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Characterization and Performance Study of DCMD in Different Configurations after Membrane Thermal Treatment

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A B S T R A C T

The current study aims to investigate the characterization and performance of membrane distillation after applying thermal treatment. The characterization was conducted in term of porosity and pore size; however, the porosity has been analyzed by two different methods; Weighing and Impregnation method, and then the standard divination was determined. The experimental performance investigations were conducted on the lab-scale DCMD setup by using RO brain (TDS = 40 g/l). The tests were carried out on DCMD setup for different MD module configurations (Single MD module, two parallel modules, two series modules and package density of hollow fibers in a single module) to investigate the performance of MD in terms of flux and conductivity. Moreover, the time of operation was also analyzed. Three MD modules were fabricated by PVDF hollow fibers, and two of them were thermally treated for 24 hours at 110°C. 150°C. Minerals analysis were conducted on permeate solution by Total organic carbon (TOC), Ionic chromatography (IC) and Inductively coupled plasma - optical emission spectrometry (ICP-OES). It was indicated that MD module, which was thermally treated at 110°C shows fewer minerals quantity over other modules; thus, this module has potential in minerals recovery. It was discovered that applying thermal treatment in MD PVDF hollow fibers increase the porosity, pore size and mass the flux which, increased by 29.6%, 27.5% and 23.6% respectively.

1. Introduction:

To conquer the shortage of pure water and to implement the naturebased solutions, seawater desalination technologies have been developed by researchers from the related fields (UNICEF, 2015) and the objective from seawater desalination plants is to produce pure water with lower salinity (normally less than 500 mg/l for potable water) and fewer minerals to suit the human need, and this objective is difficult to be obtained by sewage treatment process (A. Alkhudhiri, 2012).

Since RO technology is able to produce pure water with higher recovery product and requires lower energy consumption, it has been chosen to be the favorite distillation method among thermal distillation methods. Still, there are some drawbacks such as membrane fouling that negatively impact the operation, and brain solution rejection to seawater which harming the marine biology (**M. Khayet, 2011**). Based on some environmental constrains and related regulation, only few desalination technologies such as membrane distillation (MD) and gas hydrate desalination (G. Hyd) have been an interesting subject to recent researchers due to their limited impact on the environment, (**Al-Weshahi MA, 2013; Chong Wei Ong, 2019**).

Membrane distillation (MD) technology has been an interesting subject to researchers since it requires lower energy, pressure and lower operating cost compared to RO and thermal desalination technologies. This technology has been known for more than 50 years and still under continues development by researchers. MD is a thermal and membrane separation process that utilizes a specific type of membranes for different applications, such as water desalination and the pressure difference between membrane sides is recognized as the driving force for this process (**M. Khayet, 2008**).

A large number of biological, physical, chemical and physiochemical treatments have been tried for brine and produced water treatment (E. Drioli, 2015). Very fewer efforts have been

devoted to exploiting the potential of minerals recovery from this stream (G. Chen, 2015). This fact adds motivation of recovering minerals from these streams, particularly in the current scenario when the traditional mining industry is under extreme stress, and the difference between the demand and supply of some strategic elements is increasing beyond the sustainable limit (X. Li, 2014). Several studies have been conducted in MD to perform the optimum design. The optimum characteristics for the membrane include; high liquid entry pressure (LEP), low thermal conductivity, high liquid permeability, low fouling tendency, high thermal and chemical stability, high mechanical strength and high flux rate with stability (Jeganes Ravi, 2020). Controlling the permeability can be performed by altering membrane porosity, pore size, tortuosity and the thickness of the membrane. The permeability is mainly resulted in obtaining high mass flux, and the maximum flux was reported to be $102 kg/m^2 hr$ (M.M. Aljumaily, 2018). The development of the MD process is mainly achieved by enhancing heat and mass transfer principles. There are four basic configurations that are common for MD process, namely, direct contact membrane distillation (DCMD), vacuum membrane distillation (VMD), air gap membrane distillation (AGMD) and sweeping gas membrane distillation (SGMD) (Mohamad Anas A., 2019).

Different properties for the membranes have been proposed and investigated during the last 60 years. The porosity (ϵ) of the membranes is the measurement of empty void volume in the membrane, and a higher porosity percentage leads to larger pure water evaporation and higher flux rate. The porosity generally lays in between 0.3 and 0.85, and membrane pore sizes are varied in the range between 0.1 and 1.0 µm based on the desired permeate flux; however, if the pore size increases, the flux would be increased. Membrane pore size should be large to perform better flux, in contrast, the pore size should be small to allow vapor passage and prevent liquid penetration.

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Therefore, the optimum size of membrane pore is to be determined based on each operation condition (**El-Bourawi MS, 2006**). It was reported that applying the thermal treatment on PVDF membrane enhances the performance of the membrane (**Hideo Horibe, 2013**; **Young-Jin Kim, 2010**). The enhancement was recognized in terms of mechanical properties (such as; elongation at break and tensile strength), however, it was not carried out in terms of characterization and performance with different configurations. In the current work, a characterization for PVDF hollow fiber membrane was conducted in term of porosity and pore size after applying thermal treatment, and experimental study was performed on direct contact membrane distillation (DCMD) to investigate the MD performance in a different configuration.

2. MATERIALS AND METHOD

2.1. Hollow fiber MD Module

Three typical membrane modules were fabricated with 0.5 inches (12.5mm) UPVC Class 5 type, tube pipes and fittings. The effective length of the hollow fiber is 20 cm, and the effective module's membrane area is $7.536 \times 10^{-3} \text{ m}^2$. Tee connections were fixed at the ends of the pipe by related glue at room temperature, sealed with liquid epoxy and then dried for overnight. Two PVDF hollow fibers were thermally treated in different temperature (110 and 150°C) in Drying and Heating Chamber for 24 hours, and one MD module was fabricated without any thermal treatment (Raw).



Figure 1: MD module: A) Module photo, B) Out-In configuration, C) PVDF membrane photo.

The selected membrane hollow fibers membrane was made in, South Korea by Econity Co., Ltd. However, each MD module consists of 10 PVDF hollow fibers with the following specifications: Mean pore size is $0.1 \,\mu$ m, Membrane tensile strength is 14.2 MPa, Nominal inner diameter of the fiber id 0.75 mm and Nominal outer diameter of the fiber 12.5 mm. Fig. 1 shows the current MD module, PVDF hollow fiber membrane and feed-permeate flow movements across the membrane.

2.2. Method

The current study was conducted on the lab-scale DCMD setup by using RO brain (TDS = 40 g/l), however, the study includes membrane characterization; PVDF hollow fiber porosity by two different methods and the pore size was determined after applying the thermal treatment on the membrane. Moreover, MD performance tests were carried out on DCMD setup for different MD module configurations (parallel, series and package density of hollow fibers in the module) and time of operation to investigate the performance of MD in terms of flux and conductivity.

2.3. DCMD Characterization

2.3.1 Porosity

Many different methods have determined membrane porosity (ϵ) during previous researches (**Joseph D. Menczel, 2008**), such as liquid-liquid or gas displacement method, molecular weight method, X-ray scattering method and field emission scanning electron microscopy. These methods have been utilized based on the required accuracy, ease of use and the required materials for the selected method. Two different methods determined the porosity for PVDF hollow fiber in this work, Weighing method and Impregnation method, comprehensive details for each method are presented below. In weighing method (**Peng Wu, 2018**), PVDF hollow fibers from different thermal treated temperature (RAW, 110°C, and 150°C) were cut to small length to be weighted by a digital balance. Five different samples from each PVDF hollow fiber were cut to 20 mm (2 cm)

length by a science lab blade, and the length was measured by Digital Vernier Caliber with for accurate results. The Digital Vernier Caliber has the following specification: 40 mm Jaw size, Up to 150 mm length measuring capacity and 0.01 mm resolution. High-resolution pan digital balance with the following specifications is employed to weight the mass of each five samples from the different thermal treated hollow fibers: 0.1 mg Repeatability, 320 g Capacity, 90 mm Pan size. The mean value and standard deviation for the five samples from each thermal treated hollow fiber are calculated based on equations (1) and (2) respectively:

Mean

$$= \frac{1}{N} \sum_{i=1}^{N} x_{i}$$
(1)
$$\sigma = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (x_{i} - Mean)^{2}}$$
(2)

Where *Mean* is the mean value of the five PVDF samples, *N* is the number of the samples, which is five in our case, x_i is the mass value of each sample and σ is the standard deviation. By weighing method, the porosity of the hollow fibers is determined by equation (3):

$$\varepsilon = 1 - \frac{V_s}{V_m}$$
$$= 1 - \frac{m/\rho_s}{V_m}$$
(3)

Where V_s is the volume of solid PVDF polymer in (cm³), V_m is the volume of the membrane in (cm³), m is the mass in (g), and ρ_s is the density of solid polymer in (g/cm³). Hence, V_m is calculated by equation (4):

$$V_m = A \cdot L = \frac{\pi}{4} \left(r_2^2 - r_1^2 \right) \cdot L \tag{4}$$

Where r_1 and r_2 are the internal and external radius of the membrane, respectively. A is the area of hollow fiber in (cm^2) , L is the length of PVDF sample (cm), which is 2cm in our case. Impregnated method (X. Li, 2008) is used to determine the mass of membrane hollow fiber by measuring the weight of the hollow fiber in dry condition. After that, the hollow fiber is submerged in liquid Kerosene for 48 hours to be absorbed by the hollow fiber. Then, its weight was measured in wet condition, and the difference between both conditions was subtracted. In the impregnated method, the mass is measured for PVDF hollow fiber as indicated in the previous study (X. Li, 2008), in which PVDF hollow fibers samples from different thermal treated temperature (RAW, 110°C, and 150°C) were cut into 50 mm length by a science lab blade. Digital Vernier Caliber measured the length for accurate results. Both sides of each PVDF sample were sailed by liquid epoxy to prevent liquid solution from entering through hollow fiber two opening sides. The samples were left to dry for six hours in a thermal oven at 40°C. After measuring the weight for all samples in dry condition, they were put in Kerosene tubes and left for 48 hours. Each sample was left in a separate labelled tube to avoid mixing them. After that, all samples were removed from Kerosene tubes and before measuring the weight of PVDF hollow fibers in wet state, they were laid on soft papers for 5 seconds to remove the residual drops of Kerosene. By the impregnated method, the porosity of the hollow fibers is determined by equation (5).

$$\varepsilon (\%) = \frac{(W_w - W_d)_{/\rho_w}}{(W_w - W_d)_+ \frac{w_d}{\rho_p}}. (100)$$
(5)

Where, W_w is the PVDF polymer weight in wet condition in (g), W_d is the PVDF polymer weight in the dry state in (g), ρ_w is the density for the wet solution, ρ_p is the density for hollow fiber. The density for Kerosene and PVDF is 0.82 g/cm³ and 1.77 g/cm³ respectively.

2.3.2 Pore Size

The mean value for pore size *rm* is calculated by *Guerout Elford Ferry* equation (6) (**Lwazi Ndlwana, 2020**).

$$rm = \sqrt{\frac{(2.9 - 1.75\varepsilon)8\mu lQ_p}{\varepsilon A\Delta P}}$$
 where μ

 $= 8.9 \times 10^{-4} Pas for water$ (6)

The flow is assumed to be a piston in the membrane fiber. Here, μ is the dynamic viscosity for the water in (Pas), l is membrane thickness

in (m), Q_p is the flow rate for permeate solution (m³/s), A is the effective area of the membrane in (m²), and ΔP is the difference in operation pressure in (Mpa).

2.4. DCMD Performance Investigation

2.4.1 Using Two MD Modules

MD performance tests were carried out on DCMD setup for MD modules with thermal treatment temperature at 110°C. The tests were conducted at different module configurations (single module, two parallel modules and series modules). One MD module was fabricated with 20 PVDF hollow fibers membrane to investigate the effect of package density of hollow fibers in the module on DCMD performance in terms of flux and conductivity. Moreover, the fabricated modules (Raw and 150°C) were also tested at the same parameters. The cooler was set at 15°C during all experiments, and the heater was set at 70°C. Fig. 2 illustrates the connections for parallel and series configuration, hence the single module is shown in Fig.1. The flux is calculated based on equations (7), and the rejection is calculated based on equations (8) and (9) respectively (**Peng Wu**, **2018**).

$$J = \frac{\Delta m}{\Delta t \ A} \tag{7}$$

$$R_s = 1 - \frac{C_p}{C_f} \tag{8}$$

$$C_p = \frac{C_2 V_2 - C_1 V_1}{V_2 - V_1} \tag{9}$$

Where, J is the mass flux in (kg/m².hr), Δm is the mass in (kg), Δt is the time in (hr), A is the membrane effective surface area in (m²), R_s is the salt rejection, C_p and C_f is the concentration of permeate and feed solution respectively, C_1 , C_2 and V_1 , V_2 are the concentration of the salt and the volume of the permeate tank respectively at the required time.



Figure 2: DCMD performance for two modules in: A) Parallel and B) Series connection.

2.4.2 Using Two MD Modules for a long time

In the current test, two typical MD modules were connected in a parallel connection, as explained in the previous section to investigate the effect of operating the DCMD for 24 hours on its performance. However, the temperature and flow rate for feed and permeate solution was kept constant during the entire experiment. All parameters for feed and permeate flow such as mass, pressure, temperature, flow rate and conductivity were recorded and saved automatically through PC memory, which makes results analysis much easier.

2.5. Minerals Analysis for Permeate Solution

the analysis of permeate solution quality is considered as an important part of the current study. The permeate solution was analyzed by three different methods to measure the minerals in the solution. The three methods are namely, Total organic carbon (TOC) from Elementar Company, model Vario, made in Germany, with a temperature range up to 1200°C, Ionic chromatography (IC) from Metrohm Company, model 850 Professional IC, made in Switzerland and Inductively coupled plasma - optical emission spectrometry (ICP-OES) from Agilent Technology, model ICP-OES 720, made in the USA.

3. RESULTS AND DISCUSSIONS

3.1. DCMD characterization

3.1.1 Porosity

As illustrated in Table 1 and Fig. 3, porosity results for both methods show similar results with a standard deviation of less than 0.26. This deviation between both methods are based on the human error, and the accuracy of cutting hollow fibers samples by science lab blade, hence impregnated method provide more realistic results due to unavailability of human factor by using this method.

Fable 1.	PVDF	porosity	results
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Module	Porosity (%) By Weighing Method	Porosity (%) By Kerosene Impregnated Method	Mean	Standard Deviation	
RAW	51.06	51.91	51.49	0.25	
110°C	66.8	67.28	67.04	0.08	
150°C	54.25	54.82	54.54	0.11	

From the previous results, applying thermal treatments on PVDF hollow fibers increases the porosity by about 29.6 % by comparing the results of Raw and 110°C module; however, the porosity increased by increasing the thermal treatment temperature until 110°C, then it started to decrease. Porosity is reduced after thermal treatment at 150°C for 24 hours, which may be related to the increase of fiber diameter by the membrane shrinkage. Overall, applying thermal treatment in MD PVDF hollow fibers leads to an increase in the porosity, and this is due to decreasing the density of PVDF polymer after thermal treatment.



Figure 3: Summary for porosity results.

3.1.2 Pore Size

The mean value for pore size(rm) is calculated by equation (6), and the results are presented in Fig. 8. Where, the membrane thickness (l) is 4.5×10^{-4} m, the flow rate for permeate solution(Q_p) is 9.17×10^{-6} m³/s, the effective area of the membrane (A) is 7.536×10^{-3} m² and is the difference in operation pressure (ΔP) is 0.5Mpa. As illustrated in Fig. 4, it was indicated that the pore size for PVDF hollow fiber samples was slightly increased after applying thermal treatment until 110° C (from 0.12 to 0.153 µm with 27.5% increases), then it started to decrease. Therefore, applying thermal treatment in MD PVDF hollow fibers leads to an increase in the pore size of PVDF hollow fibers. Other's experiments (**Young-Jin Kim, 2010**) have reveled enhancement in MD performance with thermal treatment.



Figure 4: PVDF pore size results.

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3.2. DCMD PERFORMANCE INVESTIGATION 3.2.1 Using Single MD Module

DCMD performance tests were performed on the fabricated MD modules (RAW, 110°C and 150°C) to investigate the effect of thermal treatment and feed- permeate flow temperature difference on PVDF membrane hollow fibers. The feed flow rate was set at 3 l/min during all experiments. The results for mass flux and rejection are presented in Fig. 5.



Figure 5: Flux / Rejection-Time curve for: A) RAW, B) 110C and C) 150C module.

Fig. 5 indicates that mass flux for RAW, 110 C and 150 C were 11.71, 14.48 and 12.62 $kg/m^2.hr$ respectively, hence, the mass flux increased by 23.6%. The rejection was at 99.9 % for both RAW and 110 C as an indication that thermal treatment enhances the performance of the membrane, in contrast, 150 C module showed lower rejection at 86.9%, other's (**Hideo Horibe, 2013; Young-Jin Kim, 2010**) have reveled enhancement in MD performance with thermal treatment.

3.2.2 Using Two MD Modules

Different module configuration on the DCMD; two modules was connected in parallel, in series and package density of hollow fibers in the module was optimized to evaluate the performance. The feed flow rate and temperature were adjusted at 3 L/min and 70°C, respectively. The summary of the numerical details for single and double MD modules is presented in Table 2. MD module with 10 hollow fibers has more average flux than the module with 20 hollow fibers, and this is due to decreasing the active surface area for 20 hollow fibers modules since the 20 fibers are grouped together and forms as a barrier for thermal exchange between feed and permeate flow, in addition, adding hollow fibers might increase the pressure drop caused by this adding. In this module, the package density has increased, but less surface area is exposed to pure water vapors to pass through. In contrast, MD modules with 10 hollow fibers have more space around each hollow fiber which makes more surface area to transfer pure water vapors to permeate side. For the two MD modules which are connected in parallel with10 hollow fibers for each has more average flux than the two modules which are in series. This is due to increasing thermal losses in the system by using two modules in series connection.

Table 2. Summary of the effect of using different modules configurations.

Module	Number of Hollow fibers	Average Flux (kg/h.m ²)	Average rejection (%)
RAW	10	11.71	99.9
110 C	10	14.48	99.9
150 C	10	12.62	86.9
110 C- 1 Module 20HF	20	12.589	97.45
110 C- 2 Parallel Modules with 10HF each	10 x 2	14.225	99.25
110 C0 - 2 Series Modules with 10HF each	10 x 2	11.7684	99.9

3.2.3 Effect of time on DCMD performance

Two typical thermal treated MD modules at 110°C were connected in parallel connection, and the DCMD was operated for 24 hours to investigate their effect on DCMD performance. Out-in feed configuration was utilized and permeate flows configuration at 3.0 *l/m* feed flow rate. The cooler was set at 15°C, and the heater was set at 70°C during this experiment. In addition, a sealed plastic feed solution tank (size 7.0 liters) was selected and filed with 7.0 liters of R.O brine water, and it was thermally insulated by 1.5-inch rubber foam thermal insulation from all sides to minimize the heat losses in the system. The results are presented in Fig. 6.

As illustrated in Fig. 6 (A), the average flux and rejection were 11.578 $kg/m^2.hr$ and 99.195% respectively, and it shows less average flux value than the same two modules for just two hours flux (14.225 $kg/m^2.hr$) for same parameters. Hence, the flux started to decrease gradually after 200 min (3.3 hours). However, these results can be validated with other's experiments (**F.A. Banat, 1998; Qtaishat, 2008**), and this is due to increasing the concentration of feed solution, reduction in water vapor pressure or membrane fouling. However, K.W. Lawson et al. (**K.W. Lawson, 1997**) reported that if the concentration of feed solution is increased, the reduction in permeate flux will be seen and this is mainly due to three reasons; The decrease in water activity in feed solution when concentration is increased, the decrease in mass transfer coefficient at feed boundary layer based on increasing concentration polarization and the decrease in heat transfer coefficient based on decreasing of membrane surface temperature.



Figure 6: Testing two KM110 MD modules in parallel for 24 hours: A) Flux and rejection, B) Feed and permeate conductivity.

Moreover, during the process, feed conductivity increased steadily as a result of transferring pure water vapors from feed solution to permeate solution and increasing the concentration of feed solution. Since our system is a close system and the rejection is added to feed tank, and the conductivity was in continuous increase with time. The conductivity increased from 60.0 mS to reach 138.7 mS after 24 hours, and permeate conductivity increased from 2.3 µS to 92.0 µS at the end of the process, hence, the curve shows linear relationships all time during the process as shown in Fig. 6 (B). However, the temperature difference between inlet and outlet remained almost constant for feed and permeate flow by average ΔT of $7^{\circ}C$ and $4^{\circ}C$, respectively.

3.3. MINERALS ANALYSIS

Total organic carbon (TOC), Inorganic carbon (IC) and Inductively coupled plasma - optical emission spectrometry (ICP-OES) methods were performed to analyze the Composition of permeate solution and measure the quantity of the minerals in that solution comparing to feed solution which is RO. Brine. Summary of the numerical details is presented in Table 3. The results show a variety in minerals composition for the different thermal treated permeate samples in same working conditions (3 l/m feed flow rate and 70°C feed temperature). Ions indicated in the table showed good rejection for all membranes, especially for 110°C, however, 150°C membrane showed better rejection for Carbon, Chloride, Selenium and Zinc than Raw membrane. In contrast, Raw membrane shows better rejection for the remaining ions. It was indicated that MD module, which was thermally treated at 110°C shows fewer minerals quantity over other modules. This module has potential in minerals recovery. The minerals results provide supportive evidence to previous investigation results which indicates that applying the thermal treatment on PVDF hollow fibers will enhance the performance of membrane distillation.

Table 3. Minerals analysis.							
ION	TEST	Unit	Distilled Water	Permeate for (Raw)	Permeate for (110 C)	Permeate for (150 C)	RO Brine
Carbon	TOC	ppm	0.00603	0.81	0.249	0.534	1.258
Chloride	TOC	ppm	0.01339	1.978	1.081	9.5	30971.4
Bromide	IC	ppm	0.044	0.046	0.044	0.15	110.8
Sulphate	IC	ppm	0.0834	0.961	0.966	4.225	4167.2
Sodium	IC	ppm	0.0475	0.873	0.28	6.5	16814.4
Potassium	IC	ppm	0.0371	0.277	0.205	0.71	532.8
Beryllium (Be)	IC	ppm	0.009077	0.008965	0.008989	0.00906	0.008858
Selenium (Se)	ICP- OES	ppm	0.03	0.02823	0.00703	0.0027	0.37671
Zinc (Zn)	ICP- OES	ppm	0.002	0.2441	0.15353	0.15538	0.275

4. CONCLUSION

The current results showed enhancement on porosity and pore size with thermal treatment which resulted in an improvement in MD performance. Connecting two MD modules in parallel will increase the mass, but there are some thermal losses in the system, which lead to decreases the flux partially. Increasing the package density for MD hollow fibers performs as a barrier which leaves less surface area to transfer pure water vapors to permeate side, which leads to a decrease in the flux. Applying thermal treatment on PVDF at 110°C shows fewer minerals quantity over other modules, which explore the potential in minerals recovery. All tests were conducted in the same operating conditions with error margin didn't exceed 5% in most experiments, however, the current assessment includes only two different thermal treatment temperatures (110 and 150°C) and other different temperatures between them need to be evaluated for more accurate results.

5. NOMENCLATURE

Symbol	Description
А	Area of the membrane
J	Mass flux for the permeate solution
rm	Membrane pore size
Δp	Pressure difference
Δm	The mass
Δt	The time
ε	Porosity
DEFEDENCES	

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