Direct contact membrane distillation for the treatment of wastewater for a cooling tower in the power industry

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Abstract

Water abstraction for cooling requirements in energy production accounts for more than 40% of the total gross abstraction in the European Union, thus driving the interest in using alternative water resources. The current study presents the application of direct contact membrane distillation for the treatment of wastewater from a flue gas desulphurization (FGD) plant for potential applications in cooling towers. Membrane distillation (MD) performance of two commercial lab-scale membrane modules (in capillary and flat configuration) was compared in terms of flux, specific energy consumption (SEC), and rejection towards non-volatiles present in wastewater samples, pretreated according to various protocols. For the flat module, two commercial membranes were tested. In all cases, a good quality permeate was produced, with stable flux over experimental time. SEC of MD for wastewater treatment, calculated experimentally, varied from 946 to 2,830 kWh/m^3 for the various applied membranes operating under different conditions. MD allows extracting more than 80% freshwater from FGD wastewater stream while maintaining high (>99.60%) rejection towards electrical conductivity.

Key words: commercial membranes, comparative analysis, membrane distillation, wastewater treatment

INTRODUCTION

Scarcity of freshwater is emerging as a big challenge in many countries across the globe. This scenario is motivating the reduction in water consumption and encouraging the application of new efficient solutions for water treatment and reuse in domestic, agricultural, and industrial sectors. Among industrial sectors, power generation is the largest consumer of water and it has been estimated that more than 40% of total water abstraction within Europe is being used by the power generation sector (Van Vliet *et al.* 2013; Vandecasteele *et al.* 2016). This strongly encourages the use of alternative water resources and water reuse practices in this field. Currently, the European Commission has funded a project on this subject entitled Materials and Technologies for Performance Improvement of Cooling Tower in Power Industry (acronym MATChING, www.matching-project.eu). In addition to many other approaches, the project also aims at applying conventional and innovative membrane

operations for the treatment of flue gas desulphurization (FGD) wastewater to be subsequently used as an alternative water source in cooling towers of power plants.

Membrane operations have attracted significant interest in the desalination and wastewater treatment sectors. In addition to the traditional pressure-driven membrane operations, relatively less explored membrane operations are attracting interest in the desalination and wastewater treatment sectors due to their specific capabilities (Drioli et al. 2015). Membrane distillation (MD) is a prominent example in this category. The process has the potential to extract high quality freshwater from highly concentrated solutions by exploiting low-grade heat (Ali et al. 2018b). In MD, a microporous hydrophobic membrane is used to retain the hot feed solution. The hydrophobic nature of the membrane ensures the retention of solution and all non-volatile components present in it and allows the passage of vapors only (Lawson & Lloyd 1997; Khayet 2011). The vapors can be collected and condensed on the other side of the membrane according to different configurations. Direct contact membrane distillation (DCMD) is the most studied configuration of MD which applies a cold permeate or distillate stream in direct contact with the membrane to condense the vapors.

Traditionally, MD has been investigated mainly for desalination applications; however, recently interest in applying MD for wastewater treatment has emerged (Shirazi & Kargari 2015). This is due to the potentially low fouling in MD compared to pressure-driven processes, ability to achieve a very high concentration factor, and excellent quality of the permeate obtained. The investigations carried out so far have mainly focused on the flux and fouling behavior (Jacob et al. 2015), scaling issues (Yu et al. 2013), and the effect of pretreatment on the process performance (El-abbassi et al. 2013).

The performance (energy consumption as well as process stability) of MD depends upon the characteristics of the applied membrane, the operating conditions, and the properties of the feed solution. The main membrane properties influencing the process performance are pore size, overall porosity, thickness, liquid entry pressure (LEP), and type of material (Khayet 2011; Eykens et al. 2016). The main operating and process variables include thermal and hydrodynamic conditions, solution concentration, and module characteristics. The constituents of feed solutions (certain ions, organics, etc.) (Gryta 2009; Yu et al. 2013) and its pH (Warsinger et al. 2015) can also influence the process stability by creating fouling/scaling issues.

The current study aims at investigating the performance of MD for the treatment of real wastewater from a FGD plant to be used as an alternative water source for cooling tower applications in the power industry. Due to abundant availability of low-grade heat at power plants, MD can be a good candidate to treat the wastewater there. The experimentation has been carried out with lab-scale membrane modules by using both a flat module equipped with two commercial polyethylene (PE) membranes commercialized by Aquastill and a polypropylene (PP) capillary membrane module from Microdyn Nadir. The effect of various pretreatments on the membrane performance has been investigated. Experimental performance of the membranes and modules has been evaluated and compared.

EXPERIMENTAL

Wastewater

FGD wastewater was provided by the Enel Produzione SpA, the coordinator of the MATChING project, in two tranches, depending on the availability. The two tranches of water have a similar composition, differing mainly in Na content.

Wastewater pretreatment

Various pretreatments were applied to the wastewater received with the final goal of making the MD treatment process smoother and stable. In the first type of pretreatment, wastewater was softened by

Na$_2$CO$_3$.H$_2$O (Na$_2$CO$_3$.H$_2$O to Ca ratio 2.6) to remove calcium that otherwise can cause scaling at the membrane and heat exchanger surfaces. The settled solids were removed by filtration using a filter paper (pore size between 25 and 60 μm). In the second pretreatment strategy, in addition to softening, acidification was performed. The objective of acidification was to further reduce scaling issues, especially in long-term runs. The third type of pretreatment included pretreatment with Na$_2$CO$_3$.H$_2$O followed by an additional pretreatment with NaOH (NaOH to Mg ratio was 1.5), to remove magnesium, and finally by acidification and addition of anti-scalant, supplied by Aquastill (8 mg/L).

Wastewater characterization

As received and pretreated, wastewater samples were characterized in terms of total dissolved solids (TDS), quantitative analysis of various ions present, electrical conductivity, and pH. TDS and electrical conductivity were measured by using the conductivity meter (Orion Star A215 pH/conductivity meter from Thermo Scientific). A pH meter (HI2300 microprocessor conductivity meter from Hanna Instruments) was used for pH measurements at room temperature. An ion chromatograph (Metrohm 861 advanced compact ion chromatograph) was applied for the quantitative measurements of various ions present in all wastewater samples. The properties of different wastewaters are provided in Table 1.

Membrane and module properties

During the first stage of experiments, a commercial membrane module from Microdyn Nadir (MD020CP2N, Figure 1(b)) was used on the first tranche of water received. Subsequently, the

Table 1 | Properties of FGD wastewater before and after various pretreatments

	TDS (g/L)	Ca^{2+} (ppm)	Mg^{2+} (ppm)	Na$^+$ (ppm)	pH	Electrical conductivity (mS/cm)
First tranche – before pretreatment	18.1	370	404	9,648	7.88	36.1
After Na$_2$CO$_3$.H$_2$O pretreatment	17.6	91	220	9,560		35.2
Second tranche – before pretreatment	16.9	384.4	289.4	7,273	6.7	33.6
Second tranche – after pretreatment with Na$_2$CO$_3$.H$_2$O and acidification	12.46	83.83	174.70	6,421	7.88	25.08
Second tranche – after pretreatment with Na$_2$CO$_3$.H$_2$O, NaOH and acidification + antiscalant	19	46.44	68.12	6,890	7.15	38.1

Figure 1 | (a) Aquastill and (b) Microdyn membrane modules used in the study.

experimentation was performed by using two flat sheet membranes provided by Aquastill. Both the membranes are polyethylene (PE) based, however, the second one (PE2) has relatively low porosity and high contact angle compared to the first one (PE1), as listed in Table 2. The flat sheet membranes are assembled in a transparent module which has spacers on both sides (Figure 1(a)). The wastewater from the second tranche was used for experimentation with both PE membranes. The details of the module and membrane characteristics for both types of modules are provided in Table 2, while the main properties of the spacers are provided in Table 3.

MD experimentation

Pure water characterization

Both the membrane modules were characterized with pure water as feed and permeate streams before the experimentation with wastewater. For the PP module, the feed inlet temperature (T$_{fin}$) varied between 35 and 55 °C while the permeate temperature was set at 25 ± 5 °C. At each temperature, the feed flow rate varied to 23, 30, and 75 L/h (corresponding to velocity of 0.064, 0.082, and 0.21 m/s, respectively). For Aquastill membranes, T$_{fin}$ was varied to 48, 60, and 70 °C while the permeate temperature was set at 30 °C. At each temperature, the feed flow rate was varied to 24, 54, and 74 L/h (corresponding to the feed velocities of 0.04, 0.09, and 0.13 m/s, respectively).

FDG wastewater treatments

FGD wastewater, pretreated according to different protocols (see the section 'Wastewater'), was treated in MD by using both modules. FGD wastewater was initially treated at feed and permeate inlet temperatures of 48 and 30 °C, respectively, by using PP and PE1 membranes. The wastewater softened with Na$_2$CO$_3$.H$_2$O was used for the experimentation with the PP membrane while the additional acidification step was performed before treating with the PE1 membrane.

MD on various pretreated wastewater samples was also carried out at high feed inlet temperature (70 °C). High temperature (>60 °C) can destroy the integrity of fibers inside the PP membrane

Table 2 | Membrane and module characteristics for Microdyn Nadir and Aquastill modules

Supplier	Membrane designation	Configuration	No. of fibers	Module length (cm)	Membrane area (m²)	MPS (μm)	LEP (bar)	CA (°)	ε (%)	δ (μm)
Microdyn Nadir	PP	Capillary	40	45	0.1	0.2	1.40	–	73	450
Aquastill	PE1	Flat sheet	–	55	0.05	0.3	>3.8	118	88	38
Aquastill	PE2 (Oleophobic)	Flat sheet	–	55	0.05	0.3	>4	>118	83	40

MPS, mean pore size; LEP, liquid entrance pressure; CA, contact angle; ε porosity; δ thickness.

Table 3 | Properties of the spacers used in flat sheet Aquastill membrane module

Material	PP
Spacer channel thickness [mm]	2.01
Spacer filament thickness [mm]	1.005
Spacer strands/m	240
Spacer angle [°]	60
Filament length [mm]	4.17
Spacer porosity [%]	78.1

module, as cautioned by the manufacturer, therefore the high-temperature testing was performed only with PE1 and PE2 flat sheet membranes.

SEC in the current study has been calculated according to the following expression:

Calculation of SEC and freshwater recovery factor

$$SEC = \frac{Q_{Tfin-Tfout}}{m_p} \tag{1}$$

where $Q_{Tfin-Tfout}$, and m_p represent the energy required to compensate the feed temperature drop within the module and the module productivity (product of flux and membrane area), respectively. $Q_{Tfin-Tfout}$ can be calculated according to the following equations:

$$Q_{Tfin-Tfou} = \dot{m}_f\, C_p (T_{fin} - T_{fout}) \tag{2}$$

where $\dot{m}_f$, C_p, T_{fin}, and T_{fout} represent the mass flow rate, specific heat, inlet and outlet temperature of the feed, respectively.

The fraction of feed recovered as the permeate has been calculated according to the following expression:

$$RE(\%) = \frac{v_p}{v_{fo}} \cdot 100 \tag{3}$$

where v_p is the permeate volume and v_{fo} is the initial feed volume.

RESULTS AND DISCUSSION

Pure water flux

Dependence of the experimental flux on feed velocity (V_f) for PP, PE1, and PE2 membranes under different feed inlet temperatures is shown in Figure 2. It is evident from the figure that flux increases by increasing T_{fin} and V_f as well acknowledged in MD literature (Ali *et al.* 2013), however, the dependence of flux on temperature is stronger compared to V_f. For PP membrane, at V_f of 0.21 m/s, the flux increases from 0.4 to 2.5 kg/m²·h when T_{fin} is increased from 35 to 55 °C. Flux of PE1 membrane, at V_f of 0.14, increases from 11.5 to 18.40 kg/m²·h by increasing the T_{fin} from 48 to 70 °C. Increase in flux
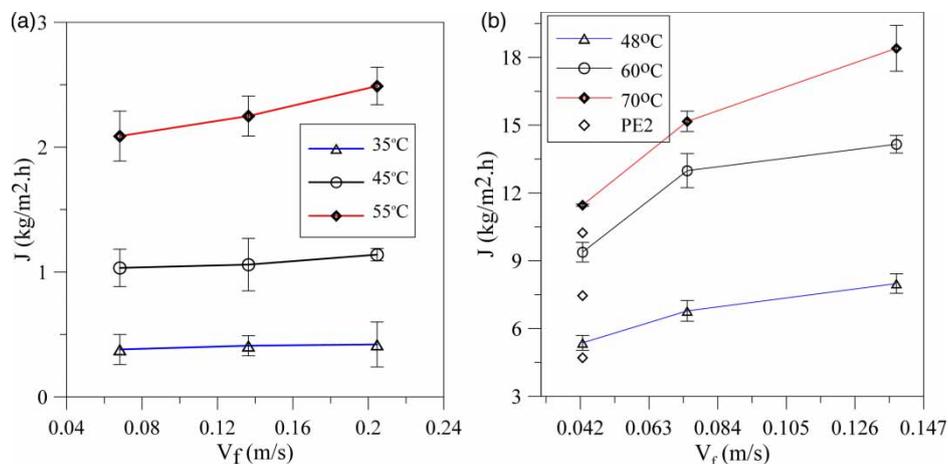

Figure 2 | (a) Pure water for PP, (b) PE1, and PE2 (diamond symbols) membranes.

with V_f is comparatively more at high feed temperatures. For instance, the flux for PP membrane increases by 10 and 25% at feed temperature of 35 and 55 °C, respectively, when V_f is increased from 0.07 to 0.21 m/s. This is due to the fact that at low feed temperature, the system is dominantly controlled by the membrane resistance and the thermodynamic vapor-liquid equilibrium (amount of vapors present at feed side), thus increase in feed flow does not significantly influence the flux (Adnan *et al.* 2012; Ali *et al.* 2013; Ho *et al.* 2014). At high feed temperature, more heat transfer takes place across the membrane through conduction and convection, thus temperature-drop along the module and temperature polarization (difference in temperature in the bulk and at the membrane surface) become high. Under these conditions, the increase in feed flow rate helps in increasing the heat transfer coefficient within the feed channel and in decreasing the temperature drop along the module, thus the flux shows more sensitivity towards flow rate (Ho *et al.* 2014). It can also be noted from Figure 2(a) and 2(b) that flux is more sensitive to V_f for PE1 membrane compared to PP membrane. For instance, at the comparable feed inlet temperatures (45 and 48 °C), the flux increases by 10 and 49% by varying the V_f values from 0.07 to 0.21 m/s and 0.04 to 0.14 m/s for PP and PE1 membrane, respectively. Quantitatively, the different response of the two membranes towards V_f could be attributed to their different characteristics (Table 2). PE1 membrane is very thin (38 μm) compared to PP membrane (450 μm) and also has high porosity. Thus, heat transfer through conduction and transport of vapors will be greater for the former which implies more temperature polarization and temperature drop along the module. Under these conditions, the contribution of liquid heat transfer coefficient in controlling the trans-membrane flux is significant. Increase in V_f increases the heat transfer coefficient at feed side and decreases the temperature drop along the module and thus the average flux will go up (Ali *et al.* 2016, 2018a). Flux for PE2 membrane at V_f of 0.04 m/s is shown with diamond symbols in Figure 2(b) and is slightly (maximum 16%) lower than PE1 membranes under all the temperature conditions considered, possibly because of its lower porosity (Table 2).

Treatment of FGD wastewater through MD

The flux as function of freshwater water recovery factor for PP and PE1 membranes operating at T$_{fin}$ of 48 °C is shown in Figure 3. For each membrane, the experiment was carried out for 4 days (~8 h each day). In both cases, the flux remains stable over the entire experimental period, with a slight loss of permeability for PE1 possibly due to increase in solution concentration which decreases the
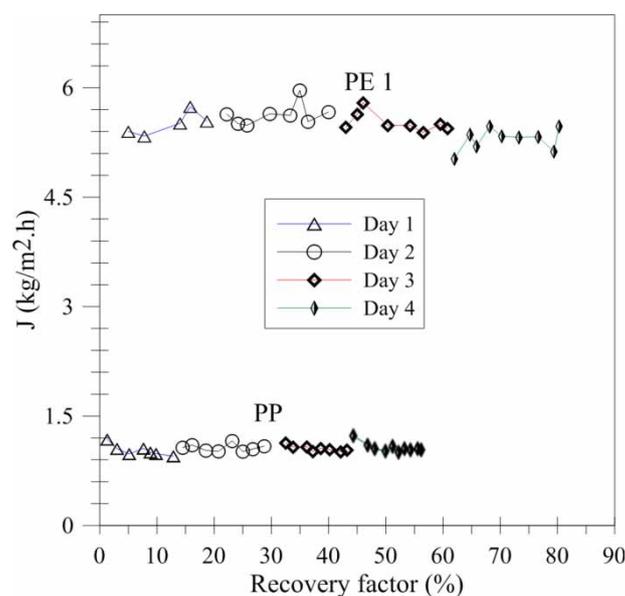

Figure 3 | Flux as function of water recovery factor for PP and PE1 membranes operating at T$_{fin}$ of 48 °C.

driving force by suppressing the water vapor pressure (Yun *et al.* 2006; Yu *et al.* 2011). Nevertheless, PE1 exhibits ~5.5 times higher flux than the PP membrane. It can be attributed to low thickness and high overall porosity of PE1 membrane (Table 2) which reduces the membrane resistance to mass transfer. In the case of PP, the experiment came to an end when the water recovery factor of ~57% was achieved due to the low feed volume available for the first tranche of FGD wastewater (the initial volume in feed tank was 5 L). For PE1, the predefined final recovery factor of ~80% was achieved by the end of day 4, as illustrated in Figure 3. The properties of the final permeate are listed in Table 4. The table shows that the membranes have good rejection (>99.50%) towards all non-volatiles present in the feed solution.

The flux for FGD wastewater, pretreated according to various protocols and treated with MD at 70 °C by using PE1 and PE2 membranes, is shown in Figure 4. It is evident from the figure that PE1 membrane shows a higher flux (on an average 16%) compared to PE2 membrane as was also evident from the pure water experiment (Figure 2(b)). A similar pattern of flux decline as a function of freshwater recovery factor for both membranes indicates that the observed decrease in flux is solely caused by the corresponding reduction in vapor pressure (and not due to the difference in membrane properties such as porosity) due to increasing solution concentration which evolves in the same way for the two membranes. The electrical conductivity of the obtained permeate was 0.0647 mS/cm, indicating the high quality of the obtained permeate. Relatively higher flux of PE1 membrane compared to PE2

Table 4 | Permeate properties for various wastewater solutions treated with PP, PE1, and PE2 membranes

Wastewater stream	Ca (ppm)	Mg (ppm)	Na (ppm)	pH	Electrical conductivity (mS/cm)	Rejection towards EC (%)
PE1-48- Na$_2$CO$_3$ softened-acidified	0 (100)	0 (100)	31.84 (99.50)	6.76	0.101	99.71
PP-48-Na$_2$CO$_3$ softened	5 (94.50)	0 (100)	217.3 (97.73)		0.0792	99.78
PE2-70- Na$_2$CO$_3$ softened-acidified	7.02 (91.63)	0 (100)	46.63 (99.27)	6.64	0.0985	99.61
PE2-70-softened (Na2CO3 + NaOH)-acidified-antiscaled	4.84 (89.53)	0 (100)	92.18 (98.66)	6.37	0.0584	99.85

Values in brackets, adjacent to concentration of each ion, refer to the percent rejection towards the ion.
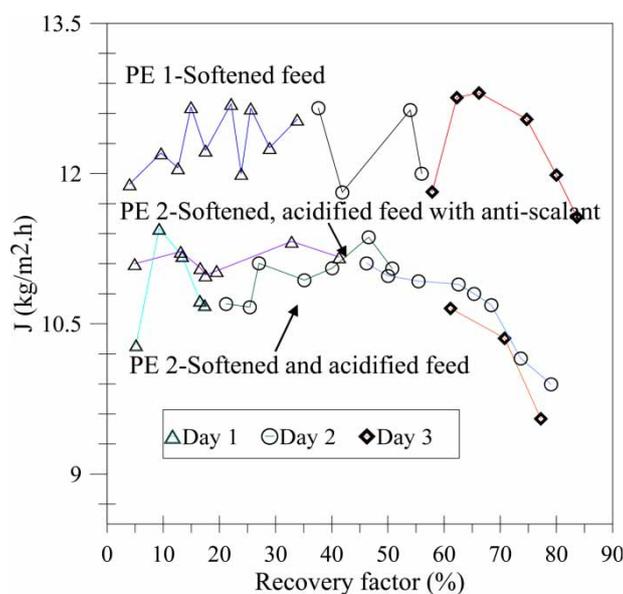

Figure 4 | Flux as function of recovery factor for different pretreated feed solutions. T$_{fin}$ and V$_f$ were 70 °C and 0.053 m/s in all cases, respectively.

membrane can be associated with its relatively high overall porosity (88%) compared to PE1 membrane (83%). However, for PE2 membrane, there was no difference in flux for softened (by Na_2CO_3) and acidified feed and softened (by Na_2CO_3 and NaOH), acidified and anti-scaled feed, as illustrated in Figure 4. Starting value of flux for any day was close to the last value of the previous day, which implies that there was no significant build-up of scaling or fouling overnight. This fact was further confirmed by the SEM images of the membrane taken at the end of the experiment, as discussed in the section below. The gradual decrease in flux after ~65% recovery factor is associated with a corresponding increase in solution concentration.

The corresponding properties for the final permeate for PE1 and PE2 membranes are provided in Table 4, which demonstrates that the membranes show a very high rejection towards all non-volatiles. Overall rejection has been greater than 99.60% in both cases. The rejection towards individual ions has also been good (>89%), as shown in Table 4. Both the flux behavior and the overall rejection suggest that the addition of the acid and anti-scalant may not be really necessary for the treatment of the wastewater considered in the current study. However, long-term experimentation is needed to further verify this observation.

Scaling/fouling analysis

Fouling analysis of the flat membranes was performed by comparatively analyzing the membrane surface with SEM before and after MD tests. The corresponding SEM images of PE1 membrane before and after MD test at 48 °C with wastewater pretreated with $Na_2CO_3.H_2O$ and acidified are provided in Figure 5(a) and 5(b), respectively. The membrane was used in the experimentation for a total of ~30 h to achieve a freshwater recovery factor of ~80% (Figure 3).

The figure illustrates that the membrane surface remains clean during the test. Several phenomena can be responsible for non-existence of scaling at the membrane surface, including short experimental time period (~30 h) (Warsinger et al. 2015), pretreatment applied to easily remove precipitating calcium salts, precipitation of calcium salts within the heat exchanger coil (observed for the same solution and membrane system in a separate experiment) where high temperature can favor the precipitation of some salts (such as $CaCO_3$), and relatively low final solution concentration which corresponds to 1,100 and 47,800 ppm of Mg and Na (solubility of these salts is 54,300 and 359,000 ppm, respectively, at room temperature). The SEM image of PE1 membrane operating at 70 °C (total experimental time and freshwater recovery factor of 22 h and 83%, respectively) on softened and acidified feed shown in Figure 5(c) also reveals that there was no visible scaling at the membrane surface.

The surface of PE2 membrane operating with $Na_2CO_3.H_2O$ softened, acidified and softened (by $Na_2CO_3.H_2O$ and NaOH), acidified and containing anti-scalant, was also scanned with SEM. Some scaling in the form of small stains over the membrane surface was observed in this case, as shown in Figure 5(d) and 5(e). The same membrane was used continuously in these two tests (no replacing elements). Thus, the observed scaling, most probably, can be associated with the extended experimental time (>45 h) period in this case as also observed in the literature (Hickenbottom & Cath 2014; Naidu et al. 2014).

Comparative analysis of thermal energy consumption

Besides testing the membrane performance in terms of process stability, energy requirements of the processes were also investigated under various operating conditions. To accurately compare the performance of the membranes, the experimentation was performed under the similar T_{fin} and V_f for all membranes. The analysis was performed by using pure water as well as the variety of pretreated wastewaters applied in the study. The overall performance was compared in terms of flux and SEC.
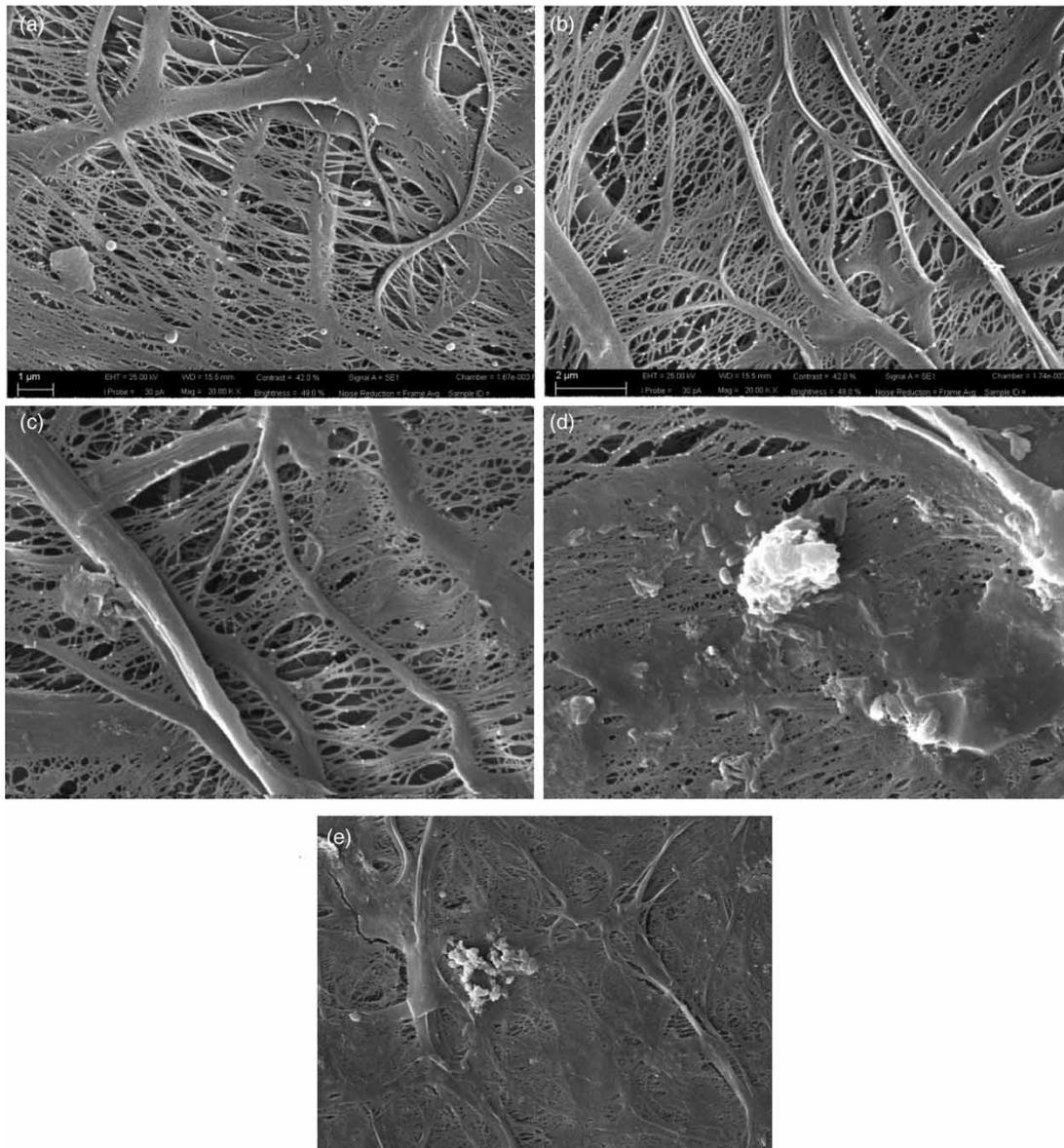
Figure 5 | PE1 (a) before and (b) after the test with pretreated (Na$_2$CO$_3$.H$_2$O softening and acid addition) wastewater (corresponding to Figure 3) at 48 °C; (c) PE1 membrane after test with pretreated (Na$_2$CO$_3$.H$_2$O softening and acid addition) wastewater at 70 °C (20,000 magnification); (d) scaling at PE2 membrane used for the treatment of softened (with Na$_2$CO$_3$.H$_2$O and NaOH) and acidified feed (containing also antiscalant) at 70 °C at magnification of 10,000 and (e) 5,000.

SEC of PP, PE1, and PE2 membrane, operating on pure water, under different thermal conditions is summarized in Figure 6(a). SEC was calculated by considering only the heat loss within the membrane module ($Q_{Tfin\text{-}Tfout}$ in Equation (1)). The figure shows that the highest SEC is observed for PP membrane under the T$_{fin}$ and V$_f$ of 48 °C and 0.042 m/s, respectively. It is evident from Figure 6(b) that the flux for PP at T$_{fin}$ of 48 °C is the lowest of all three elements. Consequently, the SEC is the highest at the lowest feed temperature considered (48 °C). On the other hand, PE1 shows 53.5% less SEC than PP under these conditions. This could be attributable to the 5.51 times higher flux of this membrane compared to PP. By increasing feed temperature from 48 to 70 °C, flux increases for both the membranes (154 and 117% for PE1 and PE2, respectively) and thus SEC drops down by 16.7% and 23.5 for PE1 and PE2, respectively. PE2 membrane shows slightly less flux and high SEC compared to PE1 membrane.
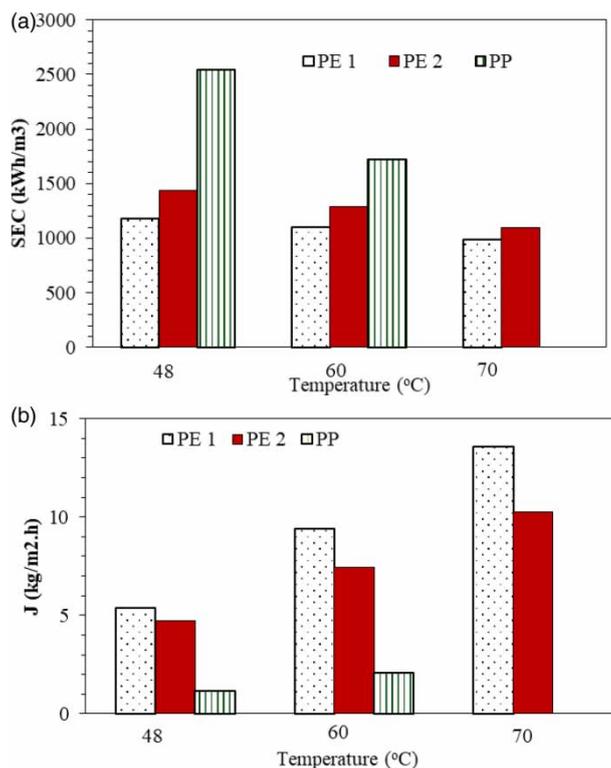
Figure 6 | Comparative analysis of PE1, PE2, and PP membranes for pure water under various thermal and hydrodynamic conditions (a) SEC and (b) flux. V$_f$ was set at 0.042 m/s.

Table 5 | Flux, SEC, and single pass recovery rate (RR, calculated by dividing the average permeate production rate with the corresponding feed flow rate) for different feed wastewater temperatures and feed velocity of 0.053 m/s

	J (kg/m^2·h)	SEC (kWh/m^3)	RR (%)
PP 48 °C	1.04	2,829.17	0.07
PE1 48	5.42	1,278.00	1.13
PE1 70	12.07	946.36	2.52
PE2 70	10	1,296.38	1.66

Miscellaneous performance parameters for the various membranes for two different temperatures, obtained by using wastewater as feed solution (first tranche for PP and second tranche for PE1 and PE2), are shown in Table 5. The parameters were calculated based on their average values over the entire experimental run. Again, the highest SEC is exhibited by PP membrane at the lowest feed inlet temperature (48 °C) considered in the study. PE1 showed the highest flux and RR (recovery rate) and the lowest SEC.

CONCLUSIONS

In the current lab-scale study, flat sheet PE membranes outperformed capillary hollow-fiber PP membrane, in terms of flux and SEC, for treatment of FGD wastewater through DCMD. Under the same operating temperature (48 °C), PE1 membrane exhibited 5.5 times higher flux and 55% less SEC compared to PP membrane. Better performance of PE membranes was attributed towards their higher porosity (>13%) and lower (>11 times) thickness with respect to PP membrane. It was observed that the performance of flat sheet membranes (PE1 and PE2) remains insensitive towards various

pretreatments applied. However, the PE1 membrane always led to higher flux and lower SEC values with respect to the PE2, due to the higher porosity. All the membranes show high rejection (>99.5%) towards all non-volatiles, confirming the efficiency of MD for the production of purified water from FGD wastewater.

Although the experimental results achieved in this work indicate a better performance of flat membranes with respect to the capillary one, some remarks need to be made about the scale-up and implementation at large scale. First of all, the properties of the capillary membrane used have not been optimized for MD applications (being the module mainly commercialized for filtration). Then, the presence of entrance/exit effects that unavoidably affect flat modules of big size, as well as the higher footprint of flat modules compared to capillary ones, are other points to be considered when moving from lab- to large-scale. A techno-economic analysis will be, therefore, at the basis of the final choice.

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