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Unsupported Electrospun Membrane for Water Desalination Using Direct Contact Membrane Distillation

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Abstract

In this project, an unsupported electrospun poly(vinylidene fluoride)-co-hexafluoropropylene (PVDF-HFP) membrane was used for water desalination using direct contact membrane distillation (DCMD). The membrane was electrospun using a laboratory-scale machine with multiple nozzles that was developed in-house. Critical process parameters, including the applied voltage and polymer concentration, were optimized to obtain bead-free electrospun membranes with fiber diameters less than 300 nm. To improve the membrane thermal stability and performance, the selected electrospun membrane was heat-pressed at 160°C. The untreated and heat-pressed membranes were tested in a DCMD setup at different feed temperatures (60, 70, and 80°C) and feed flow rates (0.4, 0.6, and 0.8 L/min), while maintaining the permeate temperature and flow rate at 20°C and 0.2 L/min, respectively. The modified electrospun membrane exhibited a very high permeate flux ($>37.5 \text{ kg/m}^2/\text{h}$) and a salt rejection rate of 99.99% at a feed temperature of 70°C. The performance of the heat-pressed unsupported PVDF-HFP electrospun membrane was nearly identical to a commercially available PTFE supported membrane. These promising results demonstrate that relatively low-cost electrospun membranes can be easily produced and successfully used in DCMD to minimize the capital cost and increase the energy efficiency of the process.

Keywords: desalination, direct contact membrane distillation, electrospun membrane, pore size, salt rejection, permeate flux

1. Introduction

Membrane-based technologies have gained considerable popularity for water desalination due to their high separation efficiency, relatively low cost, and ease of operation. Membrane distillation (MD) is a promising thermally-driven separation method that is currently used for several everyday applications, including water treatment and juice concentration. Applying MD for water desalination is an emerging technology and has the potential to replace traditional energy-intensive techniques including multi-stage flash distillation, vapor compression, multiple effect distillation, reverse osmosis, and forward osmosis. The configuration, operating conditions, and membrane properties for MD processes in the context of full commercialization have been studied extensively over the past decades. Compared to the reverse osmosis process, MD is less sensitive to fouling and can completely reject salt (99.9%). The energy consumption of the MD process can also be significantly reduced with the use of heat pipes and solar collectors.¹⁻⁴

The MD process combines fundamental concepts of seawater evaporation/condensation with membrane separation.⁵ The feed side, which contains salty water, is heated to 60-80 °C and concurrently, water vapor is passed through a hydrophobic micro-porous membrane for collection as pure water at the cold side.³ The mass transfer driving force in this separation process is the vapor pressure difference between the hot and cold sides across the membrane and is caused by the temperature difference imposed by the operating conditions. Due to the hydrophobicity of the porous membrane, only vapor molecules from the volatile feed constituents cross through the membrane pores. Consequently, the non-volatile feed contaminants remain on the hot side while only pure water is collected on the permeate side.⁶⁻⁸

Among the various types of MD, direct contact membrane distillation (DCMD) is most widely used due to its simple setup and performance.^{6,9} In DCMD, the membrane is in direct contact with both the hot feed and the cold permeate sides at atmospheric conditions.⁵ Compared to traditional distillation techniques, the DCMD process requires lower operating heating temperatures and vapor spaces. Furthermore, unlike reverse osmosis (RO), it operates at atmospheric pressure.^{6,7} Therefore, DCMD is considered a low energy-consuming technology that can be integrated with low-grade thermal waste energy resources including waste heat from electric power plants (heat transfer fluid and boiler gases), high-temperature

by-products from industrial applications, or renewable energy sources including solar and geothermal energy.^{2,4} In addition, DCMD is characterized by 100% theoretical retention of non-volatile water-soluble species including ions, colloids, macromolecules, cells, salts, and heavy metals.^{1,2,7} DCMD is also capable of purifying brine solutions that contain high salt concentrations without traditional evaporator corrosion problems or concentration polarization effects.^{1,6,7} Moreover, the precipitation of organic and inorganic molecules, fouling, and scaling are minimal in DCMD due to its configuration and operation at atmospheric pressure.²

Despite the attractive advantages of DCMD, its industrial application has been hindered by technological challenges. The most significant drawback of DCMD is its low permeate flux when compared to other techniques, such as reverse osmosis and nano-filtration.⁶ In addition, DCMD performance is susceptible to heat loss, mass transfer resistance, temperature polarization between sides of the membrane, and entrapped vapor molecules within the membrane pores.³ Although fouling or scaling is less of an issue in MD when compared to other separation methods, it is still a limitation, especially when it causes pore wetting. Pore wetting can negatively affect MD productivity and permeate quality (salt rejection %).^{3,10,11} Foulants can be either organic (natural organic matter) or inorganic (sulfate, carbonate, chloride salts) species.⁴

Several studies have attempted to improve DCMD performance by modifying the membranes: (a) to improve their anti-fouling characteristics by increasing their liquid entry pressure (LEP) and super-hydrophobicity, (b) to improve their thermal and mechanical properties by incorporation of nanoparticles and coating the final membrane surface with different agents including silicon oxide, zinc oxide, carbon nanotubes, MOFs, silica, iron, Teflons, graphene oxide, fluorosilanes, aluminum oxide, and Zirconium dioxide.¹²⁻²³

Electrospinning is a promising technique to produce microporous hydrophobic membranes. The electrospinning process depends on a high voltage difference between the tip of the feeding nozzle and the membrane collector drum. The resulting membranes are good candidates for DCMD applications because they have high surface roughness, strong mechanical properties, large contact angles, high porosities, low tortuosities, and high surface area-to-volume ratios.^{24,25} The electrospinning operating conditions, which include the voltage difference, tip to collector distance, duration, and polymeric solution composition, flow rate, and concentration, need to be optimized to obtain a suitable, defect-free membrane.^{26,27}

In this study, we developed a (13-tip) multi-nozzle large scale electrospinning apparatus to increase the speed of membrane production and fabricate PVDF-HFP unsupported hydrophobic porous membranes. Using this hand-made machine, we were able to quickly study the effect of the various operating parameters of electrospinning on the membrane fiber diameter, and to find the optimal conditions in a very short time. The heat pressed unsupported membranes were successfully used for water desalination. The “unsupported” electrospun membrane showed very promising DCMD results. The effects of various system parameters were investigated to optimize the membrane performance and highlight the potential of the proposed technique for desalination with DCMD.

2. Materials and Methods

2.1 Materials and Chemicals

Dimethylformamide (DMF) with $\geq 99.8\%$ purity, acetone with $\geq 99.8\%$ purity, and polyvinylidene fluoride-co-hexafluoropropylene (PVDF-HFP) with an average molecular weight of 455,000 g/mol and a density of 1770 kg/m³ were purchased from Sigma. Commercial PTFE membranes with an average pore size of 0.45 μm were purchased from Sterlitech, USA. Commercial polyester screen fabric with an average pore diameter of 350 μm and a thickness of 215 ± 2 μm was used to cover the cylindrical collector ($D = 10$ cm) of the electrospinning machine. All chemicals were used without further purification.

2.2 Membrane Fabrication

2.2.1 Electrospinning machine

We developed a multi-nozzle electrospinning machine with 13 nozzles to increase the membrane production speed (Fig. 1). The high voltage power supply and the syringe pump were purchased from Gamma (Ormond Beach, Florida) and Longer Pump (Model: TJ-3A, China), respectively. The drum collector was handmade using stainless steel wires attached to a controllable motor. The wire had a length of 25.5 cm and a diameter of 10 cm. The spinneret was comprised of 13 nozzles that were aligned in a single row with 1.25 cm-spacing between

each nozzle. The nozzle module was fixed on a slow sweeping system that moved at a speed of 129.6 mm/min. A schematic of the rapid, multi-nozzle electrospinning machine is displayed in Fig. 1.

Figure 1. Schematic of the homemade multi-nozzle electrospinning machine: (1) High voltage power supply, (2) Syringe pump, (3) Polymeric solution, (4) Sweeping system, (5) Multiple conductive nozzles, (6) Grounded drum collector, (7) Collector motor

2.2.2. Polymer solution preparation

PVDF-HFP polymeric membranes are widely used in DCMD applications²⁸⁻³⁴ due to their hydrophobicity, excellent thermal stability, suitable mechanical properties, and low cost.³⁰ Three different concentrations of PVDF-HFP (14%, 16%, and 18%) were prepared by adding PVDF-HFP pellets to a 1:1 mixture of DMF-Acetone (V/V). The mixture was stirred at room temperature for approximately 24 hours at an apparent speed of 600 rpm to dissolve the polymer. Before electrospinning, the polymeric solution was allowed to rest for few hours to remove entrapped air bubbles.

2.2.3. Nano-fibrous membrane fabrication

The drum collector ($D = 10$ cm) was first covered with aluminum foil and then a polyester mesh. A 60-mL plastic syringe was then filled with the polymer solution, which was then fed to the system nozzles at a flow rate of ~ 0.38 mL/h/nozzle using a syringe pump. The tip to collector distance (TCD), the collector speed (CS), and the electrospinning duration were set at 15 cm, 600 rpm, and 6 hours, respectively. The applied voltage and initial polymer concentration were varied in the range of 20-26 kV and 14-18 wt.%, respectively, to produce the smallest possible fibers without defects or beads.

2.3. Membrane Characterization

An accurate thickness gauge (MP-1, Brunswick Instrument, UK) with a probe tip and floating stand anvil was used for measuring the fabricated membrane thickness. The measurements were collected according to ASTM-D1777 by applying a pressure of (~ 1464 Pa) on the objects.

A scanning electron microscope (SEM MIRA 3 LMU Tescan, Czech Republic) with an InBeam detector was used to measure fiber diameter and study membrane morphology. Small samples from each membrane were coated with a thin layer of gold using a Q150 T turbo-pumped sputter coater (Quorum Technologies) and analyzed with the SEM. The working distance between the lenses and the sample varied between 10 mm and 16 mm, with an acceleration voltage of 20 kV. An in-beam detector at a magnification range of 20-50 K was used. The SEM micrographs of the membranes were analyzed using MIRA image-processing software to measure the average fiber diameter.

The contact angle of a water droplet (5 μ L) on the hydrophobic porous electrospun membranes was measured using an optical tensiometer (OCA 15EC, DataPhysics, Germany). The contact angle is defined as the angle formed between the liquid-vapor interface and the liquid-solid interface.

A capillary flow porometer (CFP-1100AH, PMI, NY, USA) was used to estimate the pore size distribution within the fabricated membranes. Briefly, a membrane was inserted into

the membrane cell holder and wetted using Galwick (surface tension of 15.9 mN/m) as the wetting agent. The flow of saturated air (Q), passing through the membrane was steadily increased, and the resultant pressure difference (ΔP) was measured. The pore size distribution was then calculated based on the measured flow rate (Q) as a function of pressure difference (ΔP).³⁵ The liquid entry pressure (LEP) of the hydrophobic porous membranes was also measured with the capillary flow porometer. For this measurement, a layer of water (instead of the wetting agent) was added on top of the membrane. The LEP is the maximum hydrostatic pressure that a membrane can tolerate before leaking.

2.4. Membrane heat pressing

An electrospun membrane without any support was sandwiched between two aluminum plates ($21 \times 14.5 \text{ cm}^2$) and heat-pressed in an oven by adding weights. The heat-pressing was conducted at a pressure of 6.5 kPa and a temperature of 160°C for 4 hours. The time of heat pressing was shorter than what reported in the literature³⁶ because we did not observe any further reduction of the membrane average pore size diameter after 4 h.

2.5. Membrane Testing using a DCMD Set-up

The electrospun membrane performance was tested in a lab-scale DCMD setup (Fig. 2). The setup consisted of two digital precise refrigerated/heating bath circulators supplied by (DAIHAN-brand®, South Korea). The cooling and heating water streams, at pre-determined temperatures, were circulated into Pyrex-jacketed beakers. One beaker contained the cold permeate (deionized water), and the other beaker was filled with the hot feed (salt solution). A conductivity meter (CyberScan Con 11, EUTECH Instruments) was immersed in each beaker to monitor changes in solution conductivity. Two peristaltic pumps (WT600-2J, LongerPump, China) were used to circulate the permeate and feed from the beakers to the membrane holder (CF042A-FOT, from Sterli-Tech, USA) with an active membrane area of 42 cm². The NaCl salt concentration in the feed tank was maintained at 3.5 wt% (35 g/L). To study the effect of feed temperature on the overall permeate flux, the feed temperature was varied while keeping all other operating conditions fixed. Each experiment was repeated three times with a new membrane to ensure reproducibility.

Figure 2. The lab-scale DCMD setup.

A balance connected to a computer interface was used to measure the overall flux across the membrane, and the desalinated water mass was monitored. The flux and salt rejection were calculated using Eqs. 1 and 2, respectively.

$$F_w = \frac{\Delta m}{A \cdot t} \quad \text{Eq. (1)}$$

In Equation 1, F_w represents the water flux in kilograms per square meter per hour ($\text{kg}/\text{m}^2/\text{h}$), Δm is the change in permeate mass in kilograms (kg), A is the effective membrane surface area in square meters (m^2), and t represents the sampling time expressed in hours (h).³⁷

$$R(\%) = \frac{C_f - C_p}{C_f} \times 100 \quad \text{Eq. (2)}$$

In Equation 2, R is the rejection percentage (%), C_f is the concentration of salt in the feed stream, and C_p is the concentration of salt on the permeate side.³⁸ These values were measured with the conductometer.

3. Results and Discussion

3.1. Electrospinning Optimization

The fiber morphology and orientation within electrospun membranes are highly dependent on the polymer solution properties and electrospinning operating parameters. The rheological characteristics and surface tension of the polymer solution depend on the polymer molecular weight, the solution concentration, and the solvent. The electrospinning conditions, including the applied voltage (V), solution flow rate (FR), tip to collector distance (TCD), and duration can also affect the fiber size and morphology.²⁶

Various solvents have been already used in PVDF-HFP electrospinning including acetone, DMF, tetrahydrofuran, and Dimethylacetamide (DMAc), as well as a mixture of solvents including DMF and acetone (1:1 and 4:1 w/w), DMAc and acetone (1:1 w/w), acetone and DMAc (1:1 and 7:3 w/w).³⁹⁻⁴³ Therefore, using a mixture of solvents is a common practice to adjust the evaporation speed of the mixture to ensure the most desired morphology of the produced fibers. A mixture of DMF-Acetone (1:1 V/V ratio) was found to be suitable for the electrospinning of PVDF-HFP in this study.

We performed several preliminary experiments to optimize the TCD and FR conditions. The optimal TCD and FR were 15 cm and 0.38 ml/h/nozzle, respectively. Varying the TCD between 13 and 18 cm did not have any noticeable effects on the fiber diameter and a TCD of 15 cm had been used in other studies.^{28,44,45} A FR of 0.38 ml/h/nozzle resulted in the best jet stability during the electrospinning process.

Figure 3 displays the relationship between the applied voltage and the average electrospun membrane fiber diameter at a fixed polymer concentration of 18 wt.%, a TCD of 15 cm, and a FR of 0.38 mL/h/nozzle. The average fiber diameter decreased when the operating voltage increased. The average fiber diameter was approximately 500 nm at an applied voltage between 20 and 24 kV and had a very high standard deviation (± 300 nm). The average fiber diameter decreased to approximately 400 nm with a much lower standard deviation (± 100 nm) when the applied voltage increased to 25-26 kV. The applied voltage is one of the main factors that is needed to overcome the viscous and surface tension forces of the polymer solution and is used to stretch the polymer towards the grounded collector in the electrospinning process. The applied voltage should be carefully adjusted and optimized in concert with the other

electrospinning operating parameters including the TCD, FR, and polymer concentration.⁴⁶ The trend of the average fiber diameter as a function of the applied voltage (Figure 6) is in agreement with the trends reported in other papers.^{35,43,47} However, it is important to note that the average fiber diameter can increase under the same operating conditions when the applied voltage is increased. This effect can be explained by the faster stretching of the polymer solution and a reduction in the solvent evaporation rate during the time of flight from the nozzle to the collector. This can produce fibers with larger diameters that contain more beads.³⁵ Mathematical models can be employed to estimate the average fiber diameter in electrospun membranes based on the properties of spinning solution and electrospinning operating parameters.^{27,48} These models can undoubtedly help researchers optimize their operating parameters faster.

PVDF-HFP electrospinning has been reported previously.^{29,40,49,50} Ching-Iuan Su et al. used a 8:2 mixture of Dimethylformamide and acetone to prepare the polymer solution and used an applied voltage of 30 kV, a TCD of 15 cm, and a polymer concentration of 17 wt.% to produce electrospun membranes with an average fiber diameter of 173 μm .⁴⁰ Wootthikanokkhan et al. fabricated electrospun membranes with an average fiber diameter of ~520 nm using an applied voltage of 10-14 kV and a polymer concentration of 12wt.%. They found that the average fiber diameter increased when the polymer concentration or applied voltage was increased.⁴⁹ The average fiber diameter in our PVDF-HFP membranes was smaller than what was reported in previous studies. This can be caused by multiple factors including the molecular weight of the PVDF-HFP, the polymer solution viscosity, and the applied voltage. Our data demonstrates that electrospun membranes with smaller average fiber diameters can be produced with proper optimization.

The effect of the initial polymer concentration on the average fiber diameter within the electrospun membranes was also investigated. Figure 4 illustrates that decreasing the polymer concentration from 18wt.% to 14wt.% caused the average fiber diameter to decrease to less than 300 nm. The relationship between fiber diameter and polymer solution concentration can be attributed to a change in the polymer viscosity that exits the nozzle.²⁷ A concentrated polymer solution and consequently, a high viscosity solution, increases the resistance of the newly formed fibers to stretching by charge at the nozzle; therefore, larger fibers are formed, which is consistent with our data.⁴⁶ Other groups have modeled the relationship between polymer concentration and fiber diameter as a third-degree power law.^{27,46,49} Micrographs of

the electrospun fibers are displayed in Figure 5. Overall, the optimal electrospinning conditions at room temperature were a polymer concentration of 14 wt.%, an applied voltage of 26 kV, a TCD of 15 cm, and a FR of 0.38 mL/h/nozzle.

The optimal applied voltage for our electrospinning process was 26 kV, which is higher than the values reported in similar studies (12-16 kV).^{49,51} The need for a higher applied voltage can be attributed to using a multiple-nozzle electrospinner instead of a conventional single-nozzle electrospinner. It is possible that the repulsion between the adjacent nozzles and a non-uniform electric field shift necessitated the higher applied voltage.⁵²

Figure 3. Effect of applied voltage on PVDF-HFP electrospun membrane fiber diameter. Electrospinning conditions were as follows: FR = 0.38 ml/h/nozzle, TCD = 15 cm, and [polymer] = 18 wt.%.

Figure 4. Effect of the polymer concentration on PVDF-HFP electrospun membrane fiber diameter. Electrospinning conditions were as follows: V = 26 kV, FR = 0.38 mL/h/nozzle, and TCD = 15 cm.

Figure 5. SEM micrographs of PVDF-HFP electrospun mesh at different polymer solution concentrations and heat-pressing. (a) 18 wt.%, (b) 16 wt.%, (c) 14 wt.%, (d) 14 wt.% after heat-pressing. The remaining electrospinning conditions were as follows: $V = 26$ kV, $FR = 0.38$ mL/h/nozzle, $TCD = 15$.

3.2. Effect of heat-pressing

Electrospinning is a common technique to fabricate nanofibrous membranes that are used for various applications. Chemical or physical modifications are two approaches that are used to improve the membranes' integrity and durability. For DCMD applications, there is a need for durable and thermally stable hydrophobic membranes with pore sizes less than 1 μm .⁵³ A small distribution in membrane pore sizes is required to ensure low liquid entry pressure and to prevent any leakage during the DCMD process.⁵⁴ A wide distribution of membrane fiber diameters can result in a wide distribution of pore sizes. Electrospun membranes with loose nanofibers, low inter-fiber connectivity, and large pores are vulnerable to wetting during the DCMD process.^{27,35,48} Heat-pressing is a viable technique that can fuse surface and bulk fibers together to enhance inter-fiber bonding and reduce the presence of large pores.³⁶ Heat-pressing is performed at a temperature below the melting point of the polymer. Successful heat-pressing should produce an electrospun membrane with better thermal stability, enhanced mechanical properties, and smaller pore sizes to resist against wetting during the DCMD process.

It is thought that heat treatments fuse individual fibers together at different nodes and also improves their connectivity to enhance their mechanical strength. In addition, the addition of heat may also permit the polymer to transition to a crystalline structure. Heat treatment has been applied to PVDF and PVDF-HFP polymers to improve their performance in DCMD water treatment applications.^{36,45,46,55}

In the present study, selected membranes were heat-pressed at 160°C for 4 h, a temperature that is slightly lower than the PVDF-HFP melting point. A representative SEM micrograph of a membrane after heat-pressing indicates that some of the fibers were fused together (Figure 5d). The properties of the heat-pressed electrospun membranes are summarized in Table 1. Both the average pore size and maximum pore size decreased after heat-pressing. The average pore size was approximately 0.45 μm and decreased to 0.35 μm after heat-pressing. Furthermore, the maximum pore size was 1.12 μm and decreased to 0.98 μm after the heat treatment. The decrease in the maximum pore diameter is also illustrated on the pore distribution graph shown in Figure 6. The distribution of pore diameters within the modified membranes was narrower and had a smaller peak diameter ($\sim 0.28 \mu\text{m}$) when compared to the non-heat-pressed membranes that had a peak pore diameter of $\sim 0.34 \mu\text{m}$. A narrower pore size distribution is desirable for DCMD applications because it reduces the

probability of wetting through the large pores, which would reduce the membrane performance.⁵⁶

After heat-pressing, the values of the water contact angle as well as the membrane thickness slightly decreased (Table 1). After the treatment, the liquid entry pressure (LEP) was enhanced with a new value of 5 kPa (~ 51 cm H₂O). The decrease in membrane pore diameter and thickness after heat-pressing was primarily due to nanofiber fusion caused by the elevated temperature and pressure. Because the heat-pressing was performed at a temperature close to the melting temperature of PVDF-HFP polymer ($\sim 165^\circ\text{C}$), the nanofibers partially melted and fused together. Importantly, when fibers are fused together at a high compression pressure, the void space between the fibers is minimized, which causes the membrane thickness to decrease and closes some pores.³⁶ The change in the porous membrane morphology before and after heat-pressing is visible on SEM micrographs (Fig. 5c and 5d). Consistent with our results, nanofiber fusion, a reduction in loose surface fibers, and a void reduction between fibers have also been reported in other studies after heat-pressing electrospun membranes.^{36,45,55,57}

For benchmarking, we used a commercial PTFE membrane with a reported cut-off pore size of $0.45\ \mu\text{m}$. Using capillary flow porometry, the average and maximum pore size of the membrane were found to be 0.33 and $0.72\ \mu\text{m}$, respectively. The average pore size of the heat-pressed electrospun membrane was very close average to the commercially available PTFE membrane. However, the maximum pore diameter of the heat-pressed electrospun membrane was noticeably larger, which resulted in lower LEP values (Table 1). Contact angle measurements indicated that the electrospun PVDF-HFP membrane was more hydrophobic than the commercial PTFE membrane. This could be due to the higher roughness of the electrospun membrane.

Figure 6. Pore size distribution of the selected membranes: the electrospun (Es, ■), heat-pressed (Hp, ◆), and commercial PTFE (PTFE, ▲)

Table 1. Characteristics of selected electrospun membranes (Es and Hp) and a commercially available PTFE membrane.

	Average Pore size (μm)	Maximum Pore size (μm)	Thickness (μm)	Water Contact Angle ($^{\circ}$)	LEP (kPa)
Electrospun (ES)	0.45	1.12	187 ± 17	134 ± 6	109.6 ± 0.6
Heat-pressed (Hp)	0.35	0.98	152 ± 11	131 ± 4	115.1 ± 0.6
PTFE	0.33	0.72	93 ± 8	124 ± 5	124.1 ± 46.1

3.4. DCMD Performance

To optimize the DCMD operating conditions with the heat-pressed membrane, different feed temperatures (60, 70, and 80°C) and various feed flow rates (0.4, 0.6, and 0.8 L/min) were tested while maintaining a permeate temperature of 20°C at a flow rate of 0.2 L/min. The average permeate flux and salt rejection (%) during the first two hours of the DCMD operation as a function of the feed temperature are reported in Fig. 7. As the feed temperature increased, the permeate flux increased for both the heat-pressed and commercial membranes due to an

increase in water vapor transport through the membrane.²⁴ Similar effects of the feed temperature on permeate flux have been reported in prior studies.^{24,40,45,58,59} It was notable that the permeate flux values of the heat-pressed and PTFE membranes were nearly identical. The permeate flux values exhibited a linear increase in response to increased feed temperature. The salt rejection for both of these membranes, when used for DCMD, was approximately 99.9%. These high salt rejection values confirmed successful DCMD operation without fouling or leakage.

Khayat et al. have explained the effect of temperature on permeate flux (F_w) with Eq. 3, which relates the flux to the mass transfer driving force. B_w represents the membrane's permeability and is a function of the applied temperature as well as pore size, porosity, thickness, and tortuosity of the pores in the membrane. In this system, the trans-membrane water vapor pressure difference (ΔP_w) drives the mass transfer.

$$F_w = B_w \Delta P_w \quad \text{Eq. 3}$$

The vapor pressure is a function of both the water vapor partial pressure P_w^0 and the membrane activity, a_w , as shown in Eq. 4.

$$P_w(x, T) = P_w^0(T) a_w(x) \quad \text{Eq. 4}$$

With respect to Eq. 4, a_w represents the membrane activity, P_w is the water vapor partial pressure in the solution, x is the non-volatile solute mole fraction, T is the absolute temperature (K) and P_w^0 (Pa) is the vapor pressure of pure water. Therefore, the water vapor pressure difference (ΔP_w) can be described by Eq. 5 where the subscripts w, f , and p refer to the water, feed, and permeate, respectively.

$$\Delta P_w = P_{w,f}^0 a_{w,f} - P_{w,p}^0 a_{w,p} \quad \text{Eq. 5}$$

In Eq. 4, the water vapor partial pressure in the solution P_w^0 is written as a function of the solution temperature, however, the exact relationship between water vapor partial pressure and temperature is described by Eq. 6.⁶⁰

$$P_w^0(T) = \exp \left(23.1964 - \frac{3816.44}{T - 46.13} \right) \quad \text{Eq.6}$$

As described by Eq. 6, as the temperature of the feed solution increases, the vapor partial pressure increases. An increase in $P_{w,f}^0(T)$ will increase the trans-membrane water vapor pressure difference (ΔP_w) as described by Eq. 5, when observed with a fixed value of $P_{w,p}^0$ (no change in the permeate temperature). Therefore, based on Eq. 3 when ΔP_w increases and the membrane permeability, B_w , is fixed, the permeate flux increases.

Figure 7. The permeate flux as a function of the feed temperature for the heat-pressed (Hp) and commercial PTFE membranes. The operating permeate temperature was maintained at 20°C. The feed and permeate flow rates were 0.4 and 0.2 L/min, respectively.

After increasing the feed flow rate while maintaining the remaining operating conditions, a noticeable increase in permeate flux was observed (Fig. 8). The increase in flux can be attributed to higher turbulence inside the DCMD module, which has been discussed in several other studies.^{24,40,59,61,62} The increase in permeate flux caused by a higher feed flow rate has also been attributed to a decrease in both the mass transfer boundary layer thickness and

the membrane surface temperature. Notably, the decrease in the mass transfer boundary layer reduced the effects of both concentration and temperature polarization due to an increase in the heat transfer coefficient. It also enhanced the driving force for vapor transport across the membrane to another.^{59,61}

At a flow rate of 0.4 L/min, the permeate flux values through the commercial and heat-pressed membranes were almost identical and had values of 22 kg/m²/h. The permeate flux increased for the Hp and PTFE membranes when the feed flow rate was increased. The salt rejection percentage of the DCMD process using these membranes remained at 99.9% even when the feed flow rate was varied between 0.4 and 0.8 L/min (Fig. 8).

Figure 8. The permeate flux as a function of the feed flow rate for the heat-pressed (Hp) and commercial PTFE membranes. The operating permeate flow rate was maintained at 0.2 L/min. The feed and permeate temperatures were 70°C and 20°C, respectively.

For DCMD applications, a limiting factor in efficiency is the heat transfer across the membrane boundary layer. This limitation is described by the polarization coefficient (θ), which explicitly represents the resistance across the membrane boundary layer divided by the total heat transfer resistance, as described by Eq.7 where $T_{m,f}$ and $T_{m,p}$ are the membrane

surface temperatures on the feed and permeate sides, respectively. $T_{b,f}$ and $T_{b,p}$ are the bulk temperatures of the feed and the permeate sides, respectively.

$$\theta = \frac{T_{m,f} - T_{m,p}}{T_{b,f} - T_{b,p}} \quad \text{Eq.7}$$

The polarization coefficient has a negative effect on DCMD performance, and it quantifies how a decrease in the vapor pressure difference is a driving force of the DCMD system efficiency. In the ideal situation, θ is equal to 1, but in real applications, it is usually less than 1.⁶⁰

An increase in the feed flow rate can overcome the effects of both temperature and concentration polarization. When the feed temperature is increased, the specific temperature and concentration of the non-volatile pollutants in this solution at the membrane surface become closer to the temperature and pressure of the solute in the bulk feed. The value of the polarization coefficient therefore approaches unity, as indicated by Eq. 7, and reduces the effect of mass transfer resistance on the overall DCMD performance.

These relationships and our data indicate that the performance of the heat-pressed electrospun PVDF-HFP membrane was close to the commercial PTFE membrane when DCMD feed temperature and flow rate were altered. More specifically, although the commercial membrane had a lower thickness and higher average and maximum pore diameter, our unsupported electrospun membrane still had a very high salt rejection percentage and a high permeate flux that were similar to the supported commercial membrane under harsh operating conditions. These results are clear evidence that the modified unsupported electrospun membrane successfully withstood high operating conditions during the process.

The performance, thermal stability, and durability of all the membranes were tested in the DCMD apparatus for 10 h. The measured permeate flux and salt rejection percentage under the same operating conditions are shown in Figs. 9a and 9b, respectively.

Figure 9. The stability of (a) the permeate flux and (b) salt rejection over time. The DCMD conditions were as follows: feed flow rate of 0.4 (L/min), feed temperature of 70°C, permeate flow rate of 0.2 L/min, permeate temperature of 20°C, and 3.5 % salt concentration.

In long-run performance tests, the untreated electrospun membrane had a flux value of approximately 23 kg/m²/h at the beginning of testing and decreased to approximately 19.6 kg/m²/h after only 240 min. The decrease in flux can be attributed to pore wetting caused by the presence of large pores and loose surface and bulk fibers. Pore wetting increases the amount

of salty water that enters the fibrous membrane pores and lowers the temperature difference between opposite sides of this membrane to reduce the driving force.⁶³ After an initial decrease performance, the flux increased to 23.4 kg/m²/h within the next 200 min. In the next 120 min, the flux continued to increase dramatically to 35 kg/m²/h. The severe continuous increase in the flux was due to a continuous increase in the number of wetted pores in the membrane. In the wetted pores, the salty water was able to enter gradually and continuously escape to the other side of the membrane. This indicated that the flux across the non-treated membrane was very unstable, mainly due to the membrane pore wetting. Importantly, the heat-pressed electrospun and the commercial PTFE membranes had relatively stable performance. The salt rejection percentage remained at 99.9% during the entire testing period. A slight deterioration in the permeate flux rate from 21.7 kg/m²/h at the beginning of the process to 17.4 kg/m²/h after 10 h was observed.

Both the commercial and heat-pressed electrospun membranes exhibited similar flux trends during the first 500 min of the long-run performance experiment. Although the initial flux of the commercial membrane was slightly higher when compared to the heat-pressed electrospun membrane, 22.8 kg/m²/h compared to 21.6 kg/m²/h, both membranes had a flux of 20.2 kg/m²/h after 500 min. After 500 min, the flux of the commercial membrane increased to approximately 20.9 kg/m²/h and remained almost constant for the duration of the test. Over the same testing period, the flux of the heat-pressed electrospun membrane decreased slightly to 17.4 kg/m²/h. The slight decrease in the permeate flux for the heat-pressed membrane at the end of a long-run experiment was observed in a prior study that also used a heat-pressed PVDF-HFP membrane.³⁶

During the long-run experiment, both the commercial and the heat-pressed membranes exhibited excellent salt rejection and had rejection percentages greater than 99.9% during the entire experiment. In contrast, the non-modified membrane had excellent salt rejection only during the first 400 mins of the experiment. After 400 mins, the rejection percentage started to severely deteriorate and reached a low value of 83.4% at the end of the experiment. The decrease in salt rejection can also be attributed to pore wetting, which allows more salty water to pass from the feed to pass through the membrane towards the permeate tank.²⁸

3.5. Comparison with other heat-pressed membranes for DCMD

Table 2 compares the DCMD performance of selected PVDF-HFP heat-pressed electrospun membranes and compares them to our study.^{36,45,55} The reported DCMD permeate values presented in the table range between 15 and 31 kg/m²/h and were obtained at ΔT of approximately 40 °C. As already discussed at length, the DCMD permeate flux depends on several parameters including the distribution of the membrane pore size, the temperature difference between the feed and permeate sides, the feed and permeate fluxes, and the configuration and geometry of the DCMD module. A very high permeate flux of 31 kg/m²/h was obtained for a heat-pressed electrospun PVDF-HFP membrane that had a very low thickness of 39 μm and a relatively large average pore size of 1.1 μm .⁵⁵ These values demonstrate that enhancing the integrity of electrospun nanofibers and reducing the thickness of the membrane with heat-pressing while still maintaining large pore sizes can improve DCMD performance. The membrane thickness and distribution of pore sizes are vital to water vapor transport and DCMD performance. A smaller thickness decreases the mass transfer resistance and a higher pore size diameter increases the permeability of the membrane, which both increase the permeate flux. However, a small membrane thickness requires a support to prevent the membrane's bulging and maintain constant permeate flux. The unique thing in our study, as compared to other heat pressing studies, is using a well-engineered electrospun membrane with an optimal thickness and strength that allows it to be used "unsupported" in DCMD. Using the membrane without a spacer or a support on either sides in the membrane module, not only conserves the initial surface contact area of water evaporation, but also saves money, energy, and material. Therefore, using unsupported membranes with suitable properties in a well-designed DCMD apparatus, may increase the efficiency of the process by maintaining high permeate flux and can make the process more environmentally friendly.

In addition to the membrane itself, DCMD performance is also affected by the operating conditions and the membrane holder design.⁵³ In this study, we demonstrated that a high feed temperature and flow rate result in higher permeate flux. The design of the membrane holder significantly affects the mass and heat transfer in DCMD. The membrane holder can also affect the temperature, concentration polarization effects, and membrane fouling. The membrane module should have a: (a) high ratio of membrane area to module volume, (b) low-temperature drop, (c) low-pressure drop to avoid excess trans-membrane hydrostatic pressure that may cause membrane flooding, (d) high heat transfer coefficient in the feed and permeate chambers,

(e) uniform temperatures of feed and permeate throughout the module volume, (f) uniform flow of fluids to avoid dead corners and channel formation, (g) proper sealing, and (h) good thermal and chemical resistance.⁶⁴

Forward osmosis (FO) membrane cell holders have been used in the majority of the previous studies, but are not the best choice for a non-isothermal DCMD process. Indeed, when the cold and hot flow inlets are perpendicular to the membrane surface, the streams exert additional non-uniform local pressure on the membrane, which causes local membrane wetting. The thermal stability and durability of the membrane is critically important.⁶⁴ The discrepancies between DCMD performance in the literature (Table 2) can be largely attributed to different membrane module characteristics. A better DCMD module design that directs the hot and cold flows parallel to the membrane without exerting additional local pressures would undoubtedly enhance the system performance.

In our membrane module, we added a polypropylene mesh that acted as a spacer to reduce the effect of temperature polarization. A polyester support mesh was also added to prevent membrane rupture and deflection. A pronounced decrease in the permeate flux was observed due to the reduction in effective surface area. The presence of spacers in the membrane module caused higher turbulence and modified the flow characteristics of the feed and permeate. Collectively, this reduces the effect of temperature polarization, which normally diminishes DCMD performance. The robust stability and integrity of the fabricated electrospun membrane enabled us to use it in the DCMD module without any additional support. Although other researchers may use supports or spacers in the DCMD membrane module, especially for membranes with low thickness and mechanical integrity, we were able to optimize our membrane and DCMD set-up without a spacer to maximize performance.

Table 2. Comparison of the DCMD performance of selected heat-pressed electrospun membranes.

#	Membrane type	Membrane properties ^a	T_{hot} - T_{cold} = ΔT (°C)	[NaCl] g/L	ϑ_f^b mL/min	ϑ_p^c mL/min	F_w^d (Kg/ m ² /h)	R^e (%)	Membrane Support	[Ref.] Year
1	Electrospun PVDF-HFP Heat-pressed at 200°C for 2 s	$\bar{D}$ =0.26 μ m t=110 μ m	68-24 =44	10	0.32 m/s	0.32 m/s	~20	~98	Metallic mesh-type spacers	[45] 2013
2	Electrospun PVDF-HFP Heat-pressed at 6.5 kPa, 150°C for 8 h	$\bar{D}$ =0.38 μ m t=195 μ m	60-20 =40	35	400	400	18	99.9	PP filter layer	[36] 2016
3	Electrospun PVDF-HFP Heat-pressed at 6.5 kPa, 150°C for 24 h	$\bar{D}$ =1.1 μ m t=39 μ m	60-20 =40	35	400	400	31	99.9	PP filter layer	[55] 2017
4	Electrospun PVDF-HFP Heat-pressed at 6.5 kPa, 160°C for 4 h	$\bar{D}$ =0.35 μ m t=152 μ m	60-20 =40	35	400	200	16	99.9	No support	This study
5	Commercially available PTFE	$\bar{D}$ =0.33 μ m t=93 μ m	60-20 =40	35	400	200	15	99.9	No support	This study

^a Notes: $\bar{D}$: average pore size diameter, t: thickness, ^b ϑ_f is the feed flow rate /velocity, ^c ϑ_p is the permeate flow rate /velocity, ^d F_w is the permeate flux, ^e R is the salt rejection percentage

Few studies can be found in the literature on the DCMD scale-up, energy, economic and cost analyses.⁶⁵ Two main reasons restricting the DCMD application on industrial scale are the high energy consumption and the risk of membrane wetting during the distillation. The former issue can be overcome by employing renewable energy sources such as solar and geothermal energy. More research works are needed on DCMD optimization and membrane

development to avoid the latter issue. However, there have been several attempts for scaling-up DCMD process by connecting several membrane modules in series or parallel.⁶⁶⁻⁶⁹

4. Conclusion

In this study, a homemade 13-tip electrospinning machine was used to fabricate hydrophobic PVDF-HFP membranes. The operating parameters were optimized to fabricate membranes with small average fiber diameters. The optimized conditions were a polymer concentration of 14 wt.%, an applied voltage of 26 kV, a FR of 0.38 mL/h/nozzle, and a TCD of 15 cm. Selected electrospun membranes were then heat-pressed at 160 °C for 4 hours to improve their thermal stability and durability for DCMD. The effects of the DCMD operating parameters including the feed temperature and flow rate on the permeate flux and the salt rejection (%) were also investigated. The untreated electrospun membranes failed during the DCMD process. However, the heat-pressed electrospun membranes exhibited excellent performance that was similar to commercially available PTFE membranes. The heat-pressed electrospun membranes maintained a stable permeate flux (water desalination) with 99.9% salt rejection. This study demonstrated that electrospinning could be scaled-up to fabricate membranes that are suitable for challenging applications including DCMD. The choice of a hydrophobic polymer (PVDF-HFP) for the membrane and heat-pressing at a temperature close to the polymer melting point improved the membrane integrity and thermal stability. The heat-pressing process reduced the membrane thickness, the average pore size distribution, and increased the liquid entry pressure (LEP). The commercially available PTFE membrane had slightly better performance than the heat-pressed electrospun membrane, which was probably because the commercial membrane had a smaller pore size distribution and was thinner. The average membrane pore size distribution may shift towards larger values at elevated temperatures, and the presence of large pores is responsible for pore wetting during the DCMD process. Therefore, heat-pressing the electrospun membrane greatly improved its DCMD performance. This modification was critical to a nearly stable flux and maintaining a stable salt rejection percentage of 99.9% during the entire 10 hours of the long-run performance test.

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