



## Desalination of effluents with highly concentrated salt by nanofiltration: From laboratory to pilot-plant

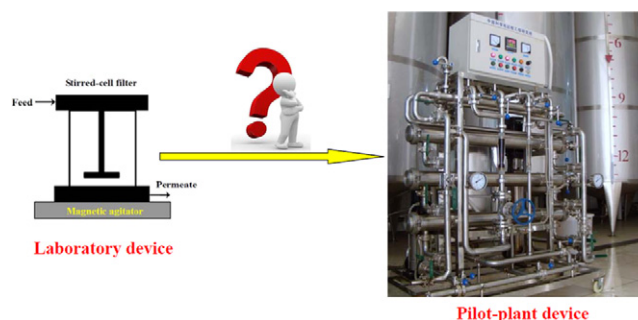
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### HIGHLIGHTS

- ▶ Highly concentrated salt facilitates solutes to diffuse through NF270 membrane.
- ▶ Highly concentrated salt increases concentration polarization.
- ▶ Water rinse can regenerate NF270 membrane for desalination of iron dextran.
- ▶ Desalination of IDA mother liquor can ensure continuous IDA production.
- ▶ Dilution–concentration mode is most suitable for desalination of soy sauce.

### GRAPHICAL ABSTRACT



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### ABSTRACT

Nanofiltration (NF) has been widely used for treatment of industrial effluents, but very few work concerns NF process in concentrated saline solution, especially for NF-desalination aiming at permeation of monovalent salts and retention of organic solutes. In this study, NF270 membrane was chosen to treat model solutions and three industrial effluents with highly concentrated salt (crude iron dextran solution, iminodiacetic acid mother liquor, and raw soy sauce), showing that with increase of salt concentration, the retention of all the solutes decreased while concentration polarization was increased. In the presence of charged organic solutes, inorganic salt retention would decline, even negative retention of monovalent salt was found. Increasing pH would induce membrane swelling in saline solution, which might be caused by the higher local salt concentration around the membrane polymers at higher pH. As NF-desalination of industrial effluents with highly concentrated salt was scaled up from laboratory to pilot-plant, the dead-end stirred filtration at constant flux could provide some important information for pilot-plant tests, such as membrane selection, optimum operating parameters and mechanism analysis, but it was necessary to re-optimize operating mode and method for crossflow filtration at constant pressure, in order to control the concentration polarization at high salt concentration.

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### 1. Introduction

The biochemical industries, especially pharmaceutical synthesis and food processing, frequently produce crude feeds or waste streams containing highly concentrated salt (e.g. NaCl, up to 20%, w/v) [1–8]. These mixtures are produced by salt-adding preparation or by acid-

alkali-catalyzing reactions followed by neutralization. A mass of salt in these industrial fluids induces many problems in their post-treatment, and also increases health risks due to an excessive intake of salt. Moreover, quite a lot of organic products are difficult to be extracted from concentrated saline solution, causing a pollution problem when they are discarded without purification. Therefore, partly removing salt from these effluents is important, not only for the environment, but also for improving product quality. The existing alternative treatment methods, such as crystallization, extraction, ion-exchange and electro dialysis, are usually too expensive to be industrialized and the cost of treatment can eventually undermine the economic viability of

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the whole process with progressing tightening of environmental regulations.

Nanofiltration (NF), as a membrane separation technology using both electric charge (Donnan effect) and pore size (sieving effect), could separate low molecular weight solutes (e.g. glucose, saccharides, amino acid, and peptide) from inorganic salt solutions [5,9,10], and simultaneously concentrate organic solutes and remove inorganic salt, showing a great potential in desalination and/or the recovery of valuable organic substances (permeation of monovalent salts). Moreover, the salt ions could easily pass through the membrane at high salt concentration [10], even the nominal monovalent salt rejection was often negative in mixtures of salts and large charged organic molecules or mixed monovalent–multivalent salts [4,5,11,12], thus greatly decreasing osmotic pressure difference across membrane. Consequently, NF technique has been considered as a promising approach to treat the effluents containing high concentration salt ( $>1$  M).

NF270 membrane was mainly applied in purification of drinking water because of its very high water production capacity [13,14]. Recently, increasing applications of NF270 on industrial effluents were reported [4,5,7,15–18], indicating that this membrane had relatively high retention and permeability as well as strong antifouling performance. Mänttari et al. [19] compared NF270 with several other NF membranes (e.g. NTR7450, Desal-5 DL, NF-PES-10) in terms of glucose retention and water permeability, and verified that NF270 had both a relatively high retention ( $>90\%$ ) and a high permeate flux ( $>12$  Lm<sup>-2</sup> h<sup>-1</sup> bar<sup>-1</sup>). This abnormally high retention–permeability property made NF270 suitable for desalination of the concentrated effluents. In laboratory scale, NF270 was successfully applied in desalination of three effluents with highly concentrated salt, soy sauce [4], iminodiacetic acid (IDA) [5] and iron dextran complex [7]. Luo et al. [4,5,7] found that, for NF-desalination of these effluents, a dilution operation could improve desalination efficiency, and alkaline pH could enhance salt removal and charged solute retention, and salt concentration affected solute retention and concentration polarization. However, NF process in concentrated saline solution should be elaborated more systematically, and in order to industrialize these applications, pilot-plant tests are needed to be carried out.

In this paper, NF270 membrane was applied to desalination of effluents with highly concentrated salt at both laboratory and pilot-plant scales, and the NF-desalination process was divided into three categories – separation of salt and neutral solutes (iron dextran),

separation of salt and charged solutes (IDA), and separation of salt and mixed solutes (soy sauce). This NF-desalination application aims at permeation of monovalent salts and retention of organic solutes. The focus of this work was to discuss salt effect on solute retention and concentration polarization, and analyze the relationships and the deviations between laboratory studies and pilot-plant tests. The present work should be very useful for understanding NF process under high concentration salt conditions and to industrialize the desalination of concentrated effluents using NF270 membrane.

## 2. Materials and methods

### 2.1. Experimental set-up and procedure

In laboratory studies, the dead-end filtration was carried out in constant flux mode, and this laboratory-scale set-up was described in detail elsewhere [4,5,7], as shown in Fig. 1. This device is fitted with a membrane disk having an effective diameter of 24 mm within the module, with an effective membrane surface area of  $4.52 \times 10^{-4}$  m<sup>2</sup>. The experiments were performed in three modes: concentration mode, diafiltration mode, and full recycle mode. First, the stirred cell was filled with feed, and then, for concentration mode, feed was continuously pumped into the cell and more and more organic solutes accumulated in retentate; for diafiltration mode, deionized water was injected into the cell with feed and the salt concentration in feed decreased continuously; for full recycle mode, a solution with the same solute concentration as permeate was put into the cell to ensure that the filtrations were performed in a way similar to the total recycling operating mode (i.e. circulating the permeate back to the stirred cell to keep the solution compositions unchanged).

In pilot-plant tests, crossflow filtration was adopted under constant pressure, and the schematic diagram of the pilot-plant NF-system is shown in Fig. 2 [20]. This homemade NF pilot device was equipped with a 100 L capacity feed tank and two multistage pumps (CRN3-36, Grundfos, Denmark). Pressure was measured by both digital and analog manometers, and, as transmembrane pressure (TMP) reference value, the mean value between the inlet and outlet of membrane module was taken, and pressure drop never exceeded 0.9 bar. Temperature was controlled by circulating water. Crossflow flux and permeate flux were determined by rotameters. The membrane module of NF270-4040 was supplied by DOW-Filmtec and consisted of a spiral-wound

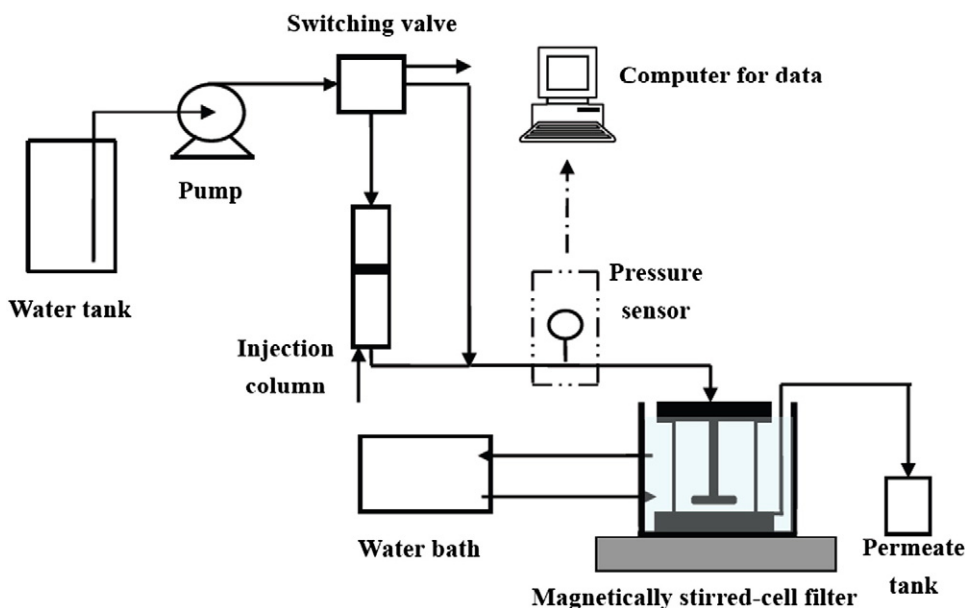


Fig. 1. Schematic diagram of the laboratory NF system.

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