Development of Porous Asymmetric Polyamide–Imide Torlon® Membranes for Physical CO_2 Absorption and Separation

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Abstract: Porous flat-sheet polyamide–imide (PAI) membranes were prepared *via* a phase inversion method to evaluate CO_2 absorption performance in the gas-liquid membrane contactors. Different amounts of polyethylene glycol (PEG-600) were introduced into the polymer solution to investigate the structure and performance of resulted membranes. The membranes were characterized in terms of gas permeation, contact angle measurement and CO_2 absorption flux. By introducing 6 wt.% PEG into the polymer dope, N_2 permeance of the membrane was significantly improved from 482 to 1320 GPU. Mean while, the effect of PEG on the measured water contact angle was in significant. From CO_2 absorption test, the developed membrane presented about 90% higher CO_2flux compared to the plain membrane at water flow rate of 70 ml/min. In conclusion, by introducing a polymeric non-solvent additive into the polymer dope, it is possible to enhance surface porosity (permeability) of PAI membranes, which is a key factor for CO_2 absorption test.

Keywords: Polyamide-imide membrane, non-solvent additive, CO_2 absorption, membrane contactor.

INTRODUCTION

Due to environmental, operational and economic impacts of acid gases such as CO_2, H_2S, NO_x and SO_x, development of new separation technologies with enhanced efficiency and low capital cost is an emerging demand in the near future. Separation of CO_2 from the gas streams has been a main challenge for a long time owing to its greenhouse effect and corrosive behavior. So far, several separation technologies like distillation, adsorption and absorption using large columns have been developed for CO_2 capture. It should be noted that the separation technologies with a small footprint and high removal efficiency such as membrane technology have attracted considerable attentions recently.

Gas absorption through membrane contactors is a promising alternative for CO_2 capture which have been applied since 1980s [1,2]. Several membrane materials, membrane modules, operating conditions and liquid absorbents have introduced in the open literature for CO_2 absorption by membrane contactors [3-10]. In membrane contactors, a non-dispersive gas liquid contact happens *via* a porous membrane. Using hollow fiber membranes in a module as a suitable configuration, fluids can be contacted on the opposite sides of the membranes. The interface between the gas and liquid phase is formed at the mouth of the membrane pores. Mass transfer occurs across the gas-liquid interface just as in conventional contacting devices. In comparison with traditional contacting devices such as absorption columns, membrane contactors offer several advantages like high surface area per unit contactor volume, independent control of gas and liquid flow rates without any operational problems, small size, modular and being easy to scale up or down [11-13]. It should be noted that the membrane material and structure play important roles on the separation performance of the membrane contactor system [14]. In gas-liquid membrane contactors, essential properties such as high surface porosity, high hydrophobicity and small pore sizes are required to enhance the separation performance. Hence, several factors such as different non-solvent additives, various polymeric materials and fabrication parameters have taken into consideration for hollow fiber membrane development. From the studies of Mansourizadeh *et al*. [15], it was found that strong non-solvent additives can enhance phase inversion rate of polyvinylidene fluoride (PVDF) and polysulfone (PSF) solutions to produce high surface porosity membranes. In addition, by using a mixture of solvent and water as a weak bore fluid, the hollow fiber membranes presented inner skinless layer which minimize the membrane resistance for CO_2 absorption [16-18]. Bakeri *et al*. [9] investigated the effect of polyetherimide (PEI) concentration in the spinning dope on the structure and performance of the membranes for CO_2 absorption.

Polyamide-imide (PAI) is a very attractive polymer for membrane preparation due to the presence of amide and imide groups of atoms in the main chain. Chemical structure of PAI is shown in Figure **1**. Indeed,

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the aromatic imide units of PAI provide high performance properties such as significant mechanical, thermal and chemical stabilities, while the flexible amide groups provide a good process ability for membrane preparation [19]. Since PAI (Torlon®) is a relatively new commercial polymer, the research on this material for porous membrane preparation specially membrane contactors are limited in the open literature. Recently, surface modification of PAI membranes for membrane contactor application investigated by Zhang *et al*. [20]. The modified membrane showed about 30 higher contact angle than the plain membrane which showed a potential for gas-liquid membrane contactor application. In present study, an attempt was made to develop PAI membrane structure for gas absorption *via* a phase inversion method. Mean pore size and surface porosity of the membranes were examined through a gas permeation method. Surface hydrophobicity of the membranes was evaluated by contact angle (CA) measurements. The CO_2absorption performance of the membranes was examined through the prepared gas-liquid membrane contactor system.

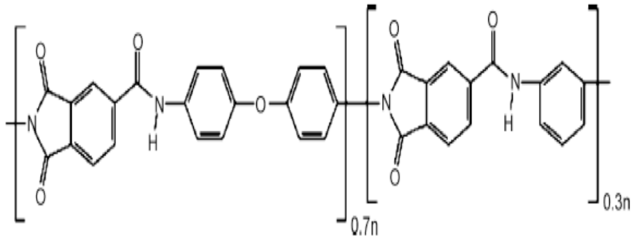

Figure 1: Chemical structure of polyamide-imide [21].

EXPERIMENTAL

Materials

Commercial polyamide-imide (Torlon® 4000T-MV)powder was supplied by Solvay Advanced Polymers. 1-Methyl-2-pyrrolidone (NMP, >99.5%) was provided by Merck and used as the polymer solvent. The polymer powder was dried in a vacuum oven at 60 ºC for 24 h before preparing the polymer solution. Pure polyethylene glycol-600 (Merck) was used as non-solvent additive in the polymer solution to enhance phase inversion process. Methanol and n-hexane (Merck) were used to post-treat the fresh membranes. Sodium hydroxide (NaOH) (>98%) pellets were supplied by Merck and used to prepare 0.05 M solution for chemical titration process. The liquid absorbent used in the membrane contactor module for CO_2 absorption was distilled water.

Preparation of Asymmetric Membranes

To prepare the polymer solution, a desire amount of PEG was dissolved in NMP following by gradual addition of the polymer powder. A homogeneous polymer solution was achieved using constant stirring at 60 ºC. After addition of 0, 3 and 6 wt.% of PEG-600, the polymer solutions were labeled as M1, M2 and M3, respectively. Prior to casting the membranes, the solutions were degassed using 30 min. sonication in an ultrasonic bath, and then kept overnight at room temperature. The membranes were cast over a glass plate using a doctor blade at constant speed of 1.7 cm/s. Then the cast film along with the glass plate was gently immersed in the coagulation bath which was tap water in this case. After 1 h of coagulation, the membranes were removed from the coagulation bath and immersed in distilled water for 2 days to remove all residual NMP and PEG from the membrane matrix. For post-treatment, the fresh membranes were submerged in methanol and n-hexane (each for 15 min.), respectively. Post treatment was conducted to prevent collapsing the pores of the membranes before drying at ambient temperature.

Characterization of Prepared Asymmetric Membranes

To study mass transfer through a porous membrane for gas absorption, pore size and surface porosity are the key parameters. The combination of Poiseuille and Knudsen flow has been used to correlate total gas permeation rate of the porous asymmetric membranes [22]. The gas permeance can be calculated by the following equation when assuming cylindrical pores in the skin layer of the asymmetric membranes:

$$J_A = \frac{2}{3}\frac{r_p \varepsilon}{RTL_p}\left(\frac{8RT}{\pi M}\right)^{0.5} + \frac{r_p^2 \varepsilon}{8\mu\ RTL_p}\ \bar{P} \quad \text{or}$$

$$J_A = K_0 + P_0 \bar{P} \tag{1}$$

Where J_A is the gas permeance (mol/m^2 s Pa); r_p and L_p are pore radius and effective pore length, respectively (m); ε is surface porosity; R is gas constant 8.314 (J/mol K); μ is gas viscosity (kg/m s); M is gas molecular weight (Kg/mol); T is gas temperature (K); and $\bar{P}$ is mean pressure (Pa).

Based on Eq. (1), by plotting $J_A$versus mean pressures, the mean pore size and effective surface porosity over pore length, ε/L_p, can be obtained from the intercept (K_0) and slope (P_0) of the line as following equations:

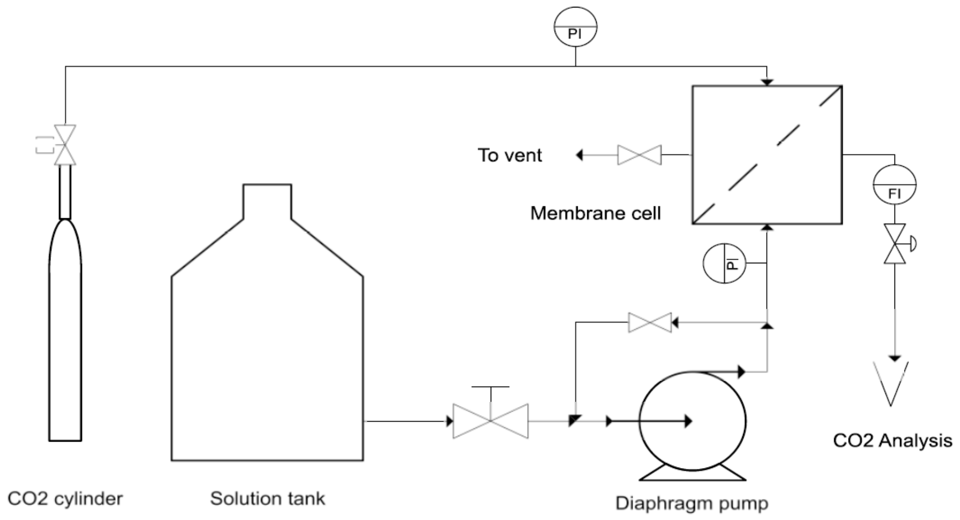

Figure 2: Schematic of experimental CO_2 absorption set up.

$$r_p = 5.333\left(\frac{P_0}{K_0}\right)\left(\frac{8RT}{\pi M}\right)^{0.5}\mu \tag{2}$$

$$\frac{\varepsilon}{L_p} = \frac{8\ \mu\ RT\ P_0}{r_p^2} \tag{3}$$

The feed gas was applied in the gas permeation experiment was pure nitrogen. The test apparatus was based on the constant pressure method. The circular flat sheet membrane with effective area of 1.25×10^{-3} $m^2$was placed and sealed into the membrane module to measure gas permeability. Using a regulator, the feed gas pressure was increased by 20 kPa intervals from 80 to 200kPa. Using a soap-bubble flow meter, the N_2 permeation rate at 25 $^\circ$C was measured. N_2permeance was calculated based on surface area of the membrane at each pressure interval.

As for contact angle (CA) measurement, the membrane sample was dried in a vacuum oven at 60 $^\circ$C for 12 h. The Force Tensiometer (KRÜSS) equipment was used to measure contact angle of outer surface of the membranes. By setting water droplet size of 0.2μl and drop rate of 0.5 μl/s, the droplets were placed one by one at various positions of the outer surface. The mean value of contact angle for left and right sides of each drop let was measured as a function of time by the equipment. For each membrane sample, the mean CA value was obtained by averaging the data at ten various positions.

CO_2 Absorption Experiment

In order to measure CO_2 absorption flux of the flat membranes, a membrane contactor system with a semi-batch operation was fabricated. The flat membranes with effective area of 1.25×10^{-3} m^2 were sealed in the membrane module. Pure CO_2 was used as the feed gas and pressurized at 100 kPa in the membrane module. Distilled water was used as the liquid absorbent and flowed continuously through the module to measure CO_2 absorption flux. Distilled water was flowed on the outer surface of the membrane in contact with the skin layer to minimize wetting. To prevent bubble formation in water during the absorption test, water pressure was set 20 kPa higher than the gas phase pressure. Using a chemical titration method, CO_2 concentration in water at the outlet of the module was measured to determine the CO_2 flux at various flow rates. Prior to take samples, all the experiments were conducted for 30 min to achieve a stable condition. The schematic of semi-batch operation used for CO_2 absorption is shown in Figure **2**.

RESULTS AND DISCUSSION

Characteristics of Prepared Membranes

In present work, an attempt was made to prepare structurally developed PAI membranes for membrane gas absorption application. The prepared membranes were characterized in terms of gas permeation and CA measurements which are the key factors for gas separation in gas-liquid membrane contactors.

The correlation of N_2 permeance of the membranes with mean pressure is shown in Figure **3**. As can be seen, the solid lines indicate the best linear fit to the experimental data. According to Eqs. (2) and (3), the intercept and slope of the lines were used to estimate the mean pore size and effective surface porosity of the membranes. The obtained results for pore size and

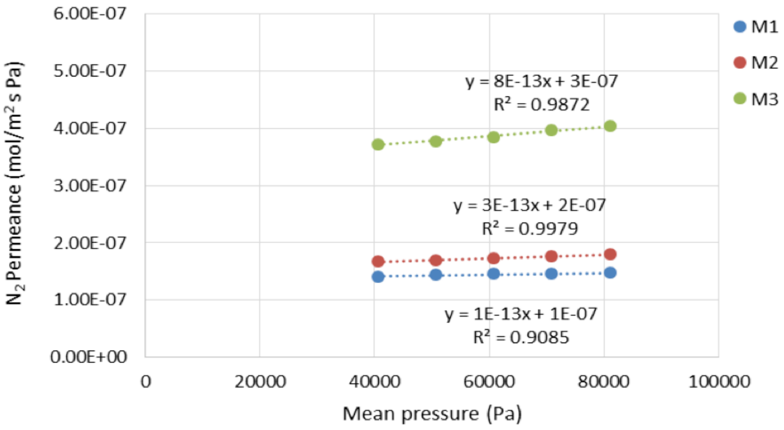

Figure 3: N_2 permeance of the membranes versus mean pressure.

surface porosity are given in Table **1**. N_2 permeance of M3membrane significantly improved by addition of PEG in the polymer dope. This phenomenon can confirm higher surface porosity and larger pore sizes of the membrane. In addition, due to higher slope of the line for M3 membrane, it seems that the Poiseuille flow controls N_2 permeation through the membrane which represents larger pore sizes. On the other hand, slope of the lines for M1 and M2 membranes is relatively lower which means the Knudsen flows controls gas transfer through the membranes. This can confirm the smaller pore sizes of the membranes as shown in Table **1**. By addition of PEG-600, M2 membrane showed relatively higher surface porosity than M3 membrane. This means that the optimum value of PEG-600 in the solution could be in the range of 3 to 6% which should be confirmed by further experiments. In fact, when the amount of PEG-600 in the polymer solution increases the solution turn to a thermodynamically unstable condition which tends to a higher phase separation rate during coagulation. In general, this phenomenon can result in a membrane with finger-like morphology, higher surface porosity and thinner skin layer [23]. Therefore, the increase in the N_2permeance of M3 membrane can be related to the membrane structure.

The mean value of outer surface water contact angle of the membranes as a function of time is given in Figure **4**. For all the prepared membranes, about five degree reduction in the CA value was observed after 60 s exposer of the droplet to the surface. This relatively high decrease of CA can be related to hydrophilic nature of the PAI. In comparison, M3 membrane presented higher mean CA value of about 76 °. It should be noted that not only surface porosity but also surface roughness of the membrane play important roles on the CA variation. In fact, the water droplet can penetrate into the membrane pores due to the capillary force which can result in reduction of CA value. Although, addition of 6% PEG-600 resulted in high surface porosity, higher CA of M3 membrane might be attributed to higher surface roughness produced during phase inversion process. It should be noted that many factors such as surface heterogeneity, surface roughness, measurement time and the droplet size can influence the contact angle of the porous membranes [24].

CO_2 Absorption Performance of Membranes

CO_2 absorption flux of the membranes was measured at various water flow rates, constant temperature of 25 °C and pressure of 100 kPa. The

Table 1: Characteristic of Prepared Membranes

Membrane	N_2 permeance at 100 kPa (GPU)	Mean pore size (µm)	Effective surface porosity, ε/L_p (m^{-1})	Water contact angle (°)
M1	482	0.09	17.4	72.6 ± 0.70
M2	587	0.15	23.4	70.4 ± 0.54
M3	1320	0.23	19.6	74.2 ± 1.03

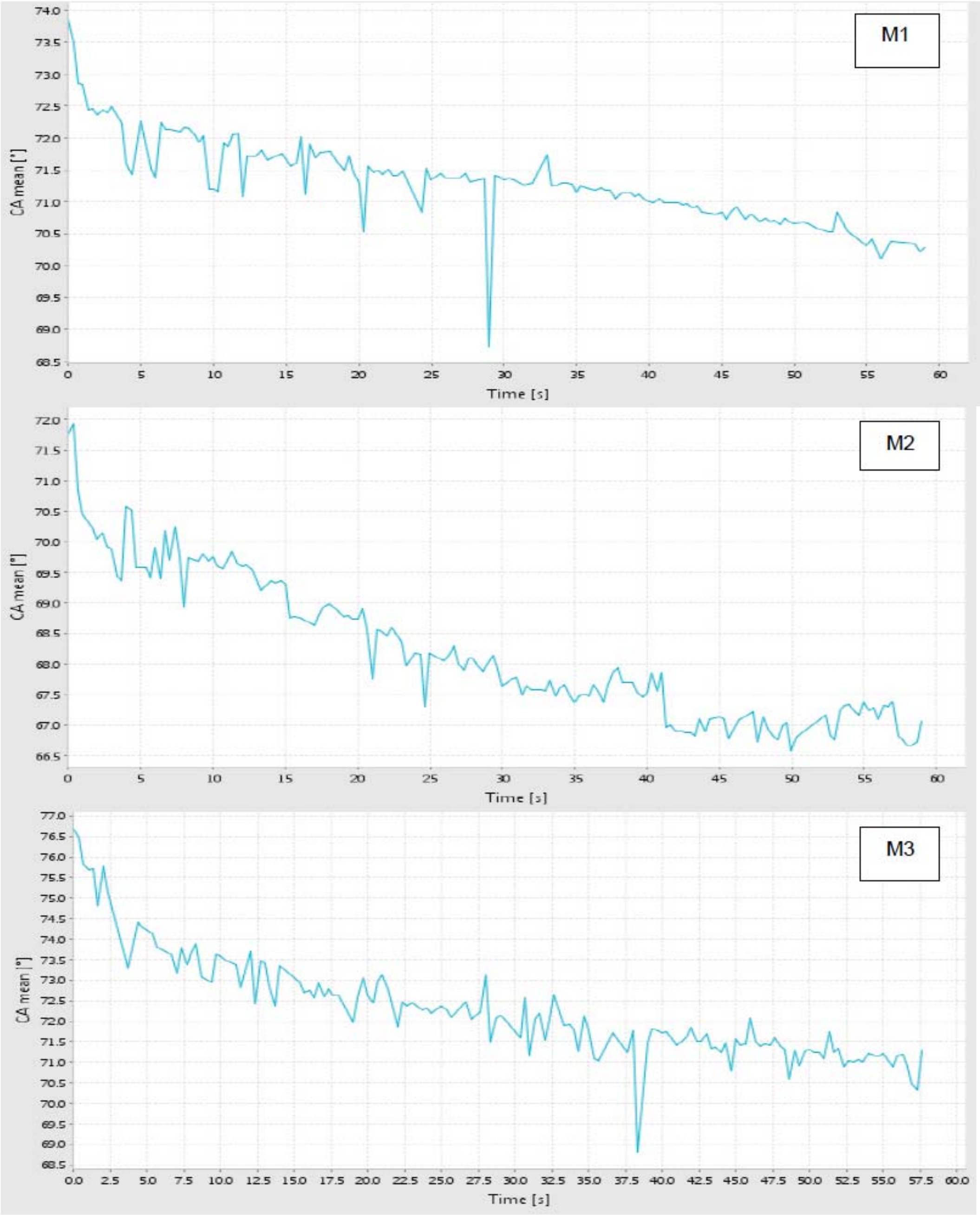

Figure 4: Variation of water contact angle of the membranes as a function of time.

results of CO_2 flux versus water flow rate are given in Figure **5**. By increasing water flow rate a significant increase in the CO_2 flux was observed for the membranes. This can be a result of reducing concentration boundary layer thickness on the outer surface of the membrane. In fact, at higher water velocity, the mass transfer resistance decreases which consequently increases the CO_2 absorption flux [25].

In comparison, M3 membrane showed a considerably higher CO_2 flux than the other PAI membranes. As discussed earlier, M3 membrane presented a developed structure with higher permeability and CA value. Indeed, these two factors play important roles on the performance of the membrane contactor for gas absorption. Membranes with high permeability (surface porosity) provide high gas-liquid contact during CO_2 absorption and result in higher CO_2 flux. In addition, higher surface hydrophobicity can minimize membrane wetting and CO_2 flux reduction. It is proven that partial wetting of the membrane can significantly deteriorate the absorption flux [25]. At water flow rate of 80 ml/min, M3 membrane showed a CO_2 flux of about 52 % higher

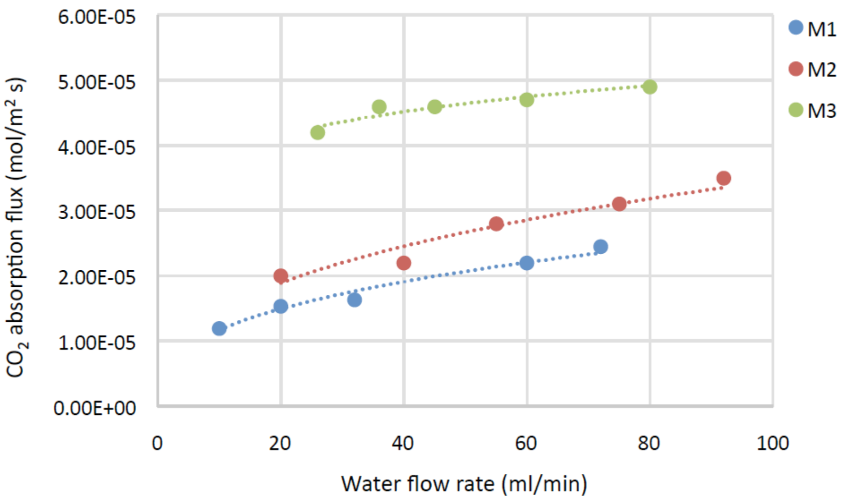

Figure 5: CO_2 absorption flux of the membranes as a function of water flow rate.

than M1 membrane. For M2 membrane, although the permeability slightly increased by addition of PEG, no significant change in the CO_2 flux might be related to the lower CA value of the membrane.

At the same operating condition, CO_2 absorption performance of different polymeric hollow fiber membrane contactors was compared in Table **2**. As can be seen, hydrophilic membranes such as polyethersulfone (PES), PSF and PEI also showed a good performance at a short-term operation. This can be related to high surface porosity or permeability of the prepared membrane. However, it should be noted that gradual wetting of hydrophilic membranes at a long-term operation can significantly deteriorate the absorption performance. Hydrophobic membranes such as polypropylene (PP) and polytetrafluoroethylene (PTFE) are favorable options for a long-term stable operation. However, since these membranes are prepared by thermal and stretching methods, achieving high permeability is challenging. On the other hand, porous hydrophobic PVDF hollow fiber membranes have extensively developed for CO_2 absorption due to easy preparation by phase-inversion process. Recently, PAI membranes have attracted attentions for CO_2 absorption thanks to high chemical, thermal and mechanical stability of the membranes. Improvement of structure and surface hydrophobicity of PAI membranes can enhance CO_2 absorption performance. In present work, by development of the PAI membrane structure, CO_2 flux of 5.0×10^{-5} mol/m^2 s at absorbent velocity of 0.01 m/s was achieved which is a reasonable result compared to hydrophobic surface modified PAI membrane (Table **2**). Therefore, it can be concluded that permeability and hydrophobicity of the PAI membranes are two key factors for CO_2 absorption which can be controlled during fabrication process.

Table 2: CO_2 Absorption Performance of Various Polymeric Membrane Contactors

Membrane	Pore size (µm)	N_2permeance at 100 kPa (GPU)	Absorbent velocity (m/s)	CO_2 flux (mol/m^2 s)	Ref.
PES	0.19	15460*	0.01	7.0×10^{-4}	[26]
PSF	0.07	3400	0.02	5.0×10^{-4}	[27]
PEI	0.12	1437*	0.015	5.7×10^{-4}	[28]
PVDF	0.09	11600	0.02	6.5×10^{-4}	[27]
Commercial (PP)	NA	NA	0.008	1.5×10^{-4}	[16]
Commercial (PTFE)	NA	NA	0.008	3.5×10^{-4}	[16]
Commercial PVDF	0.2	NA	0.3	6.5×10^{-4}	[29]
Surface modified PAI	NA	NA	0.1	6.5×10^{-4}	[21]
Present work	0.23	1320	0.01	5.0×10^{-5}	-

*Helium permeance.

CONCLUSION

Structure of PAI flat sheet asymmetric membranes was improved by addition of PEG in the polymer solution. The prepared membranes were characterized in terms of gas permeation and water contact angle measurements. As compared with plain PAI membrane, by increasingPEG-600 in the polymer solution, the prepared membranes presented higher N_2 permeance which is a promising factor for membrane contactor applications. Meanwhile, the effect of PEG-600 on the contact angle of the membranes was insignificant. The improved membrane showed a mean pore size of 0.24 µm, N_2 permeance of 1320 GPU and outer surface contact angle of 74º. CO_2 absorption performance of the membranes was compared through a semi-batch membrane contactor operation. The improved membrane demonstrated a CO_2 flux of approximately 52 % higher than the plain membrane which could be related to the membrane structure with high permeability. It can be said that a developed PAI membrane with high surface porosity and hydrophobicity can be a potential alternative for membrane contactor application.

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