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VACUUM MEMBRANE DISTILLATION OF LIQUID DESICCANTS UTILIZING HOLLOW FIBER MEMBRANES

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Research highlights

- Effective separation and fresh water recovery from high-salinity liquid desiccant draw solutions
- An average vacuum membrane distillation flux of $8 \text{ kg m}^{-2} \text{ h}^{-1}$ was obtained using 30 wt% magnesium chloride solution at a feed temperature of 50°C and an absolute pressure of 25 mbar on the vacuum side of the membrane
- Permeate recovered from liquid desiccant regeneration is of sufficient quality for use as agricultural irrigation water or drinking water.

Graphical Abstract

Abstract

This paper documents the testing of a vacuum membrane distillation system intended for use with liquid desiccants. Liquid desiccants offer the possibility for low-energy, ambient temperature dehumidification. Effective desalination and purification of diluted desiccants outputs two important products: a concentrated desiccant for reuse in dehumidification and fresh water. In this study, vacuum membrane distillation was used in the laboratory to purify diluted liquid desiccants. Calcium chloride and magnesium chloride were the desiccants selected for testing. Desiccant solutions were pumped through the lumens of poly(vinylidene fluoride) (PVDF) hollow fiber membranes at varying feed inlet temperatures, solution velocity rates and vacuum set points during membrane distillation. An average flux of $8 \text{ kg m}^{-2} \text{ h}^{-1}$ was obtained using 30 wt% magnesium chloride solution at a temperature of 50°C while applying vacuum to achieve 25 mbar absolute pressure on the air side of the membrane. The results are promising for the development of a full-scale vacuum membrane distillation process for desiccant solution regeneration and fresh water recovery. In addition, the recovered condensate was of sufficient quality for use in agricultural irrigation or drinking water.

Keywords: Desalination; membrane distillation; liquid desiccant; controlled environment agriculture.

1 Introduction

1.1 Background

Dehumidification, the removal of water vapor from the air, is responsible for a significant amount of the energy consumption during mechanical cooling of air in humid climates. The energy demand for cooling comprises approximately 52% of the total energy demand in Saudi Arabia in summer, including in humid coastal cities like Jeddah [1]. The energy used for dehumidification leads to both higher costs of electricity and a large carbon footprint where grid energy is sourced from fossil fuels. Estimates are that the energy efficiency of cooling equipment can be improved by up to 33% using innovative dehumidification technologies, such as liquid desiccants (LD) [2]. Concentrated salt solutions are commonly used as the liquid desiccant draw solution. The capacity of a desiccant to remove moisture from the air is governed by its concentration and temperature. A cool, concentrated desiccant is best for dehumidification [3, 4]. Therefore, it is necessary to remove water and heat from the LD solution to maintain a low vapor pressure and drive dehumidification.

1.2 Solution regeneration

Liquid desiccants also offer the opportunity to use renewable energy sources, like solar energy, for desalination and regeneration of the draw solution [5-15]. Desalination of the draw solution also offers a unique and sustainable source of fresh water, which is of special interest for use in agriculture in desert areas. As it relates to the regeneration of liquid desiccant solutions, multiple types of regeneration have been proposed. However, the best candidates for fresh water recovery that have both energy efficiency and a compact design are processes typically used in seawater desalination, including electrodialysis [26-28], reverse osmosis [29], and membrane distillation [16-22]. The use of membrane contactors with inorganic salt solutions has been used for a number of processes, including but not limited to dehumidification, forward osmosis, and water treatment [22-38]. Membrane distillation (MD), e.g. direct contact, air-gap, and vacuum (VMD), including multistage systems, is a desalination process with very good separation efficiency, providing high salt retention in the regenerated concentrate and high purity water recovery in the permeate [21, 22, 31]. VMD has been specifically applied by others for liquid desiccant regeneration with

flat sheet membranes. [17-20] In the current work, the use of hollow fiber membranes is proposed because of the high surface area provided per unit volume [39]. Tailored porosity of hollow fibers can offer efficient moisture transport if leakage can be prevented. In addition, the current work utilizes the vacuum pump in addition to the temperature difference to drive VMD flux.

In the present work we propose and test VMD of magnesium chloride and calcium chloride salt solutions pumped through the lumens of PVDF hollow fiber membranes. Calcium chloride and magnesium chloride were chosen for experiments because they are common salts for use in liquid desiccant applications. In addition, they are lower-cost alternatives when compared to salts such as lithium chloride [32, 40]. The driving motive behind this work is to integrate a dehumidification and water supply system inside a recycling-water controlled environment agriculture system. Of particular interest is the application of such a system in a closed, artificially lighted, vertical hydroponic or aquaponic production system such as those being operated for urban agriculture in major world cities. Such a system would allow local, sustainable crop production with a very low fresh water footprint [21].

2 Experimental

2.1 Hollow fiber fabrication

2.1.1 Materials

Poly(vinylidene fluoride) (PVDF), used for hollow fiber membrane fabrication in this work, was purchased from Kynar®/ Arkema Inc., Dubai (Grade – HSV 900). The polymer was dried overnight under vacuum at 60°C before use. The polymer dope solution was prepared by dissolving 12 wt% PVDF in a mixture of 8 wt% ethylene glycol (EG) and 80 wt% N-methyl-2-pyrrolidinone (NMP, ≥99.5%, Merck). The polymer was added in small portions to solvent mixture (EG+NMP) to avoid any lump formation and continuously stirred at 400 RPM overnight using an overhead mechanical stirrer. This process was carried out in a three neck flask, which was maintained at 70°C using oil bath.

2.1.2 Fabrication

A dry-wet jet spinning line (SeptraTec Inc. Korea) was used to fabricate PVDF hollow fiber membranes by a non solvent-induced phase inversion process. The prepared dope solution was transferred into the feed reservoir of the hollow fiber machine and degassed for at least 12 h, maintained at 50°C. The effect of the dope and bore liquid concentration and flow rates, air gap, take-up wheel speed etc. were investigated. A gear pump was used to pump the dope through the annular space of a double spinneret (needle ID – 0.3 mm, OD – 0.6 mm and orifice ID 1.0 mm) at a rate of 9 RPM (5.6 ml/min). Deionised water was used as bore liquid at 2.5 ml/min pumped using a dual head HPLC pump (with reduced pulsation). Air gap of 10 mm was maintained between the spinneret tip and 1st coagulation water bath (22°C). The stretching roller wheel speed was 11.25 RPM inside the 2nd coagulation water bath (22°C). The take-up wheel speed was approximately 4.8 RPM and used to collect the coagulated hollow fiber onto a bobbin, maintained within water bath temperature of 50°C. The hollow fibers were washed continuously with 50°C hot water for at least 12 h and stored in RO water for 3 days (with exchange of water every 12 h) to remove any residual solvents.

2.2 Hollow fiber characterization

2.2.1 Scanning Electron Microscopy (SEM) Analysis

The produced PVDF hollow fibers were dried at room temperature before storage for further use. The morphology of the surface and cross-section were observed using field emission scanning electron microscopes (FEI NovaNano and Zeiss Merlin) at an accelerating voltage of 5 kV. Fibers dried overnight in a vacuum oven (40°C) were carefully fractured in liquid nitrogen for cross-section analysis and sputter-coated with platinum (~3 nm-thick, on a Quorum Q150T ES).

2.2.2 Pore size distribution

The pore size and pore size distribution of PVDF hollow fiber membranes were measured using a Porolux™ 1000 IB-FT instrument (Germany). Porefil (perfluoroether) with a surface tension of 16 dynes cm⁻¹ was used as the membrane pore filler and up to 34.5 bar pressure range was applied to measure all the different pore sizes within the hollow fiber membrane. The liquid entry point (LEP), which is the minimum pressure required for liquid to pass through the membrane, was measured during this analysis.

2.3 Vacuum membrane distillation experimental setup

Membrane distillation experiments were carried out in a controlled laboratory environment. Figure 1 shows a schematic of the vacuum membrane distillation experimental setup. The salt solution was heated via a jacketed glass heat exchanger fabricated in the KAUST workshop. Heating fluid was delivered to the heat exchanger from a Lauda type RE 630 G heating unit with digital control of heating fluid temperature and flow rate. The temperature of the solution was logged in 15 minute intervals using an inline PT100 platinum resistance sensor (3 mm diameter x 150 mm length) immediately before entry into and after exit from the hollow fiber membrane module. The PT100 was connected to a PT-104 screw terminal adapter (PP660 from Pico Technology) and plugged into a Pico Technology PT-104 PT100 data logger (St Neots, United Kingdom). The pressure of the solution at the entry to the hollow fiber was monitored using a Keller brand Mano LEO 1, type -1 to 30 bar with a range of 0 to 31 bar absolute pressure (Winterthur, Switzerland). Flow rate of the solution was controlled and maintained by a Masterflex® L/S model no. 7523-80 peristaltic pump, size 0.1-600 RPM at 0.1 horsepower. The tubing used in the pump for experiments was Masterflex® 06440-25 Tygon® E-LFL. Refractive index, used to determine the concentration of the desiccant before and after experimental runs, was checked using a Reichert Technologies Brix/RI-Check Refractometer (Buffalo, New York, USA). The refractometer was calibrated daily with distilled water. Two vacuum systems were used to maintain vacuum in the system: (1) Buchi type V-700 with V-850 vacuum controller; (2) Vacuubrand type PC 3004 Vario vacuum system with digital control. The weight of the solution was monitored using a Sartorius CPA34000 weighing scale. The condensate from the membrane distillation process was collected in glass condensers with built in condenser coils. Condensers included a removable condensate collection flask. The flask was weighed

before and after experimental trials to determine the amount of condensate recovered. Two condensers were plumbed in series to condense water vapor: one before (under vacuum conditions) and one after the vacuum pump (at atmospheric pressure). To cool the condenser, coolant was delivered to condenser coils from a Huber Minichiller equipped with digital control of coolant temperature. To evaluate the condensate water quality, conductivity was measured using a WTW inoLab Multi 740 meter, with a TetraCon® 325 conductivity probe.

Figure 1. Vacuum membrane distillation experimental setup and flow diagram.

The various experimental variables as studied were:

- Solution type* – Calcium chloride and magnesium chloride solutions were evaluated during the course of this work.
- Solution concentration* - The solution concentration varied from 15-40 percentage by weight (wt%) calcium chloride and 15-35 wt% magnesium chloride in the

experiments. The actual concentration of the solution increased over time as water was taken out via membrane distillation.

- (c) *Variable temperature* – The solution temperature was varied from 25-65°C throughout all experiments. In the specific temperature effect experiments, the temperature was set at 40°, 50°, and 60°C respectively. A constant temperature was maintained for the duration of individual experimental runs. The vapor pressure of the solution, and therefore the flux of the water vapor out of the solution, varied as a result of the input temperature and the changing concentration.
- (d) *Vacuum pressure* – The vacuum in the membrane distillation process was applied such that the pressure varied from 3-100 millibars absolute on the air side of the membrane throughout all experiments. In the experimental trials to test the effect of the vacuum on the VMD flux, vacuum was applied to achieve absolute pressure levels of 25, 50, and 100 mbars.
- (e) *Solution velocity* – The solution velocity was varied from 0.7 to 1.1 m s⁻¹ throughout all experiments. The impact on vapor flux of solution velocity was evaluated at velocities of 0.7, 0.9 and 1.1 m s⁻¹.

Average flux was calculated based on the amount of condensate collected over time in each experimental run. In general, experimental runs lasted between 10 and 24 hours, depending upon the starting concentration of the desiccant solution (more concentrated solutions required longer experimental runs to recover similar volumes of condensate due to a lower average flux). Equation 1 shows the flux calculation and input variables.

Equation 1: Flux calculation for VMD

$$f = \frac{w}{t \times a}$$

Where:

f = average flux in kg m⁻² h⁻¹

w = weight of condensate collected in kg

t = time of experimental run in h

a = membrane surface area in m²

The wt% of salt in the solution was determined at the beginning and end of experimental runs by measuring the refractive index of the solution. The refractive index was then used to find the wt% of salt in solution from the relationship between solution concentration and refractive index [41]. The average wt% of the salt in the solution during each experimental run was used in plots of experimental results (see Figures 4-7).

3 Results and discussion

3.1 Scanning electron microscopy (SEM) analysis

PVDF hollow fiber membranes fabricated using 12 wt% polymer dissolved in mixture of EG (8 wt%) and NMP (80%) were examined by SEM. Images of the fiber cross-section, wall and surface scan are shown in Figure 2. SEM micrographs show macrovoids extending from both inner and outer walls forming an asymmetric pore distribution across the walls of the fabricated hollow fibers. Table 1 shows the measured characteristics of the PVDF hollow fibers.

Figure 2. Images of PVDF hollow fibers collected via SEM: (a) cross-section of fiber; (b) outer surface cross-section; (c) outer surface of hollow fiber; (d) inner cross-section; (e) inner surface view.

Table 1: Properties of hollow fibers

Membrane Type	PVDF
Total fiber length	395 mm
Fiber inside diameter	0.60 mm
Fiber outside diameter	0.87 mm
Active surface area per fiber	0.00074 m ²
Number of fibers per module	1
Module length	395 mm

3.2 Pore size distribution and liquid entry point (LEP)

Hollow fiber membranes fabricated for this work have a narrow pore size distribution around 290 nm measured by porosimetry. The liquid entry pressure (LEP) value was about 1.25 ± 0.2 bar, which corresponds to a pore size of 511 nm. The LEP values were measured using porofil (perfluoroether, surface tension 16 mN/m) which has lower surface tension than water (72 mN/m), hence the LEP value would have been higher if water were used as the wetting liquid.

3.3 Effect of solution concentration

Effects of the solution concentration on membrane distillation flux were observed in the results of all experimental trials, and are therefore presented and discussed in the following sections.

3.4 Effect of salt solution type

The first set of experimental trials compared the flux for calcium chloride and magnesium chloride. To allow a clear comparison, the concentration (wt%) of each solution was normalized to an Equilibrium Relative Humidity (ERH) value [42]. The ERH is the minimum humidity at which water vapor moves from surrounding air into a liquid desiccant and is directly correlated to the vapor pressure of the solution. Figure 3 shows that flux increased with ERH, as expected. The higher the vapor pressure of the solution, the higher the driving force between the desiccant solution and the vacuum side. Average flux varied from $2.0 \text{ kg m}^{-2} \text{ h}^{-1}$ to $6.7 \text{ kg m}^{-2} \text{ h}^{-1}$ over the measured range of desiccant solution ERH. The

membrane distillation flux varied primarily as a function of the solution vapor pressure, while the type of inorganic salt used was not influential.

The flux obtained in the present work was higher than the flux obtained in VMD of desiccant solutions at equivalent desiccant ERH values in studies conducted by others. [19, 20] However, the current work was conducted in a much different experimental setup and at a much different temperature and pressure conditions than the previous studies, making the results difficult to compare.

Because the type of desiccant salt was not influential upon the VMD flux, further experiments to evaluate the effects of other variables, such as temperature and vacuum, were carried out using only one type of salt. Magnesium chloride was selected over calcium chloride because of its lower corrosion potential with the stainless-steel used in the in-line temperature sensors [43]. Temperature sensors began to show significant corrosion effects from calcium chloride solution after only three days of experimental run time. In contrast, the temperature sensors showed only minor corrosion effects from magnesium chloride over the course of all experiments.

Figure 3. Membrane distillation water flux as a function of the equilibrium relative humidity using feed solutions of calcium chloride and magnesium chloride. Fiber dimensions: 0.62 mm internal diameter and 400 mm length; temperature 30°C; 0.83 m s⁻¹ solution velocity; 3 mbar absolute pressure

3.5 Effect of solution temperature

The temperature of the solution entering the membrane distillation process greatly affected the flux. Figure 4 shows the flux obtained in each trial. Note that each point in the

figure represents a complete individual trial, where the points are the average solution concentration and flux obtained, not instantaneous. In general, both the flux at each solution concentration and the final concentration of solution that was achieved increased with the desiccant feed temperature. As well, flux decreased as the concentration of magnesium chloride increased.

Implications of these results for application in full-scale liquid desiccant regeneration include:

1. Investing in heating of the solution and/or better heat retention/utilization will pay off in terms of expected flux from the VMD process. If solar thermal is used as the heat input into the system, this will require either more solar thermal collection area or higher efficiency (and more costly) collection equipment.
2. Operating the process at a lower solution concentration will pay off in terms of a higher flux from the VMD process when using the same membrane area. The solution concentration set point will need to be selected taking into account both the desired flux from VMD and also the minimum required solution concentration for dehumidification.

Figure 4. Flux from VMD at varying feed temperatures. Fiber dimensions: 0.66 mm internal diameter and 400 mm length; 0.83 m s⁻¹ solution velocity; 100 mbar absolute pressure

3.6 Effect of membrane distillation vacuum strength

Figure 5 shows that the flux increased with increasing vacuum strength (lower absolute pressure) and decreasing salt concentration.

Figure 5. Flux from VMD at varying vacuum set points. Fiber dimensions: 0.66 mm internal diameter and 315 mm length; temperature 50°C; 0.7 m s⁻¹ solution velocity

As well, the quality of the fresh water condensate from the membrane distillation process varied with the vacuum strength in these experiments. The average conductivity of the condensate collected was 130 $\mu\text{S}/\text{cm}$ at 25 mbar_{abs}, 190 $\mu\text{S}/\text{cm}$ at 50 mbar_{abs}, and 3600 $\mu\text{S}/\text{cm}$ at 100 mbar_{abs}. It is thought that the increase in conductivity resulted from a small leak in the module combined with wetting that took place at higher absolute pressures. That is, at a module pressure of 100 mbar_{abs}, condensation took place on the walls of fiber module itself, rather than in the condenser. The walls of the fiber module were exposed to room temperature, which ranged from 20-23°C over the course of the experiments, much cooler than the 50°C desiccant solution in the hollow fiber. Therefore, at 100 mbar_{abs}, the water vapor condensed in the module as it was exposed to its dew point temperature. At the lower absolute pressures of 25 and 50 mbar_{abs}, the dew point was not realized within the hollow fiber module. Hence, salt accumulation may have taken place as the water transitioned to vapor under the low absolute pressure, but salt transfer out of the module did not occur. Vapor in these lower pressure trials was not exposed to dew point temperatures until contacting the cooling coil in the condenser. Because the module may have had dry salts accumulating at low absolute pressures from the small leak, water droplets that condensed in the module at 100 mbar_{abs} may have carried salt that otherwise would remain in the module out and into the condenser.

Practically, a lower vacuum set point in a production-scale VMD system will provide a better flux and potentially a better quality of the recovered fresh water, but will also require more energy for operation. A cost/benefit analysis of the vacuum set point is recommended for completion in connection to a commercial-scale system.

3.7 Effect of solution velocity through hollow fiber

The impact of solution velocity on vapor flux through the hollow fiber was evaluated at velocities of 0.7, 0.9 and 1.1 m s⁻¹. For each trial, flux and pressure drop through the fiber were recorded. Figure 6 shows the average flux for each velocity tested. The velocity of the solution through the hollow fiber had a small affect on the flux at the velocities tested. For example, at around 32% salt by weight, there appears to be less than 1 kg m⁻² h⁻¹ of flux increase when the velocity is increased from 0.7 m s⁻¹ to 1.1 m s⁻¹.

The pressure drop through the hollow fiber, however, was more affected by the increase in velocity (Figure 7). At around 32% magnesium chloride by weight, the pressure drop increases from around 0.25 bars to 0.33 bars when the velocity is increased from 0.7 m s⁻¹ to 1.1 m s⁻¹.

Figure 6. Flux from VMD at varying velocities. Fiber dimensions: 0.66 mm internal diameter and 315 mm length; solution temperature 50°C; 25 mbar absolute pressure

Figure 7. Pressure drop through hollow fibers in VMD at varying velocities. Fiber dimensions: 0.66 mm internal diameter and 315 mm length; solution temperature 50°C; 25 mbar absolute pressure

Flux was expected to increase with increasing solution velocity because of a lower solution residence time in the hollow fiber. It was expected that a lower residence time in the hollow fiber would lead to a lower exiting solution salinity. Lower salinity corresponds to a higher vapor pressure, and thus an expected higher driving force leading to increased flux. The mass transfer resistance coefficient of the membrane phase ($\frac{1}{k_m}$) and gas ($\frac{1}{k_g}$) on the distillation side were not expected to change with varying solution velocities, only the average vapor pressure of the solution in residence within the hollow fibers was expected to increase with increasing solution velocities.

In addition to flux, pressure drop was also expected to increase with solution velocity through the fiber lumens. Pressure drop due to friction increases proportionally with velocity in closed, pressurized flow channels. A larger pressure drop correlates to a higher pumping energy requirement and also leads to a higher risk of hollow fiber breakage or leaks.

4 Conclusion – Outlook

Experimental results confirmed that vacuum membrane distillation using PVDF hollow fibers was effective in desalination of the calcium chloride and magnesium chloride draw solutions. Increasing the inlet temperature of the solutions increased the desalination flux. Reducing the vacuum set point increased the desalination flux and improved condensed fresh water quality within the lab scale experimental setup. Increased solution velocity through the

hollow fibers had a small effect on increasing the desalination flux but had a much higher effect on increasing the headloss through the hollow fiber lumens. Desalinated water collected during liquid desiccant regeneration has very low salt concentration and is suitable for use in irrigation.

The integration of liquid desiccant dehumidification and water recovery systems are of special interest for use in controlled environment agriculture (CEA). Irrigation in world agriculture is estimated to contribute to as much as 70-80% of global fresh water resources use. Commonly consumed vegetables like tomatoes and lettuce can have water footprints (the amount of water required to irrigate a crop to produce one kilogram of product) approaching 200 liters per kilogram. The growing of crops indoors allows for the potential recovery of plant transpired water vapor. In such a recycling-water system, the water footprint of the same vegetables may be reduced to as low as 1-4 liters per kilogram of harvest, a potential water footprint reduction approaching 98-99%.

A pilot-scale experiment is planned to scale up from the laboratory module to an outdoor, solar-powered vacuum membrane distillation system. This pilot membrane distillation system is planned for testing in combination with a liquid desiccant dehumidification system to be built inside of a sustainable, low water footprint, controlled environment agriculture system. The inlet solution temperature achieved by solar heating, the cutoff vacuum level, and the flow rate through the hollow fibers as a function of pumping energy are planned for assessment during the pilot scale experiments.

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List of abbreviations and nomenclatures

PVDF - Polyvinylidene fluoride

LD – Liquid desiccant

MD – Membrane distillation

°C – degrees Celsius temperature

EG – Ethylene glycol

NMP - N-methyl-2-pyrrolidinone

RPM – Rotations per minute

OD – Outer diameter

ID – Inner diameter

mm – millimeters of length

SEM - Scanning electron microscopy

LEP - Liquid entry point

wt% - percentage by weight

mbar – millibars of pressure

mbar_{abs} – millibars of absolute pressure

m/s – meters per second velocity

ERH – Equilibrium relative humidity

K_m – Membrane phase mass transfer coefficient

K_g – Gas phase mass transfer coefficient