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Short Communication

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# Effects of anti-scaling and cleaning chemicals on membrane scale in direct contact membrane distillation process for RO brine concentrate



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#### ABSTRACT

Membrane distillation (MD) is a promising desalination technology for water recovery from reverse osmosis (RO) brine. However, calcium sulfate as the major pollutant in RO brine concentrates induces scale occurrence in MD. Effective control of membrane scaling and cleaning of the membrane module are the key technologies for the industrialization of MD. In this work, effects of four anti-scaling and five cleaning agents on calcium sulfate scaling in direct contact membrane distillation (DCMD) process were investigated and discussed. It was found that membrane scaling and wetting was significantly alleviated with the addition of an effective anti-scaling agent. The flux recovery of a membrane with five cleaning agents was also improved and the flux recovery using a 2% EDTA-4Na solution was obtained as high as 92.8% at 77 °C.

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#### 1. Introduction

Reverse osmosis (RO) brine discharge is an important issue in RO desalination due to the loss of water resources and the disposal problem. RO brine minimization is proposed as a solution as it can recover precious water and mineral resources as well as alleviate environmental problems. Several potential methods for RO brine minimization have been explored [1,2]. Membrane distillation (MD), based on the thermally driven transport of vapor through the pores of a hydrophobic microporous membrane, is a promising technology for water recovery from high salinity solutions such as RO brine. Mass and heat transfers occur simultaneously in MD [3], but this process is not limited by concentrate polarization and can be used for concentrated RO brine.

However, as in all other membrane processes, significant bottlenecks occur in MD because of membrane fouling [4]. In MD, notably in direct contact membrane distillation (DCMD), a high pressure difference is absent between the two sides of the membrane, membrane fouling is not so serious as that in other membrane processes such as ultrafiltration, nanofiltration and reverse osmosis. However, pollutants may be present in DCMD, which include suspended particles, colloids, organic matters and

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inorganic deposits [5-12]. CaCO<sub>3</sub> and CaSO<sub>4</sub> are believed to be the most important pollutants in desalination.

CaCO<sub>3</sub> and CaSO<sub>4</sub>, as two insoluble salts, are widely present in RO brine concentrates. Compared with CaCO<sub>3</sub>, CaSO<sub>4</sub> is more difficult to remove and requires more attention. When a saturated solution of CaSO<sub>4</sub> was concentrated using a DCMD, CaSO<sub>4</sub> was found to precipitate on the membrane surface and penetrate into the membrane pores. This penetration led to an increase in the conductivity of the distillate from a few to thousands of uS/cm within hours [13]. Sun et al. [14] concentrated RO brine via membrane distillation and found substantial deposits in the RO brine if no pretreatment was taken. This deposition blocked the membrane pores and resulted in a decline in membrane flux. After an acidification pretreatment, the flux decrease reduced to a certain extent because acidification avoided the deposition of CaCO<sub>3</sub>. However, CaSO<sub>4</sub> and silicide were still deposited on the membrane surface. When 50 mg/L PC-290 (an anti-scale agent) was added into the RO brine, no apparent deposition was found on the membrane surface after 112 h and the flux decreased by 10.9% only [14].

An effective control of fouling and improvement of cleaning plays important roles in prolonging the continuous work time and life of a membrane, which can reduce the operation cost of MD processes. The effective control of membrane fouling and the cleaning of the membrane module are the key technologies in the industrialization of MD. However, the current techniques for the fouling control are mainly feed pretreatment and membrane

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cleaning [15]. Few studies have been conducted on membrane scaling control and cleaning when a RO brine with a high CaSO<sub>4</sub> concentration is treated with MD. This technology must be explored for RO brine reduction. He et al. [16] investigated the effects of two antiscalants on membrane scaling using calcite and gypsum in a DCMD process.

Previously, we found that, if RO brine was concentrated with MD, an enrichment factor significantly affected the flux and electrical conductivity of the distillate. When the enrichment factor was less than 3.5, CaSO<sub>4</sub> did not crystallize. Membrane fouling was exacerbated after the enrichment factor reached 3.5 and the major pollutant was found to be CaSO<sub>4</sub> crystals. Square CaSO<sub>4</sub> crystals were formed and completely covered the membrane surface at 77 °C, whereas only snowflake crystals are sporadically adhered to the surface at 55 °C [17]. In this paper, we focus on the antiscaling efficiency in CaSO<sub>4</sub> scale using anti-scaling and cleaning agents. The effects of four agents on the flux and the electrical conductivity of the distillate in the DCMD concentration of RO brine and the effect of five cleaning agents on CaSO<sub>4</sub> scale were comprehensively investigated.

#### 2. Experimental

#### 2.1. Materials

The chemical agents used in this study including sodium chloride (NaCl), potassium chloride (KCl), magnesium sulfate (MgSO<sub>4</sub>), magnesium chloride (MgCl<sub>2</sub>) and calcium sulfate (CaSO<sub>4</sub>) in analytical grade were purchased from Beijing Chemical Works. Anti-scaling reagents, MDC-220, MDC-150, and MDC-706, were obtained from GE (USA) while PC-191 was received from NALCO (USA). Calcon-carboxylic acid powder was purchased from J&K Scientific Ltd.

The polyvinylidene fluoride (PVDF) hollow-fiber membrane modules were obtained from the Institute of Biological and Chemical Engineering (Tianjin University of Technology, Tianjin, China) and their characteristics are shown in Table 1.

#### 2.2. Preparation and analysis of feed solutions

A simulated RO brine solution at 46.5 g/L was used as a feed, which was prepared from tap water and salts in the composition of primary RO [17]. The electrical conductivity of tap water was  $625-650 \mu$ S/cm. The RO brine compositions are as follows: 36.21 g/L NaCl, 1.03 g/L KCl, 4.52 g/L MgCl<sub>2</sub>, 2.91 g/L MgSO<sub>4</sub> and 1.80 g/L CaSO<sub>4</sub>. The Ca and SO<sub>4</sub><sup>2-</sup> concentrations in the RO brine were 0.53 g/L and 3.60 g/L, respectively. The pH and electrical conductivity of the RO brine were in the ranges of 7.0–7.5 and 85-95 mS/cm, respectively. The pH (using a Leici; PHS-3C; China) and the electrical conductivity (using a Ridao; DDS-12A; China) of the feed and distillate solutions were measured at regular time intervals.

#### Table 1

Properties of PVDF membrane modules used in experiments.

Parameters	Data from manufacturer
Fiber o.d/i.d (mm)	1.2/1.0
Fiber thickness ( $\delta$ ) ( $\mu$ m)	200
Maximum pore radius $(r_{max})$ (µm)	0.22
Mean pore radius (r) (µm)	0.16
Porosity $(\varepsilon)$ (%)	85
Water entry pressure LEP <sub>w</sub> (kPa)	150
Water contact angle ( $\theta$ ) o.d/i.d (°)	84.3/86.4
Effective fiber length $(L)$ (m)	0.23
No. of fibers ( <i>n</i> )	50
Effective membrane surface area ( $\times 10^{-2} \text{ m}^2$ )	3.45

#### 2.3. DCMD process

The DCMD process was studied in a continuous mode using a fully automated experimental configuration (Fig. 1) [17]. The hot feed flowed in the lumen side and the cold distillate flowed in the shell side. The feed was continuously pumped from the thermostatted water bath to the membrane module by a peristaltic pump (Longer Pump, BT600-2J, China). Two temperature sensors were placed at the inlet and outlet of the hot side of the membrane module, respectively. The heating element and the temperature sensor were connected to a temperature control unit that was used to regulate the feed temperature. After the feed started flowing through the membrane module, a given volume of the concentrated solution was continuously discharged and the remaining flow was returned to the thermostatic water bath. A storage tank continuously supplied the feed solution. The distillate was circulated from a 500-mL distillate tank through the membrane and back to the distillate tank. The distillate tank allowed the excess distillate water to overflow into a measuring cylinder. This cylinder was used to continuously measure the volume. Two other temperature sensors were installed at the inlet and outlet of the distillate side, respectively. The distillate was cooled in a 60-cm graham condenser. The flow rates of the feed and distillate were monitored using two rotameters and were maintained equally to each other and constantly.

Each DCMD experiment lasted approximately 500 min. At the start of the experiment, the RO brine at 46.5 g/L in the water tank was continuously pumped to the thermostatic water bath. Deionized water with an electrical conductivity of 4  $\mu$ S/cm was circulated on the cold side of the membrane. The rotary speed of the peristaltic pump was adjusted to a specific value. The inlet temperatures of the feed and the distillate were 77 °C and 35 °C, respectively, with an accuracy of ±0.5 °C. The flux and electrical conductivity of the distillate were measured at a steady state. The solutions in the thermostatic water bath and the water tank were stirred to prevent CaSO<sub>4</sub> precipitation. The concentrated solution was discharged from the membrane module to maintain the enrichment factor at 3.5, which was the optimum value for the DCMD concentration of RO brine [17].

The flux of the distillate was calculated as follows:

$$J = \frac{m}{S \times t} \tag{1}$$

where *J* is the flux of the distillate,  $kg/(m^2 h)$ ; *m* is the weight of the distillate, kg; *S* is the effective membrane area,  $m^2$ ; and *t* is the operating time, h.

The membrane module was dried after each experiment. It was firstly flushed with tap water for approximately 1 h, dried in air for 1 h and finally dried in a vacuum drying oven (100 °C, 10 h). The initial flux varied with the different modules, and thus the change in the flux, *J*, could not be used to accurately represent the extent of membrane wetting and fouling. First, the initial pure water flux ( $J_0$ ) was measured. A relative flux ( $J/J_0$ ), the ratio of the actual flux (J) to  $J_0$ , was used as an index of the variation in flux to eliminate the effect of  $J_0$ . The  $J/J_0$  value may have exceeded unity when  $J_0$  was measured at a feed flow rate of 0.205 m/s, However, *J* was obtained at a higher feed flow rate.

#### 2.4. Anti-scaling tests

When Na<sub>2</sub>SO<sub>4</sub> and CaCl<sub>2</sub> solutions are mixed, the Ca<sup>2+</sup> and SO<sub>4</sub><sup>2-</sup> in the solution form CaSO<sub>4</sub> crystals and the Ca<sup>2+</sup> concentration in the mixture significantly declines. If an anti-scaling agent is in the mixture, CaSO<sub>4</sub> crystallization is inhibited. Therefore, the Ca<sup>2+</sup> concentration in the mixture will not significantly decrease. The Ca<sup>2+</sup> concentration in the mixture can reflect the anti-scaling

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