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Study on Membrane Fouling in Vacuum Membrane Distillation for Desalination

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Abstract—*In this work, membrane fouling was studied in vacuum membrane distillation for different pore sizes and membrane materials. Hydrophobic membrane of PTFE and PVDF was taken of two different pore diameter viz0.22 and 0.45 micron in a membrane module. The experiments were carried out using aqueous feed solution of NaCl at different feed concentrations over a period of 200hrs and 340hrs for different membranes. The decline in permeate flux was observed as 1.7% & 4.0 % for 0.22 µm PTFE membrane and 4.0 % & 9.0 % for 0.22 µm PVDF membrane in 200 hrs and 340 hrs continuous run respectively. Similarly the decline in permeate flux was observed for 0.45 µm PTFE & PVDF membranes, 2.6 % & 7.2% and 6.3% & 12.5% respectively in 200hrs and 340 hrs continuous run. SEM analysis was also carried out new brand and used membranes after 200hrs and 340hrs run time which confirmed the deposition of feed solute on membrane surface. This minor scale deposition is also evident from pore size distribution (PSD) which indicated that average pore size in PSD curve shifted from 0.22 µm to 0.18 µm and this little problem was very easily overcome by water washing.*

Keywords*: Vacuum membrane distillation, Specific energy, PTFE, Ion meter, NaCl, Reverse Osmosis.*

1. INTRODUCTION

Membrane distillation (MD) is known to be one of the growing non-isothermal membrane separation processes [1]. MD is a technique which leads toan almost complete water recovery. MD is defined as thermally driven transport process of vapor through hydrophobic membranes, the basic driving force in MD is the vapor pressure difference through membrane thickness. However, in other membrane separation processes, the basic driving force is the chemical potential difference through the membrane thickness [2–4]. Different MD configurations such as direct contact MD, sweeping gas MD, air gapMD, and vacuum membrane distillation (VMD) are used rapidly for various applications (desalination, water-reuse, food, medical, etc.). The resulting driving force of vapor pressure difference produces a flux of water vapor through the membrane, and thus, aqueous brine solutions can be concentrated and crystallized. This process can work on high solute concentration at feed side, at low concentration gradients, moderate temperature, and atmospheric pressure [5].

The advantages of VMD compared to other more popular separation processes are theoretically 100% rejection of ions, colloids, macro molecules, and other non-volatiles, lower operating pressure than conventional pressure-driven membrane separation processes, lower operating temperature than conventional distillation, and reduced vapor pressure compared to conventional distillation processes. VMD differs from the other membrane technologies in that the basic driving force for desalination is the difference in vapor pressure of water across the membrane, rather than total pressure. In VMD configuration, the vapor permeated does not condense in cooling chamber, but is drawn out by vacuum and condenses externally in a condenser. The pressure difference between the two sides of the membrane creates a convective mass flow along the pores that contribute to the total mass transfer for VMD.

In this paper, membrane fouling was extensively studied on membrane surfaces of different pore sizes and membrane materials. The deposition of salts was determined using the scanning electron microscopy (SEM) and the reduction of pore size was confirmed using pore size distribution analysis.

2. MATERIAL AND METHODS

The experimental VMD permeate flux (N, kg/m^2 h) is calculated by equation (1):

Where V is volume of permeate water (l); ρ is density of permeate water (kg/l); A is effective membrane area (m2) and t is the running time of VMD. The concentration of ionic species in the feed water (C1, mg/l) and in permeate water (C2, mg/l) were calculated by the conductivity meter [1,8]. The percentage removal (% R) of the species was calculated from equation (2):

$$R = \frac{C_1 - C_2}{C_1} \times 100$$

3. EXPERIMENTAL

A partially heated feed salt (NaCl) solution of different concentration was fed continuously from a feed tank to the hydrophobic PVDF membrane module as shown in VMD set up of Fig. 1. The vacuum was applied at the downstream side of the membrane as a result the vapour evolved in the upstream side of the membrane passes through the pores of the hydrophobic membrane and then condensed in the receiver using cooling water. The membrane characteristics are shown in Table 1. The permeate sample concentration was measured using conductivity meter.

Table 1: Membrane characteristics

Properties	Specifications			
Membrane material	PTFE	PTFE	PVDF	PVDF
Surface property	Hydrophobic			
Diameter, mm	90			
Effective membrane diameter, mm	52			
Pore size, µm	0.22	0.45	0.22	0.45
Thickness, µm	175	175	125	125
Porosity %	70	85	70	85
Liquid Entry Pressure, bar	2.80	1.24	2.04	1.05
Effective membrane area, m^2	0.00212			
Maximum operating temperature, °C	130	130	90	90
Supplier	Millipore			

4. RESULTS AND DISCUSSION

4.1 Effect of Membrane Fouling on Permeate Flux

In a continuous operation, the salt concentration of 5000 ppm was used as a feed solution for a VMD setup and the trans-membrane permeate flux collected continuously for about 200 hours for PTFE and PVDF membrane respectively at two different pore diameter of 0.22 and 0.45 µm. In 200 hrs of operation the flux remained almost constant for both, PTFE and PVDF membranes of 0.22 µm and 0.45 µm pore size as shown in Fig. 2. At 338 K, the permeate flux was 89.3 kg/m^2hr for PVDF membrane and 89.21 kg/m^2hr for PTFE membrane which remained nearly constant till 200 hours at 9.0 kPa permeate pressure and 6 lpm feed flow rate for pore diameter of 0.22 µm. It can be observed from the Fig. 2, that the flux decreased nearly 4% for PVDF and 1.7% for PTFE membrane respectively in 200 hours, which may be due to the minor fouling on the membrane surface. After, 200 hours the water washing was done and the membrane performance was checked again and it was observed that the flux regained to about 88.32 kg/m^2h for PVDF and 87.88 kg/m^2h for PTFE membrane under the same process conditions with 99.9% salt rejection. Further, it is also evident from the Fig. 2, that the decrease in flux for PTFE membrane is less as compared to PVDF membrane for both the pore sizes of 0.22 and 0.45 µm which may be because PTFE is more hydrophobic than PVDF. As shown in Fig. 1, the percentage decrease in permeate flux was more for higher pore size membrane of same membrane material. This may be due to the reason, the probability of solute entrapment inside the membrane pore is more likely, when the pore size of membrane is higher.

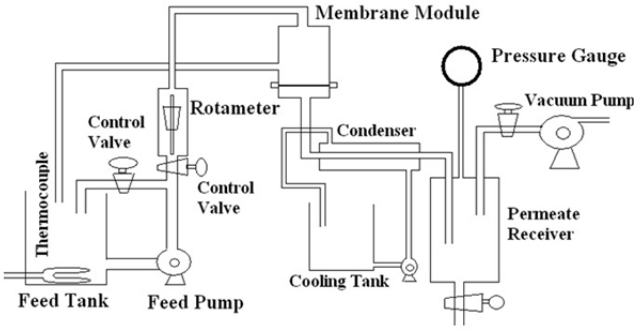


Fig. 1: Schematic representation VMD Setup

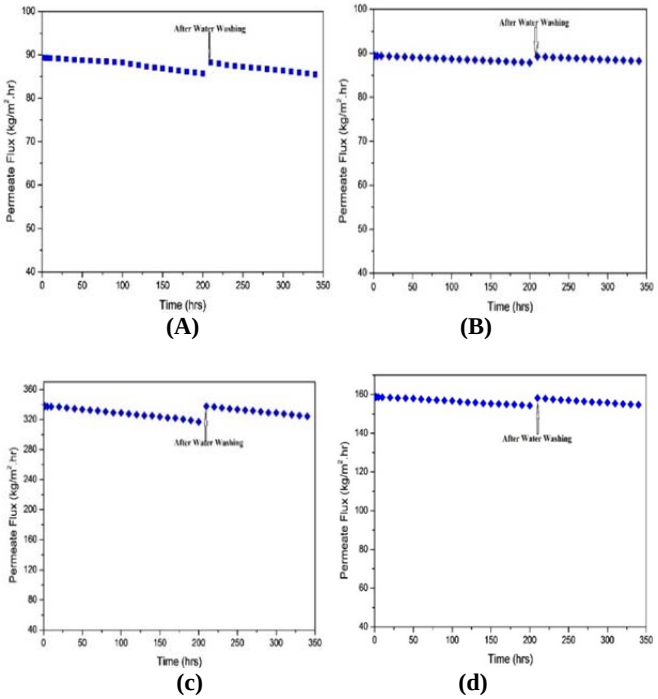


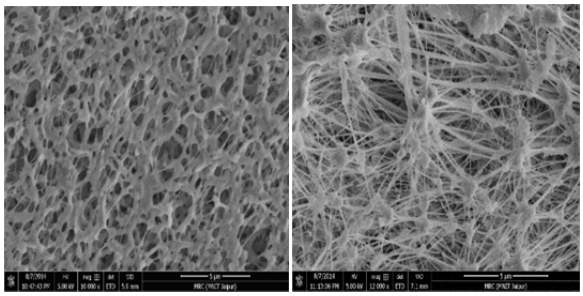
Fig. 2: Effects of operating time on permeate flux, (a) 0.22 µm PVDF, (b) 0.22 µm PTFE, (c) 0.45 µm PVDF, (d) 0.45 µm PTFE[Feed bulk temperature 65°C, feed salt (NaCl) concentration5000ppm, feed flow rate 6 lpm & permeate pressure 9.0 kPa]

4.2 Comparison of Membrane Morphology before and after Use of the membranes:

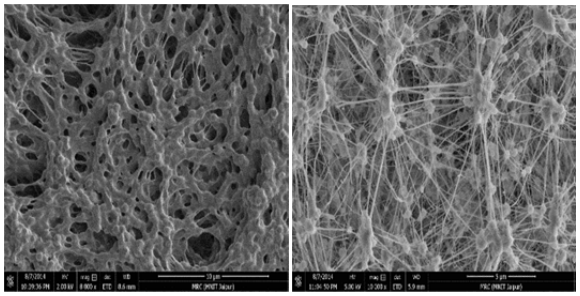
The membrane morphology was tested by scanning electron microscope (SEM). The membranes before and after its use in VMD process were analyzed. The SEM micrograph of brand new PVDF and PTFE membrane morphology for two different pore diameters of 0.22 and 0.45 µm are shown in Fig. 3. It is observed that the new membranes used in SEM study,

one pore diameter of PVDF and PTFE membrane found up to 0.35 µm and 0.30µm respectively, whereas the average pore diameter of the PVDF and PTFE membrane were 0.22 µm as per the specifications given by the manufacturer (Millipore). Similarly, from Fig. 3(c) and (d), the largest pore for PVDF and PTFE membranes were found to have diameter of 0.59µm and 0.54µm respectively, however, few pore were having diameter of less than 0.45 µm whereas the average pore diameter for the PVDF and PTFE membranes were 0.45 µm as per the manufacturer (Millipore). Hence, it may be concluded that average pore diameter reported by supplier was matching with the pore diameter measured from SEM. The other characteristics of fresh PTFE membranes are given in Table 1. Large pores of size 10 µm were also observed through SEM by other researchers Khayet et al. 2004, Tang et al. 2010,Banat and Simandl 1996,Karakulski et al. 2002 inspite the average pore size of accrual PP S6/2 membrane mentioned as 0.22 µm.

The performance of PTFE and PVDF membranes were checked by continuously using the membranes in VMD setup separately under 9.0 kPa of permeate pressure, feed flow rate of 6 lpm, feed inlet temperature of 65 °C and feed salt concentration of 5000 ppm for 200 hr run. The SEM micrograph of these membranes is shown in Fig. 4(a), (b), (c), (d). Minor fouling/scaling can be observed over the membrane surface. Therefore, little reduction in permeate flux was observed for PTFE membrane of pore diameter 0.22 and 0.45 µm after 200 hours of continuous use of membrane.



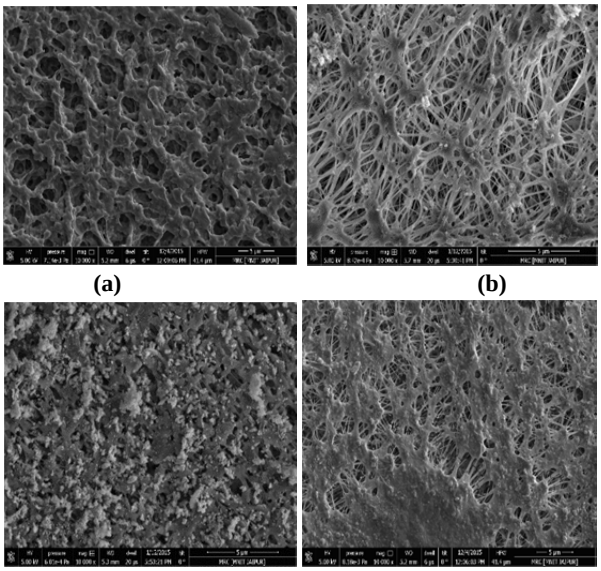
(a) **(b)**



(c) **(d)**

Fig. 3: SEM micrograph depicting pore size (new brand) (a) 0.22 µm PVDF (b) 0.22 µm PTFE (c) 0.45 µm PVDF (d) 0.45 µm PTFE.

Similar results were observed for PVDF membrane of pore diameter 0.22 and 0.45 µm also. The flux was almost found to regain its original value after washing the membrane with water. In the present work as shown in Fig. 2, the decrease in permeate flux is observed to 4% and 6.3 % for PVDF membrane of pore size 0.22 and 0.45 µm respectively and for PTFE membrane of pore size 0.22 and 0.45 µm decline in flux was observed 1.7% and 2.6%, respectively at 5 g/l of feed salt concentration over 200 hour run. This happened due to deposition of salts at the membrane surface as shown in Fig. 4.



(a) **(b)**

(c) **(d)**

Fig. 4: SEM image of used (a) 0.22 µm PVDF (b) 0.22 µm PTFE (c) 0.45 µm PVDF (d) 0.45 µm PTFE, after 200 hours run[Feed bulk temperature 65°C, feed flow rate 6 lpm, feed salt (NaCl) concentration 5000 ppm & permeate pressure 9.0 kPa]

The SEM micrograph of PTFE and PVDF hydrophobic membrane of different pore diameter of 0.22 µm and 0.45 µm after use of 340 hours continuously, at 9.0 kPa of permeate pressure, feed flow rate of 6 lpm, feed inlet temperature of 65°C and feed NaCl salt concentration of 5,000 ppm are shown in Fig. 5(a) (b) (c) (d). The Fig. is showing deposition which may be because of high NaCl feed concentration. Also, at this high concentration, the declination of 9% and 4% in permeate flux was observed for PVDF and PTFE membrane respectively for 0.22 µm pore size. On the other hand, the declination in permeate flux was found to be 12.5% and 7.2% for 0.45µm PVDF and PTFE membranes respectively. This type of decrease in permeate flux was supported by the SEM picture as NaCl deposit lapped feeble portion of the membrane surface which increases the temperature polarization effect and reduces the membrane permeability due to salt deposition.

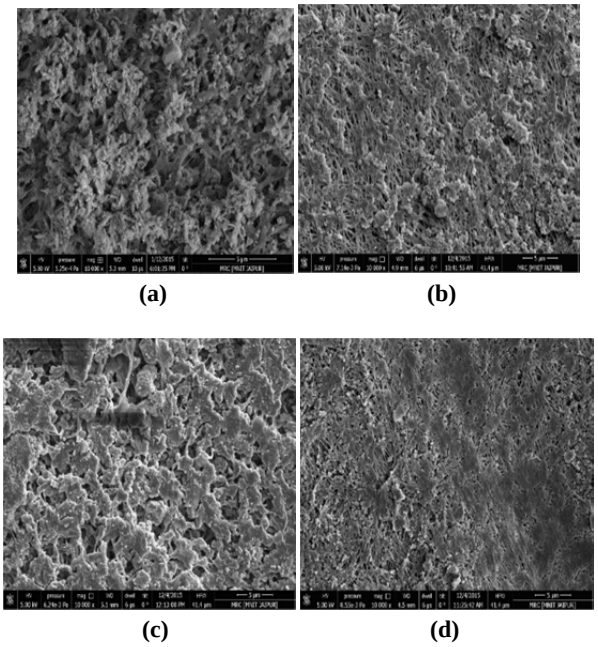


Fig. 5: SEM image of used (a) 0.22 µm PVDF (b) 0.22 µm PTFE (c) 0.45 µm PVDF (d) 0.45 µm PTFE, after 340 hours run.

Ultimately, the vapor pressure difference was reduced since there is reduction in partial pressure of the water vapor and significant decrease in the permeate flux was observed in the experimental run under the above mentioned condition. The membrane scaling and deposition was also reported by other workers Tang et al. 2010, Hou et al. 2012,Zhou et al. 2014 for tap water purification. Reason for decrease in permeate flux after continuous usage can also be attributed to variation in pore size distribution before and after use of both membranes. SEM images of new membranes, used membranes of PTFE and PVDF were taken to determine the pore size distribution (PSD) using software *ImageJ*. The developed PSD images for PTFE and PVDF membrane are shown in Fig. 6 and Fig. 7 respectively. As shown in Fig. 6, the average pore size for new PTFE membrane of 0.22 µm has reduced to 0.154µm after use of 200 hrs and it further reduced to 0.099µm after use of 340 hrs. This clearly shows that there is blockage of membrane pores with respect to time of its usage which has resulted in reduction of average pore size of the membrane. Similar trend has been observed in case of PVDF membrane also as shown in Fig. 7. The average pore size has reduced from 0.22µm for a brand new membrane to 0.14µm after using the membrane for 200 hrs. and further reduced to 0.094µm after use of membrane for 340hrs.

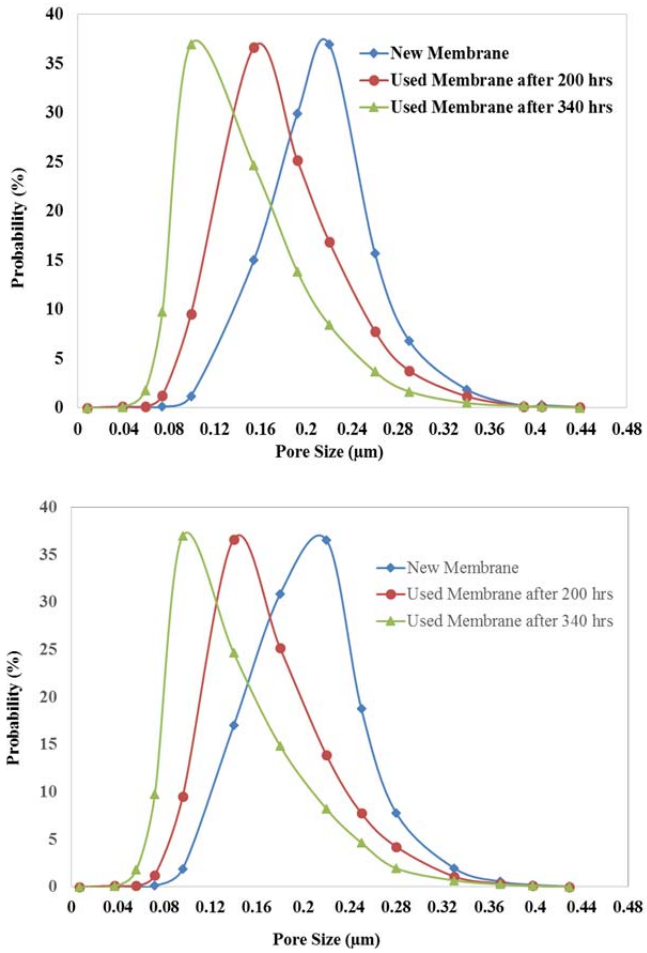


Fig. 6: Pore size distribution of PTFE & PVDF membranes before and after use respectively.

5. CONCLUSIONS

In the present work, negligible declination in permeate flux of 1.7 % and 4 % was observed for PTFE and PVDF membranes respectively of pore diameter 0.22 µm after 200 hours continuous run which may be due to the minor scaling on the membrane surface. This minor fouling was also depicted in SEM image as shown in Fig. 4 & 5. Moreover, this minor scale deposition is also supported from pore size distribution (PSD) as shown in Fig. 6 & 7 which indicate that average pore size in PSD curve shifted from 0.22 µm to 0.18 µm. At 65°C, the permeate flux were found to be 89.42 kg/m^{2}hr for PVDF membrane and 89.31 kg/m^{2}hr for PTFE membrane which remain nearly constant till 200 hours at 9.0 kPa of permeate pressure and feed flow rate of 6 lpm for pore diameter of 0.22 µm. Therefore, after every 200 hours the water washing of both membranes was done and the membrane performance was checked again and it was observed that the flux regained to about 98.76% i.e. 88.32 kg/m^{2}h for PVDF and 98.39% i.e. 87.88 kg/m^{2}h for PTFE membrane under the same process conditions with 99.9% salt rejection.

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