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Assessment of atomic force microscopy for characterization of PTFE membranes for membrane distillation (MD) process

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ABSTRACT

The membrane distillation (MD) process is an emerging and under-developed technique, which is currently investigated for various applications, e.g. desalination and wastewater treatment. As specific membranes for MD are not yet commercially available, most of the applied membranes for MD experiments are those microfiltration membranes made of hydrophobic polymers. Characterization of such kinds of membranes is important in order to achieve a better and clearer understanding of their performance, which helps to fabricate specific membranes for the MD process. In this work, atomic force microscopy, which is a high-resolution technique and newly applied for characterization of MD membranes, has been used for the topographical study of different polytetrafluoroethylene membranes, which are typically recommended for various MD applications. The membranes were characterized for their pore size, pore size distribution, surface roughness, and nodule aggregate. Moreover, the other two important specifications, liquid entry pressure and surface hydrophobicity, were measured and compared. A sweeping gas MD experimental setup was used for solute rejection evaluation of the applied membranes by use of four different feed samples.

Keywords: Membrane distillation (MD); Atomic force microscopy (AFM); PTFE membranes; Permeation flux; Characterization

1. Introduction

Membrane distillation (MD) has been in use for around 40 years and is currently used mostly at the lab-scale, with relatively few pilot plants in use around the world [1–3]. Details on the MD process have been widely reviewed by various researchers [1–6]. It holds the potential of being an efficient and cost-effective separation process for various purposes

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that can utilize low-grade waste and/or renewable energies such as solar, wind or geothermal which are widely available in arid regions like the Middle East and Persian Gulf regions. Compared to other membrane separation processes (MSPs), which are mostly pressure driven such as reverse osmosis (RO), nanofiltration (NF), and microfiltration (MF), MD has its own unique advantages, which could make it a preferred technique in some niche applications [7–10].

MD is a thermally driven separation process that utilizes hydrophobic, microporous membranes as a contactor. The fundamentals of MD are based on the vapor–liquid equilibria. After the feed stream is heated, it is brought into contact with the membrane which allows only the vapor phase to cross through the membrane's pores. This permeated vapor then condenses on the cold side. This vapor pressure difference is imposed by the temperature difference between two sides of the applied membrane [11].

One of the most important parts of a MD process is the applied membrane. The main requirements for the MD membrane are that the membrane must exhibit low resistance to mass transport, must not be wetted by the process liquid, must be hydrophobic, must be as porous as possible, must be as thin as possible, and must exhibit high resistance to conductive heat transfer and have good chemical, physical, and mechanical stability [4]. Since very few laboratory researches have been performed on the fabrication and/or modification of membranes prepared specifically for the MD process, most of the applied membranes are those fabricated by hydrophobic polymers and for MF purposes, such as polyvinylidene fluoride (PVDF), polypropylene (PP), and polytetrafluoroethylene (PTFE) [12–14]. The important characteristics of these commercial membranes such as thermal conductivity, chemical resistance, liquid entry pressure (LEP), hydrophobicity, and porosity have been studied extensively [12–18], however, less attention has been paid to characterization of MD membranes using atomic force microscopy (AFM) [19].

He et al. [13] used nine commercial hydrophobic membranes made of PVDF, PP, and PTFE with various pore sizes for the production of drinking water from saline water. Some characteristics of these membranes such as LEP, contact angle, and gas permeability were tested in their work. Moreover, the influence of various operating parameters as well as module design was investigated. In another work, they used the same membranes for air gap MD experiments [14]. In our previous work [12], we investigate the desalination of real Persian Gulf seawater using three commercial hydrophobic membranes (from various manufacturers) through direct contact membrane distillation (DCMD). The effect of various operating parameters and permeation flux recovery by acidifying the feed stream was investigated. Further results were obtained by characterization of these membranes using AFM, and glucose rejection tests through the sweeping gas membrane distillation (SGMD) process in a separated work [19]. Results obtained from these works indicate that the most suitable membrane for MD applications among those commercially available is the PTFE membrane. In fact, PTFE represents an ideal material for MD membrane fabricating since it exhibits one of the highest hydrophobic surfaces among polymers and also one of the best chemical resistance and thermal stability. The basic disadvantage of PTFE lies in its difficult processibility. Moreover, at the present time, commercial PTFE membranes are usually prepared through complicated extrusion, rolling, and stretching procedures [4].

As mentioned earlier, the application of the AFM method for characterization of MD membranes, and more specifically the PTFE membranes have not yet been clearly addressed. In this work, three different PTFE membranes with various pore sizes were comprehensively characterized by AFM analysis and were investigated for the SGMD process. The solute-rejection performance as well as the effect of feed type (solute type) on the permeation flux and solute rejection was investigated.

2. Experimental

2.1. Materials

Three commercial hydrophobic PTFE membranes, two of them with 0.22 μm pore size (supplied from Millipore and Chang-Qi, and named M1 and M2, respectively) and another one with 0.45 μm pore size (supplied from Membrane-Solutions, and named M3) were used for the experiments.

Glucose (BASF, Germany), sodium chloride (NaCl) (Merck, Germany), ethylene glycol (EG) (Arak Petrochemical Co., Iran), and glycerol (Fluka), all except the EG with analytical grade were used as received for SGMD experiments.

2.2. Topographical observation

AFM was performed with non-contact mode on a DUALSCOPE 95-200E AFM apparatus equipped with DS95-200 E scanner and DUALSCOPE C-21 controller (DEM, Denmark). The samples were attached to glass slides using double-sided tape. The scanning was performed in the air medium and ambient conditions. The

images were scanned using a silicon nitride probe. The specifications of the applied cantilever and its tip are presented in Table 1. The scanning was performed at a speed of 5 μm/s (1 Hz), force of 0.15 nN, and scan size of 5 μm. Phase shift was 215.4 and a sampling resolution of 300 points per line was selected.

2.3. Hydrophobicity

Hydrophobicity of the membranes' surface was measured by use of a contact angle measuring system (KRUSS G-10, Germany).

2.4. LEP

The relationship of LEP with pore size, liquid-membrane contact angle and liquid surface tension can be expressed by the Cantor equation as follows:

$$\mathrm{LEP} = \frac{2X\sigma\cos\theta}{r} \quad (1)$$

where X is a geometric factor determined by pore structure, σ is the surface tension of the process liquid, θ is the contact angle, and r is the membrane pore radius.

2.5. SGMD apparatus

A SGMD apparatus was used for the experiments. The system was equipped with a plate and frame multipurpose MD module with 0.0169 m^2 effective area for the applied membrane. A diaphragm pump was used for re-circulation of hot feed in a closed loop. An oil-free compressor provides the sweeping gas stream. Fig. 1 presents a general scheme of the applied apparatus. Further details on the applied apparatus and the SGMD experimental procedure could be found in the previous work [19].

Table 1
The specifications of cantilever and tip applied in AFM analyses

Cantilever characteristic	Value
Length (μm)	160
Width (μm)	45
Thickness (μm)	4.6
Spring/force constant (N/m)	42
Resonance frequency (kHz)	285
Slope (°)	10
Tip characteristic	**Value**
Material	Silicon nitride
Height (μm)	10~15
Tip curvature radius (nm)	10>

3. Results and discussion

3.1. Pore size and pore size distribution

Regardless of the method used in membrane fabrication (e.g. phase inversion, sintering, electrospinning, etc. [4,6,20]), characterization of such membranes could provide a better and more in-depth understanding in order to evaluate their performance in the MD process. Pore characteristics (i.e. pore structure, pore size, and pore size distribution) are the most important specifications of microporous membranes, either polymeric or ceramic [21,22]. The pore size and its distribution are two major specifications, which could directly affect the membranes' vapor transfer performance for various MD applications. In this case, the optimal range for pore size of a suitable MD's membrane is 0.1–0.5 μm [19]. Moreover, the pore size distribution should be as narrow as possible. In this work, the three investigated membranes characterized for their pore size and distribution using AFM analysis are based on the method described in the literature [23]. In order to measure the pore sizes, cross-sectional line profiles were selected to traverse micron (5 × 5 μm) scan surface areas of the AFM images that are presented in Fig. 2. The pore diameter was measured by a pair of cursors along the reference lines. The horizontal distance between each pair of cursors was taken as the diameter of the pore. The AFM software used (Dualscope™/Rasterscope™ SPM, Version: 2.1.1.2; in this work) allowed quantitative determination of pores by use of the images. Pore sizes were determined for at least 45 points on each membrane sample, and then the mean values were reported.

As could be observed in Table 2, the reported value for pore size is 0.22 μm for all membranes, whilst the measured value for M1, M2, and M3 membranes is 0.26, 0.29, and 0.51 μm, respectively. As the first result, it could be concluded that the reported pore size by the manufacturer is not completely trustable. In other words, in such cases as the MD process that the pore size is a critical specification, the pore size should be characterized using a standard method such as AFM, in which the membrane is either commercial or synthesized. One of the most weak points of the MD process is the pore wetting with process liquid(s). In such cases, the process liquid, either from the hot side or from the cold side (mostly in DCMD mode), enters the pores and consequently rejection coefficient and in more general terms, the

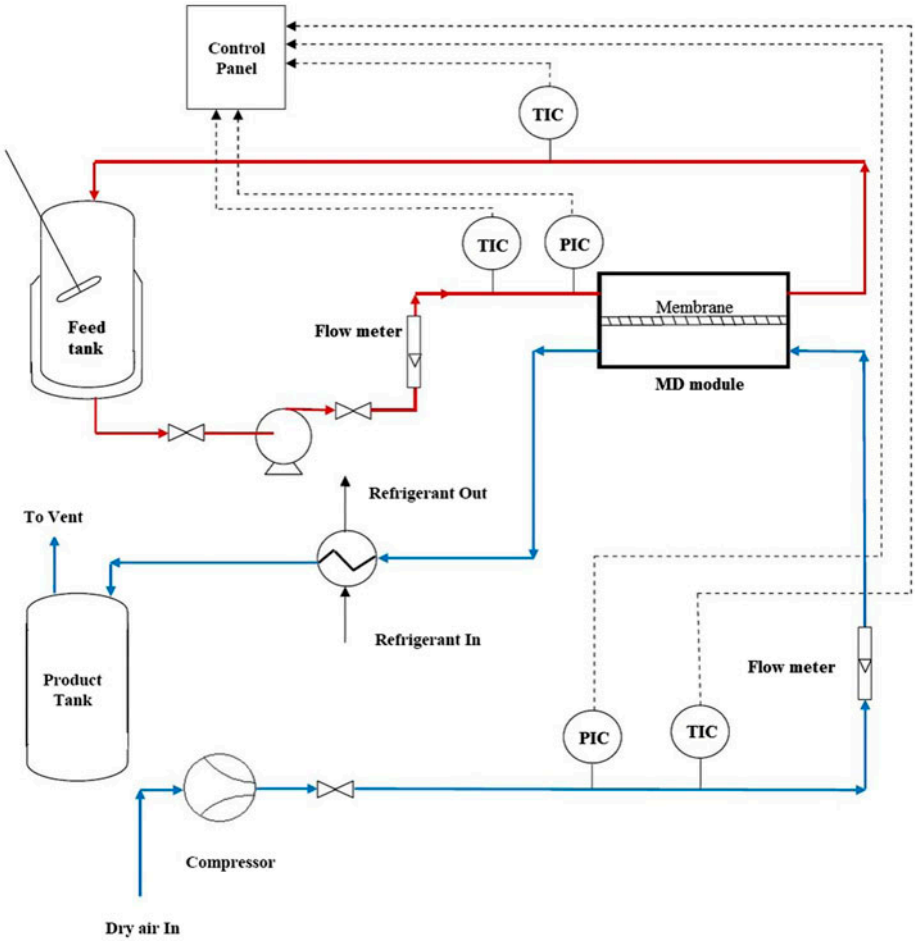

Fig. 1. The general scheme of the experimental setup.

overall efficiency decreases. Based on the obtained results, although larger pore sizes were determined, the membranes' pore sizes were in the optimum range.

Fig. 3 shows the pore size distribution of the three applied membranes. As mentioned earlier, like other MSPs, the pore size distribution of the applied membranes for the MD application should be as narrow as possible. This is one of the important parameters that shall be considered for fabricating specific membranes for the MD process. As could be observed, for the M1 membrane more than 45% of the measured pores were in the range of 0.2–0.3 μm, while for the M3 membrane about 41 and 33% of the measured pores were in the range of 0.4–0.5 μm and 0.5–0.7 μm, respectively. Regarding the M2 membrane, a wide pore size distribution was observed. The most narrow pore size distribution was observed for the applied membranes as the following arrangement, M1 > M3 > M2. This difference could be explained by the fact that various manufacturers apply different methods for fabricating the PTFE membranes; moreover, the properties of the raw materials shall be considered too.

3.2. Surface roughness and nodule aggregate

Surface roughness is also an important topographical property of polymeric microporous membranes and could be presented as average roughness (R_a), root-mean-square roughness (R_q), and/or peak-to-valley height (R_z). Surface skewness (R_{sk}) is a measure of symmetry of the height distribution. Negative skewness values correspond to the dominance of valleys, associated with the porous-like surface, whilst positive skewness values suggest that peaks dominate the surface. Surface kurtosis (R_{ku}) describes the sharpness of the height distributions. Kurtosis values lower than 3 indicate a flat and repetitive surface, while values greater than 3 suggest a sharper height distribution. These roughness parameters were estimated from

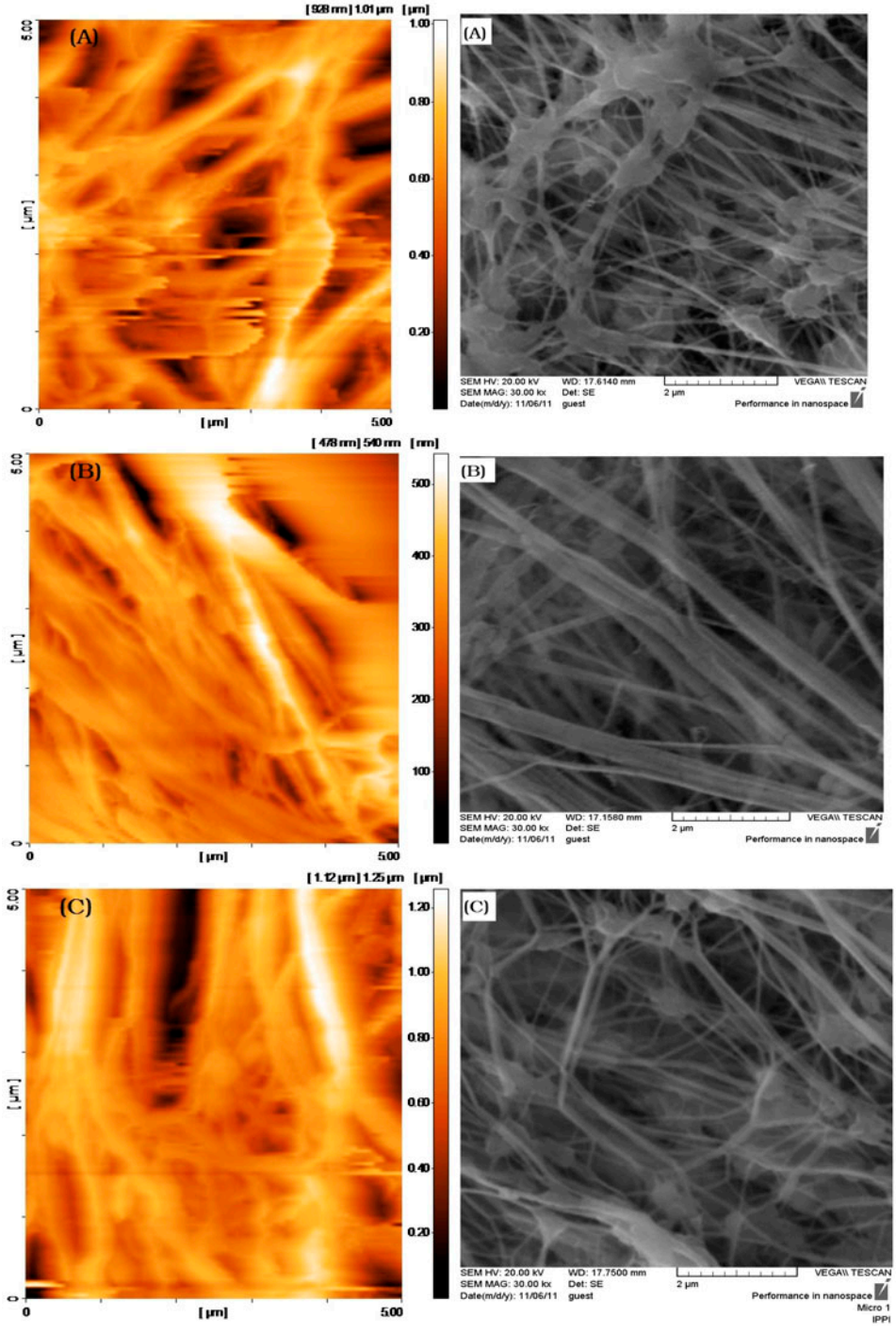

Fig. 2. AFM (left) and SEM (right) images of the investigated PTFE membranes, (A) M1, (B) M2, and (C) M3, respectively.

images scanned over an area of $5 \times 5\,\mu m$ from each sample and are presented in Table 3.

As could be observed, the average roughness (R_a), which shows the deviation in height, was higher for the M3 and lower for the M2, at 155 nm and 53.8 nm, respectively. Similar to R_a, the R_q, which represents the standard deviation of surface heights, as well as the third roughness value, R_z, were also higher for

Table 2
The reported and the characterized specifications of applied membranes

Membrane	Reported			Measured		
	Pore size (μm)	Thickness (μm)	Porosity (%)	Pore size (μm)	LEP (kPa)	CA (°)
M1	0.22	175	70	0.26	152.5 ± 0.1	132.5 ± 0.5
M2	0.22	230	80	0.29	117.72 ± 0.1	115.6 ± 0.5
M3	0.45	140	75	0.51	82.66 ± 0.1	124.4 ± 0.5

Note: CA: Contact angle (°).

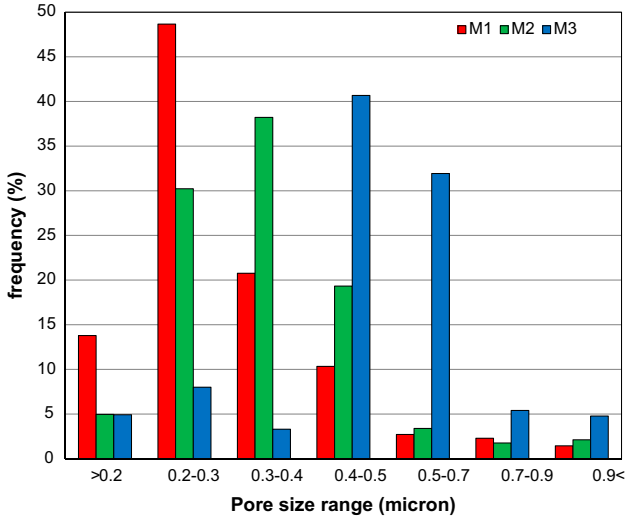

Fig. 3. Pore size distribution of applied membranes based on the AFM analysis (M1, M2, and M3).

the M3 and lower for the M2. This means that the M2 membrane had a smoother surface compared to the M1 and the M3 membranes.

On this basis, the following points could be concluded. When a polymeric microporous membrane is used in a pressure-driven MSP, such as MF or NF, lower surface roughness leads to lower fouling risk and higher surface hydrophilicity (if the polymer is hydrophilic in nature) [22]. These could be attractive findings for number of applications such as MF of biological solutions in which the applied microporous membranes should be as smooth as possible. However, in the MD application, in which the inlet pressure in the feed side is close to atmospheric pressure, higher surface roughness could be considered as an attractive characteristic. This could be explained as follows. As mentioned earlier, the MD process is a thermally driven separation process, therefore only vapor molecules are allowed to pass through the membranes' pores. Therefore, both temperature polarization (mostly when the feed contains the volatile compounds such as ethanol [10]) and concentration polarization (mostly when the feed contains the non-volatile compounds such as salt or sugar [9]) phenomena occur in the feed-membrane interface boundary layer [11]. Both temperature and concentration polarizations are insufficient phenomena that have a negative effect on the permeation flux in the MD process [2–4,24]. One of the practical solutions in order to reduce the effect of polarizations isto increase the turbulency of the membrane-feed interface (temperature and concentration boundary layers). In a constant inlet flow rate, an increase in the surface roughness could increase the turbulency (from a microscopic point of view) and consequently reduce the polarizations' effect. Therefore, membranes with rougher surface are more attractive for MD applications.

Table 3
Roughness parameters of the applied membranes based on the AFM analysis

Membrane	Roughness parameter				
	R_a (nm)	R_q (nm)	R_z (nm)	R_{sk}	R_{ku}
M1	135	168	921	−0.42	2.82
M2	53.8	70.5	489	−0.24	4.01
M3	155	200	1,150	−0.60	3.63

Skewness is the third moment of the profile amplitude probability density function and is used to measure the profile symmetry about the mean line. When the height distribution is symmetrical, R_{sk} is zero. None of the investigated membranes in this work had zero skewness value, meaning that no symmetrical profile was observed for any of the membranes investigated. In fact, if the height distribution is asymmetrical, and the surface has more peaks than valleys, then the skewness moment is positive, as could not be observed for all membranes (Table 3). On the other hand, if the surface is more planar and valleys are predominant, the skewness is negative, as was the case for the M1, M2, and M3 membranes. Moreover, it could be concluded that higher skewness

values for the M3 membrane was due to its higher porosity (Table 3).

Kurtosis moment is the fourth moment of the profile amplitude probability function and corresponds to a measure of surface sharpness. R_{ku} value of M3 represents the Gaussian amplitude distribution, and the surface is called Mesokurtic. Smaller values of R_{ku} represent the flat surface, as observed for the M1 and the surface is called Platykurtic; whilst higher values than 3 represent more peaks than valleys, as in the case of the M2. The R_{ku} value for M1 and M3 membranes were measured at 2.82 and 3.63, respectively. It is worth noting that the R_{ku} value of the M3 (the membrane with 0.45 μm) was closer to Gaussian (Mesokurtic) distribution than those of the M1 and M2 membranes, revealing the uniform structure of the M3 membrane.

AFM has the potential to provide additional resolution allowing measurement of the nodule aggregate. This analysis could yield more in-depth understanding of the topographical architectures of the MD membranes. Therefore, the PTFE membranes were analyzed for their nodule aggregate. In order to measure the nodule aggregates, cross-sectional line profiles were selected to traverse micron (5 × 5 μm) scan surface areas of the AFM images. The diameter of the nodules (i.e. height peaks) were measured by a pair of cursors along the reference lines. The horizontal distance between each pair of cursors was taken as the diameter of the nodule.

Table 4 presents the mean, maximum, and minimum values of nodules sizes based on the AFM analysis. As could be observed, the mean nodule aggregate was significantly lower for the M2 membrane; and was higher for the M3, compared to the other two PTFE membranes. The mean value of the nodule size was measured at 121, 26.8, and 148 nm for M1, M2, and M3 membranes, respectively. This nodule aggregate difference could be described based on the difference between the various fabrication methods applied by various suppliers which could be stretching [4].

Table 4
Nodule aggregate (min., max., and mean) of the applied PTFE membranes

	Nodule size (nm)		
Membranes	Min.	Max.	Mean
M1	58.0	221	121
M2	1.74	86.5	26.8
M3	6.85	283	148

3.3. SGMD efficiency test

As mentioned earlier in the literature [3], one of the most important weak points of the MD process is pore wetting during the experiments. This drawback affects various parameters such as LEP value, hydrophobicity, maximum pore size, and interaction between the process liquid and the membrane. For instance, the surface energy of the hydrophobic polymers could significantly change in the presence of alcohol, i.e. ethanol [10]. On the other hand, the hydrophobicity and the LEP value should be as high as possible for a MD membrane. These values are presented in Table 2. As could be observed, the M1 membrane had the higher surface contact angle (static contact angle between the deionized water drop and the membrane surface) among others; and consequently, the higher LEP value was measured at ~152 kPa for this membrane. On the other hand, the lower surface contact angle was measured for the M2 membrane while the lower LEP value was ~82 kPa regarding the M3 membrane. The higher contact angle and lower LEP of the M3 membrane compared to the M2 membrane could be explained as follows.

As could be observed in Eq. (1), the LEP value has reverse dependency to the membrane's pore size (r, the pore radius). Therefore, higher pore size (0.45 μm as the reported pore size and 0.26 μm as the measured pore size based on the AFM analysis) led to lower LEP value for the M3 membrane. However, it should be noted that the M3 membrane had higher surface contact angle compared to the M2 membrane. Two important points shall be investigated here. First, the LEP may be more influenced by the pore size than the contact angle. Second, the higher hydrophobicity for the M3 membrane could be expected due to its higher pore size. This could be explained based on the well-known Cassie–Baxter theory, which describes the hydrophobicity of the heterogeneous surfaces. Based on this theory, when a water droplet is placed on a porous surface, it can be held up by the trapped air in the membrane's pores. This means that the air placed under the water droplet affects the surface hydrophobicity, as the 180° water–air contact angle. On the other hand, it should be noted that if the pore size is larger (as was the case for the M3 membrane), the capillary effect overweighs the effect of air–water surface tension and consequently leads to lower LEP values (~82 kPa for M3).

A preliminary test was conducted in order to evaluate the solute rejection performance of the investigated membranes. Therefore, in this step, all membranes were used for the SGMD process with

identical operating conditions of 65˚C feed temperature, 600 mL/min feed flow rate, and 0.453 Nm^3/h sweeping gas flow rate. NaCl was used as a solute for preparation of the feed sample with 10 g/L concentration. The rejection was measured using the following equation:

$$\%\ R_s = 100 \times \left(1 - \frac{\text{Permeate concentration}\left(\text{kg/m}^3\right)}{\text{Feed concentration}\left(\text{kg/m}^3\right)}\right) \quad (2)$$

Fig. 4 shows that the M1 and M3 membranes had higher solute rejection compared to the M2, which indicated that these membranes had better performance than that of M2. However, it is worth noting that the M1 had even better performance compared to M3, which could be due to the smaller pore size of the M1 membrane. On the other hand, the M3 membrane had larger pore size than that of M2, and the achieved solute rejection was higher than M2. This can be explained by the fact that the quality of the raw material used by the supplier for fabricating the membranes as well as the applied preparation technique, can all affect both morphology and topography of the membrane and consequently its performance like solute rejection. As could be observed in the SEM images of the membranes, both M1 and M3 membranes had better and more regular morphology.

Based on the obtained results from the characterization step which is well described above, and the solute rejection efficiency test, the M1 membrane was selected for the SGMD efficiency test using various feed samples including the glucose syrup (GS) (10 g/L), NaCl solution (10 g/L) (NS), and EG and glycerol mixture (5 wt.%) (GM). The same operating conditions of 65˚C feed temperature, 600 mL/min feed flow rate, 0.453 Nm^3/h sweeping gas flow rate, and the cross-current flow arrangement inside the MD module with 2 mm channel depth for both feed and permeate sides were used for experiments.

Fig. 5 presents the variation of the permeation flux versus operating time for various feed samples. As could be observed, the permeation flux was decreased for all feed samples. The higher and lower permeation flux was achieved for GS and aqueous GM, respectively. Moreover, both glucose and salt solutions achieved higher permeation flux compared to the EG and GMs. This was due to the significant higher affinity of these chemicals (EG and GM) to the water, which lead to harder dewatering.

As a comparison, the experiment achieved higher permeation flux by use of salt solution during the first 50 min compared to the test conducted by GS; however, after almost 60 min, the SGMD had the same behavior for both feed samples (GS and NS).

Having compared the EG and GM feed samples, it could be observed that the permeation flux obtained for the EG feed was significantly higher than that of GM feed. This result could be explained by the fact that the glycerol has three –OH groups in its chemical structure that significantly increase its affinity to the water. Therefore, water removal of GM solution had lower efficiency compared to dewatering of the EG feed sample.

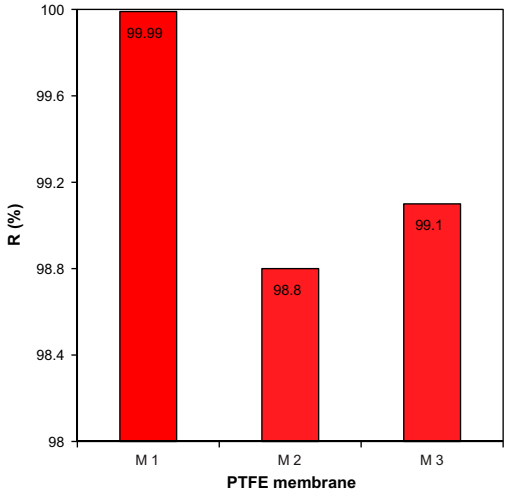

Fig. 4. Solute-rejection evaluation of the applied PTFE membranes (65˚C feed temperature, 600 mL/min feed flow rate, and 0.453 Nm^3/h sweeping gas flow rate).

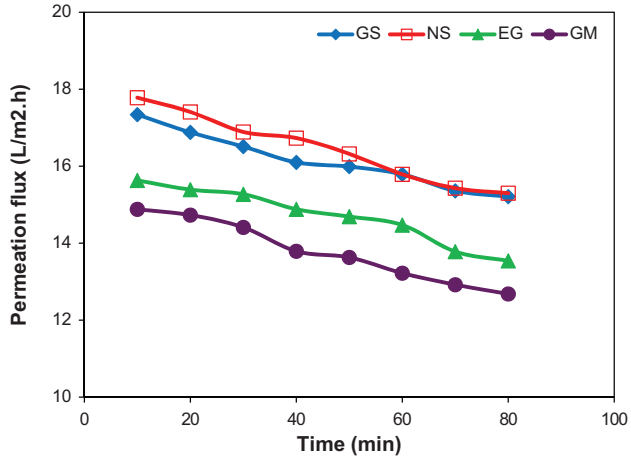

Fig. 5. Permeation flux for various feed samples versus operating time (65˚C feed temperature, 600 mL/min feed flow rate, and 0.453 Nm^3/h sweeping gas flow rate).

4. Conclusions

Typically, PTFE membranes have been used for various MD applications due to their better performance, e.g. higher hydrophobicity, higher LEP, higher solute rejection, etc. However, these PTFE membranes are fabricated by various suppliers and consequently have different characteristics. Therefore, after concluding that the PTFE membranes are the first choice for the MD process, the most suitable and better characteristics for a PTFE membrane should be studied. In fact, a comprehensive comparison between various specifications of various PTFE membranes, e.g. pore size, pore size distribution, nodule aggregate, solute rejection, and permeation flux in the presence of different feed samples could open new windows for selecting the most suitable one for the MD application. In this case, the AFM technique could act as one of the most suitable methods.

AFM could provide various roughness parameters; therefore, the users could select the rougher membranes because higher surface roughness has a positive effect on the MD process performance due to reduction of the effect of temperature and concentration polarizations. Moreover, AFM could provide the skewness and kurtosis parameters as well as nodule aggregate in which more in-depth understanding of the PTFE membranes' performance could be achieved. Having these characteristics obtained by AFM, the prediction and discussion on the obtained experimental results for the MD process are more feasible and more practical results could be concluded. Overall, the AFM is proven to be a powerful and high-resolution method for studying and characterizing the MD membranes, especially PTFE ones.

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