postulate that there are no permanent nanopores inside the membrane. In any case, the model capability to predict the ions rejections from complex mixtures has been shown to be rather limited. At the same time, the use of reasonably justified simplified models can be of significant practical value. Thus for instance, the SDEFM can describe the transport of electrolyte mixtures through NF membranes [16,17]. This model assumes that there are no permanent pores in the membrane active layer but there is a free volume instead. SDEFM describes the trans-membrane ion transport taking into account diffusion and electro-migration within the active layer as well as external concentration polarization [16]. SDEFM can describe and fit the experimental rejection curves of both dominant salt and trace ions. From this fitting, the membrane permeances towards each ion can be estimated.

Membrane permeances with respect to cations and anions of the dominant salt are probably different; this means that one of the ions is transported through the membrane easier than the other. The differences between the membrane permeances with respect to cations and anions of dominant salt always lead to electric fields that arise to keep to ion transfer stoichiometric (and the electric current zero). These electric fields influence the transport of trace ions and negative rejections may occur [18].

Generally, ionic permeances are functions of feed composition and may also change across the membrane due to the ion rejection. To take this into account, Bason et al. [15] postulated a procedure to model rejection data by using two general phenomenological coefficients: the concentration-dependent salt permeance and the Péclet coefficient. Recently, taking as base that constant permeances can be used as first approximation especially in mixtures where anything more elaborated would be hopelessly complex, Fridman-Bishop [19] analyzed permeance values accuracy to model seawater rejection and the potential uncertainties of parameter estimation and the importance of permeance to ions compositional dependence.

The model used in this work is an extension of the classical solution-diffusion model, which includes electrolyte mixtures. The majority of reported studies in which ion rejection in electrolyte mixtures were studied used numerical approaches and no analytical solutions were obtained. In a previous publication, Yaroshchuk et al. [16] studied the case of a dominant salt and trace ions by means of the model and an analytical solution was obtained. The analytical algebraic solution of transport equations enables rapid exploration of trends in the behavior of membrane systems [20]. In the SDEFM rapid calculations of ion rejections at

different transmembrane fluxes through a NF membrane are possible by only adjusting the single-ion permeances as parameters of differential equations. Then, it can be said that the SDEFM is a good advanced engineering model, because it employs only a limited number of adjustable parameters obtainable from a well-defined set of experiments and at the same time it takes into account the principal physico-chemical phenomena.

In this study, the rejections of sodium, chloride, nitrate and ammonium ions by a commercial NF membrane were studied experimentally. Solutions of NaCl (as dominant salt) of various concentrations were mixed with NH_4Cl and NaNO_3 as traces in order to study the effect of dominant salt concentration on the trace ions rejection (NH_4^+ and NO_3^-). Besides, the rejection data were modelled by means of the SDEFM to describe the ion rejection behavior as a function of trans-membrane flux. As a result, the membrane permeances to each ion (Na^+ , Cl^- , NH_4^+ and NO_3^-) were also estimated. The dependences of salt and ion permeances on the feed solution composition were analyzed.

Trace ammonium and nitrate rejections from dominant sodium chloride solutions have been modelled by the SDEFM for the first time. These ions usually appear as trace ions in salty solutions and are of interest because of environmental concerns. In this work, sodium chloride was used as the main salt and its concentration effect on the rejection of trace ions has been studied and modelled obtaining good fits for all of them: dominant salt as well as trace ions. The experimental data were successfully modelled and ion permeances were determined as functions of the dominant-salt concentration for the first time.

2. Materials and methods

2.1. Experimental set-up description and operation

A flat-sheet cross-flow test cell with a NF-270 membrane, supplied by Dow Chemical, was used to carry out the experimental part of this work. The membrane was a polyamide thin-film composite with 0.014 m^2 of effective area.

The experimental setup was described previously [21,22]. The feed solution was placed in a refrigerated tank and pumped into the membrane test cell. After the NF process, both permeate and concentrate streams were recirculated to the feed tank in order to keep constant the feed solution composition. The concentrate stream passed through a cartridge filter before entering the feed tank again to keep this stream free of particulate matter. The permeate stream was monitored in terms of pH, conductivity and temperature during the experimentation. Permeate samples were periodically collected by using a three way valve, and the ion analysis was done by chromatography on the completion of experiments. Inlet and outlet pressure of the feed circuit of membrane test cell as well as the concentrate flow rate were also monitored throughout the experiments. From the inlet and outlet pressure values it was possible to calculate the average transmembrane pressure (TMP) inside the test cell.

Before starting each experiment, de-ionized water was pumped into the membrane test cell for one hour and a half at the maximum TMP and cross-flow velocity. Immediately thereafter, the same procedure was repeated with the synthetic salt solution to minimize irreversible membrane compaction in the course of the ion-rejection measurements. Then, the experiment was started. An initial sample of the feed solution was taken. During the experiment the temperature and the flow rate were kept constant, while the TMP was increased in steps of 2 bars from 1 to 20 bar. At each TMP, several permeate samples were collected after the permeate conductivity reached a constant value. When increasing the TMP,

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