



Occurrence of salt breakthrough and air-vapor pocket in a direct-contact membrane distillation



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HIGHLIGHTS

- Salt breakthrough and vapor pocket in membrane distillation was investigated.
- Salt permeation will occur in hydrophobic polyvinylidene fluoride membrane.
- Hydrophobic polytetrafluoroethylene membrane prevents wetting and salt permeation.
- An increase in packing density increases the membrane distillation flux.

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ABSTRACT

A polyvinylidene fluoride (PVDF) hollow fiber module with a low packing density and the permeate orifices on one side was used in a direct-contact membrane distillation process (DCMD). Comparative tests were also performed using a highly hydrophobic polytetrafluoroethylene (PTFE) membrane and a low hydrophobic PVDF membrane with different packing densities. Under varying operating conditions such as the feed and permeate flow rates as well as temperature, the performances of the DCMD were comprehensively investigated. It was rarely found that salt breakthrough in an idle mode and air-vapor pocket in an operative mode could occur. Partial wetting was for the first time found tending to happen in the PVDF membrane in idle mode, which led to salt breakthrough. High feed temperature and flow rate accelerated partial wetting. On the contrary, the PTFE membrane prevented partial wetting and blocked salt permeation. The air-vapor pocket formation at the shell side was attributed to a high vapor flux and incomplete condensation. An increase in packing density and the inversion of permeate orifices could help the mixture of hot vapor and cold permeate eliminate the air-vapor pocket, which was for the first time observed and illustrated.

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1. Introduction

In the past few years, a growing interest has been focusing on membrane distillation (MD). The publications show that MD is becoming an increasingly important process for water treatment. Except for some new configurations with an improved energy efficiency, higher permeate flux or smaller foot print [30], there are usually four basic MD configurations: direct-contact membrane distillation (DCMD), vacuum membrane distillation (VMD), air-gap membrane distillation and sweep-gas membrane distillation. DCMD has gained wide-spread interests for researchers because of its simplicity, high flux and no requirement for a high vacuum [10,15,26,27,34].

Membrane wetting, however, is a major constraint on MD industrialization. In general, surface hydrophobicity depends upon free energy per unit area of a surface. A local hydrophilic area may occur on a hydrophobic surface, which will be easily wetted in a MD process. Fouling and scaling also reduce the local hydrophobicity and result in surface wetting. For example, the deposits of a non-volatile solute, salts, carbohydrates and proteins will exacerbate wetting. Goh et al. [6] found fouling, especially due to amphiphilic matters, accelerated the wetting and reduced the hydrophobicity of membrane surface. Gryta found that CaCO₃ and CaSO₄ could lead to surface wetting [7]. Limonene could also induce membrane wetting [1,31].

Membrane wetting occurred in two steps: surface wetting and partial wetting [9]. During the early stage of a MD process, water filled in the big pores located at the membrane surface (surface wetting). With MD process running, a significant decline in the permeate flux happened and certain fragments of the membrane wall became completely

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wetted (partial wetting). An increase in the permeate conductivity usually suggested the occurrence of partial wetting [19,33].

In the DCMD process based on hollow fibers, the feed usually flows at lumen-side and the permeate flows at shell-side of the fibers. We recently found salts at lumen-side could penetrate through the PVDF membrane, leading to an increase in conductivity of the water at shell-side, when the MD process was stopped. However, if a polytetrafluoroethylene (PTFE) module was used, the conductivity of the permeate might decrease. These variations suggested that salts had permeated from the lumen side through the membrane into the shell side. Our tests also showed that, despite numerous washing of the shell-side loop with ultrapure water, the conductivity of the permeate was still relatively high when the test began. For examples, during 90-h continuous operation, the conductivity of the permeate could decrease from 40 $\mu\text{S}/\text{cm}$ to 15 $\mu\text{S}/\text{cm}$ for a PVDF hollow fiber membrane [5]. The observations were also reported in other investigations [23–25], but no one discussed this phenomenon of the salt breakthrough in the MD process at idle conditions. Practically, MD systems would be in idle conditions during the maintenance or solar-DCMD systems are at night-time operation.

Besides the salt breakthrough, a gas phase occasionally appeared during DCMD operation using a counter-current flow due to larger driving temperature difference and higher permeate flux [8,13]. Gryta found an accumulation of inert gases at the shell-side when the hot feed was in the lumen and the cold permeate was in the shell of fibers, resulting in reduced mass transfer and permeate flux [8]. Then, he investigated the effects of different arrangements of the module (horizontal and vertical) and the flow directions of permeate (upward and downward) on gas accumulation and suggested a solution using an additional port-valve in the upper part of the vertical module, showing that the decline of the permeate flux was prevented.

We also found the similar issue of gas accumulation in DCMD. The gas was mainly composed of water vapor and air from the feed. During the long-term processing of either pure water or saline solution, water vaporization occurred at the upper section of the module shell and the section covered by the air and vapor would gradually expand, leading to appearing of an air-vapor pocket at the shell-side. During the expansion process, the permeate flux decreased rapidly and the salinity of the permeate increased sharply.

As the salt breakthrough and air-vapor pocket are important issues for DCMD operation, but they have not been well studied before. In this work, we firstly presented a systematical investigation and analysis to elucidate their occurrence under the different operation conditions. The aims of this study were to present an insight into the relationship between air-vapor pocket, salt breakthrough, flux decrease and membrane wetting in the DCMD process and to guide further work on the design of hollow fiber modules and optimization of operating conditions to facilitate the MD commercialization.

2. Experiments

2.1. Materials

The chemicals used in this study include sodium chloride (NaCl), potassium chloride (KCl), magnesium sulfate (MgSO_4), magnesium chloride (MgCl_2) and calcium sulfate (CaSO_4), supplied by Beijing chemical works. A concentrated RO brine solution was used as a feed made from tap water and the above salts in the same composition as primary RO [5], which is composed of 32.61 g/L NaCl, 1.03 g/L KCl, 4.52 g/L MgCl_2 , 2.91 g/L MgSO_4 and 1.80 g/L CaSO_4 . The properties of RO brine are summarized in Table 1, and the concentrations of Ca^{2+} , Mg^{2+} and SO_4^{2-} are close to those in RO brine from North China [28]. The electrical conductivity (measured using a conductivity meter, DDS-12 A, Ridao, China) of the synthetic RO brine was 85–95 mS/cm. When the RO brine was concentrated in the continuous DCMD process, a concentration factor of 3.5 was maintained, and the total salt concentration increased to 162.8 g/L

Table 1
Water quality of RO brine.

Composition	Concentration
pH	7.0–7.5
Na^+ (g/L)	12.8
Ca^{2+} (mg/L)	529
Mg^{2+} (mg/L)	1724
Cl^- (g/L)	23.7
SO_4^{2-} (mg/L)	3598

[5]. Thus, the RO brine was used as the feed flowing in the hot lumen-side of hollow fibers

The PVDF and PTFE hollow-fiber membrane modules were supplied by the Tianjin University of Technology and their specifications are presented in Table 2. The housings of the PVDF and PTFE modules were made of glass tubes. This enables the observation of vapor pocket at the shell-side. The parameters of membrane, including porosity, LEP_w and water contact angle, are provided by the manufacturer.

2.2. DCMD process

The DCMD process was studied in a continuous mode using a fully automated experimental configuration (Fig. 1) [5]. The hot feed flowed at the lumen-side, and the cold permeate flowed at the shell-side. The feed and permeate were separately pumped into the hollow fiber membrane module using two peristaltic pumps (Longer Pump, BT600-2J, China). Two temperature sensors were placed at the hot side of the module, one at the inlet and the other at the outlet. A heating element and temperature sensors were connected to a temperature-controlled unit to regulate the feed temperature. A cartridge filter (5 μm polypropylene, Xiamen Tianshu Trading Company, China) was placed in line to prevent serious damage to the membrane surface from big CaSO_4 particles [12]. After the feed started flowing through the fiber lumen, a given volume of the concentrated solution was continuously discharged to maintain the feed content, and the remaining flow was returned to a thermostatic water bath (20 L). A storage tank (20 L) continuously supplied the feed solution. The permeate was circulated from a 500-mL collecting flask through the membrane and fed back to the collecting flask. The collecting flask allows the excess permeate to overflow into a measuring cylinder, which was used to continuously measure the volume. Two conductivity or temperature sensors were installed at the permeate side, one at the inlet and the other at the outlet. The permeate was cooled in a 60-cm Graham condenser. The flow rates of the feed and the permeate were monitored by two rotameters and were maintained at a constant rate.

The flux of the permeate was calculated as follows:

$$J = \frac{m}{S \times t} \quad (1)$$

Table 2
Properties of PVDF and PTFE membrane modules used in the experiments.

Parameters	PVDF	PTFE
Fiber o.d. (mm) / i.d. (mm)	1.2/1.0	1.2/0.9
Fiber thickness (δ) (μm)	100	150
Maximum pore radius (r_{max}) (μm)	0.22	0.75
Mean pore radius (r) (μm)	0.16	0.20
Porosity (ϵ) (%)	91	78
Water entry pressure LEP_w (kPa)	150	250
Water contact angle (θ) o.d. ($^\circ$)/i.d. ($^\circ$)	84.3/86.4	125/125
Effective fiber length (L) (m)	0.23	0.20
No. of fibers (n)	50/100/150	40
Effective membrane surface area ($\times 10^{-2}$ m 2)	3.45/6.9/10.35	2.26
Packing density (%)	15.1/30.3/45.3	14.2

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