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Oxygen/Nitrogen Gas Separation by Polyetherimide Hollow Fiber Membrane: Effects of Bore Fluid Rate on Permeance and Selectivity

K. C. Chong*, S. O. Lai, H. S. Thiam & S. S. Lee

Lee Kong Chian Faculty of Engineering and Science, Universiti Tunku Abdul Rahman, Jalan Sungai Long, Bandar Sungai Long
43000 Kajang, Malaysia

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ABSTRACT

The demand of the oxygen and nitrogen gas had been increasing exponentially since the industrial evolution. The conventional gas production technique such as cryogenic distillation and pressure swing absorption able to produce high gas purity and production rate. However, the conventional technique required extensive plant size and energy requirement. Membrane technology has been a key research over the past decades due to its dignified separation technology where no addition of chemicals, low energy usage as well as low setting up requirement. Polymeric hollow fiber membranes are commonly fabricated by dry-jet wet phase inversion attributed to the ability of mass membrane production. To date, many literature reported the effect of the fabrication parameter on the spinning of the hollow fiber membrane such as air gap and force convection on the properties of the membrane, however to the best of our knowledge, there is few attention to study the effect of bore fluid flow rate. In this work, polyetherimide (PEI) is fabricated by dry-jet wet phase inversion techniques with different bore fluid flow rate to evaluate its effect on physical properties and gas permeation of oxygen/nitrogen. Generally, the PEI membrane possess similar morphology of possesses asymmetric thin and dense structure supported by finger like structure. However, the thickness of the membrane decreases with the increase of bore fluid flowrate. The gas permeation study suggested that PEI membrane with highest bore fluid flow rate (1.0 mL/min) in this work possess yield the highest selectivity (3.92), gas permeance for both oxygen (47.15 GPU) and nitrogen gas (12.03 GPU).

Keywords: Oxygen, Nitrogen, Gas Separation, Polyetherimide, Bore Fluid Flowrate

1.0 INTRODUCTION

The demand on oxygen and nitrogen is increasing annually due to the industrialization. Industries such as steel industry, agriculture industry and food processing industry required massive amount of these gaseous to operate continuously to ensure the supply of their products able to fulfil the world demand [1]. The most common type of nitrogen used in industry is in the form of nitrous oxide and liquid nitrogen. Moreover, oxygen is used as oxy-fuel or liquid oxygen in industry. The conventional techniques use for the oxygen or nitrogen production are cryogenic distillation and pressure swing absorption. These processes able to produce medium and large scale of high purity gas production in the range of 20 to 300 tonnes per day [2]. Specific product requirement and purity of the gas product are the problem faced by the producer since the industrial processes such as mixtures separation and purification are indispensable [3].

* Corresponding to: K. C. Chong (email: chongkc@utar.edu.my)
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Drying, evaporation and distillation are the conventional separation processes that required high setting up capital and prone to pollute the environment. Additionally, the conventional methods are energy extensively as it takes up of 10 to 15% proportion of the overall world energy consumption.

Membrane technology has been a prominent technology throughout the years since the earliest industrial operation in gas separation using Prism membrane to separate hydrogen gas from the purge stream of ammonia plants in 1980 attributed to its low energy and space requirement relative to conventional gas production [4]. The driving force of a gas membrane separation is pressure difference whereby gases travel under a pressure gradient and separated when it passes through membrane in merit of the contrast in solubility and diffusivity. There are some studies reported in literature which uses concentration difference, partial pressure difference and electrical potential as driving force in the binary gas separation [5]. As no phase change is required for separation in a membrane process, only low energy consumption is required. Some other advantages of using membrane technology also include ease of scaling up due to its simple modular, simple operation and environmentally friendly [6, 7]. Despite of the advantages of the used of membrane technology in gas separation, this technique is always limited by the trade-off between permeance of the gases and selectivity [8]. Robeson first introduced the trade-off threshold between the gas permeance and selectivity in 1991 following by the re-visitation at 2008. The trade-offline introduced by Robeson consist of hundreds of literature reference based on the literature review and a good gas separation membrane shall recorded the performance more than the trade-off line for commercial viability [9, 10].

Polymeric hollow fiber membranes are commonly fabricated by either electro-spinning or dry-jet wet phase inversion. Most of the literature reported the hollow fiber membrane fabricated by the dry-jet wet phase inversion attributed to the ease of the setup and required low energy consumption [11, 12]. Many researchers study the effect of the fabrication parameter on the spinning of the hollow fiber membrane such as air gap, winding drum speed, type of coagulant used and force convection on the physical properties of the membrane. Generally, a thin and dense membrane is desired in gas separation due to low mass resistance and the good mechanical strength to withstand the pressure during gas permeation study [13]. To the best of our knowledge, there are little work done in the study on the effect the bore fluid flow rate as the fabrication parameter in the PEI hollow fiber membrane fabrication on the membrane structure. In this work, polyetherimide (PEI) membrane was used to study effect of the bore fluid flow rate in the membrane fabrication parameter and evaluate its effect on the oxygen/nitrogen gas separation. The selection of PEI membrane in this work attributed to its superior gas separation performance, excellent thermal and chemical stability as reported in previous literature studies. [14] The fabricated membranes were characterized physically, and performance tested by gas permeation study. Lastly, the gas separation performance yield by the membrane were compared with the Robeson upper bound.

2.0 MATERIALS AND METHOD

2.1 Materials

Polyetherimide (PEI) in pellet form obtained from Merck, Germany is used for the fabrication of hollow fiber membrane. The solvent used for bore fluid was dimethylacetamide (DMAc) obtain from Sigma Aldrich, United States. The co-solvents used to improve the miscibility of the dope solution in this study are tetrahydrofuran (THF) and ethanol obtained from Sigma Aldrich, United States and QREC, Malaysia, respectively.

2.2 Fabrication of Hollow Fibre Membrane

PEI comes in a pellet form required zero moisture before preparing the dope solution. Thus, the pellet form PEI was left to dry for a day in a vacuum oven at around 70ºC. Next, the dope solution was prepared by slowly adding 30 wt% PEI into 30 wt% dimethylacetamide (DMAc), 30 wt% tetrahydrofuran (THF) and 10 wt% ethanol respectively. The pre-treated pellets were added into the solvent slowly and mechanical stirring is applied until it became a homogenous solution. The dope solution was degassed to remove all the air bubble by ultrasonic sonicator. In this study, the hollow fiber membrane was fabricated by dry-jet wet phase inversion method (Figure 1). The gear pump in the spinning machine drawn the dope solution from the dope reservoir through the annular spinneret to the spinneret. In spinneret, solvent/non-solvent exchange occurred when the bore fluid is flow in the middle of the dope solution and formed a lumen. The PEI hollow fiber membrane under spinning conditions of bore liquid flow rate at 0.1 mL/min, 0.3 mL/min, 0.7 mL/min and 1.0 mL/min are labelled as PEI-1, PEI-2, PEI-3 and PEI-4, respectively. The hollow fiber membrane began to form in shape in the coagulation bath and being sent to the washing bath. The hollow fiber membrane was then immersed in the water followed by put into the oven to remove all the solvent and moisture residue before tested in the gas permeation system. The details of the hollow fiber spinning parameter were tabulated in the Table 1.

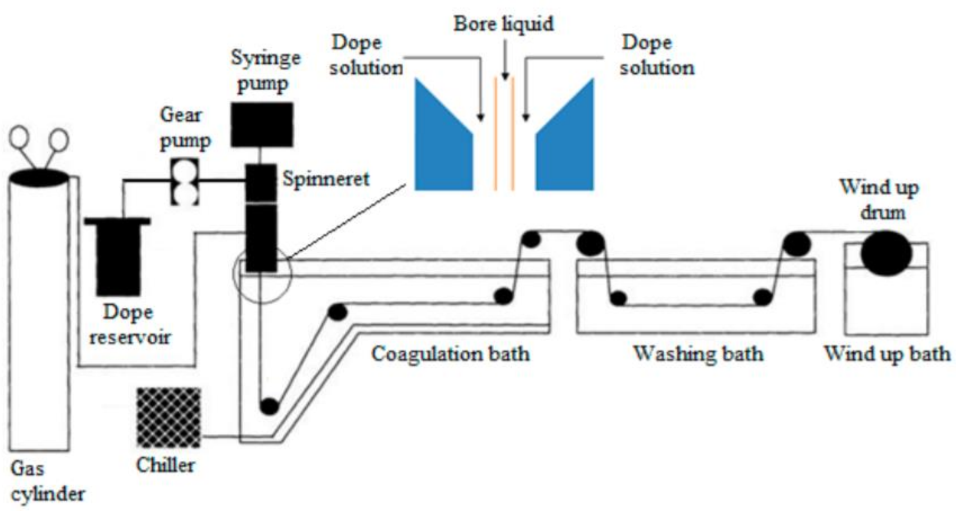

Figure 1 Dry-Jet Wet Phase Inversion Schematic Diagram [15]

Table 1 PEI hollow fiber membrane fabrication parameter

Spinning parameter	Value
Spinneret outer diameter (mm)	0.6
Spinneret inner diameter (mm)	0.3
Bore liquid	Distilled water
Bore liquid flow rate (mL/min)	0.1 (PEI-1) 0.3 (PEI-2) 0.7 (PEI-3) 1.0 (PEI-4)
External coagulant	Tap water
Air gap (cm)	30

2.3 Membrane Characterization Study

Characterization methods were employed in determining the nature and physical properties of a material. Scanning Electron Microscopy (SEM, Hitachi S3400N) was used to examine the morphology of the membrane in this study. In the preparation of the membrane sample for the SEM analysis, pre-treatment was required for the membrane sample. The hollow fiber membrane was fractured cryogenically with the aid of liquid nitrogen so that a clearer morphology result can be obtained. The cracked sample were coated with a thin layer of gold by Emitech, SC-7620, sputter coating machine to increase the surface conductivity of the membrane sample.

The membrane porosity (ε) defined the fraction of the void presence in the membrane where a good membrane for gas separation should be non-porous and dense membrane to avoid low separation performance [16]. The membrane samples are dried in the vacuum oven at 70°C for four hours in order to remove any moisture content. Five membrane samples were cut into a predetermined length are weighted by digital balance as dry weight. Subsequently, the membrane was wetted in isopropanol (Thermo Fisher, United States) for two hours. Later, the wetted membrane was dry by hand to remove extra moisture on the surface. The dry and weight were measure as W_1 and W_2, respectively. The membrane porosity (ε) was calculated by gravitational method as reported by Wahab *et al*. as shown in the following equation, where ρ_i and ρ_p are the density of isopropanol (0.79 g cm^{-3}) and PEI (1.27 g cm^{-3}), respectively [16].

$$\varepsilon = \frac{(W_1-W_2)/\rho_i}{(W_1-W_2)/\rho_i+W_2/\rho_p} \times 100\% \qquad (1)$$

2.4 Gas Permeation Study

The schematic diagram of gas permeation test was shown in the Figure 2. The permeation of the single gas (pure oxygen and nitrogen gas with the purity more than 99.99%) was tested by using the PEI hollow fiber membrane. With the aid of the bubble flow meter, the gas permeance (P_A/l, GPU) could be obtained through Equation 2 as reported by Zulhairun *et al*. where Q is the volumetric flowrate (cm^3 s^{-1} STP), A is the effective membrane area at 9.61 ± 2.3 cm^2, ΔP is the transmembrane pressure (cm Hg) and T is the room temperature at 27°C [17].

$$\frac{P_A}{l} = \frac{273.15\times10^6 Q}{A\Delta PT} \qquad (2)$$

On the meantime, the selectivity of the membrane ($\alpha_{A/B}$) was calculated with the following equation as reported by

Zulhairun *et al*. where P_{O2} and P_{N2} are the permeance of oxygen gas and nitrogen gas, respectively [17].

$$\alpha_{\left(\frac{A}{B}\right)} = \frac{P_{O2}}{P_{N2}} \quad (3)$$

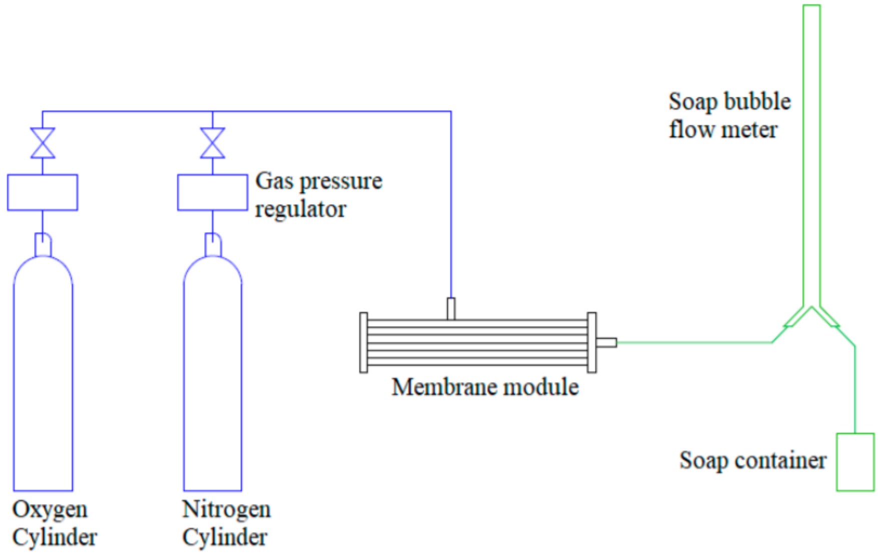

Figure 2 The Schematic Diagram of Gas Permeation Study [15]

3.0 RESULTS AND DISCUSSION

3.1 Effect of the bore fluid flow rate on the physical properties of PEI hollow fiber membrane

The morphologies of the fabricated hollow fiber membranes were shown in the SEM images in Figure 3(a) to (d). The morphologies of the PEI hollow fiber membrane are similar under spinning conditions of bore liquid flow rate (BFR) at 0.1 mL/min (PEI-1), 0.3 mL/min (PEI-2), 0.7 mL/min (PEI-3) and 1.0 mL/min (PEI-4) where the membrane inner layer can be seen to be made up of thin and dense spongy structure supported by finger like structure which is the structure of an asymmetric membrane as a result of quick phase separation. The finger-like structures accountable for the increase of permeability in gas separation are formed from pore initiation and growth. During phase separation, two phases are developed, polymer-rich and polymer-lean. The finger-like structure is initiated by polymer-lean phase nuclei and created under the top layer [18]. The growth of these nuclei is maintained if the solvent supplied are adequate and ends when initiation of new nuclei occurs. The termination of growth can also occur at glass transition temperature for polymer-lean phase [19]. With the application of water which is a relatively strong non-solvent for both bore fluid and coagulation bath, the solvent/non-solvent exchange occur aggressively forming the finger like structure [20].

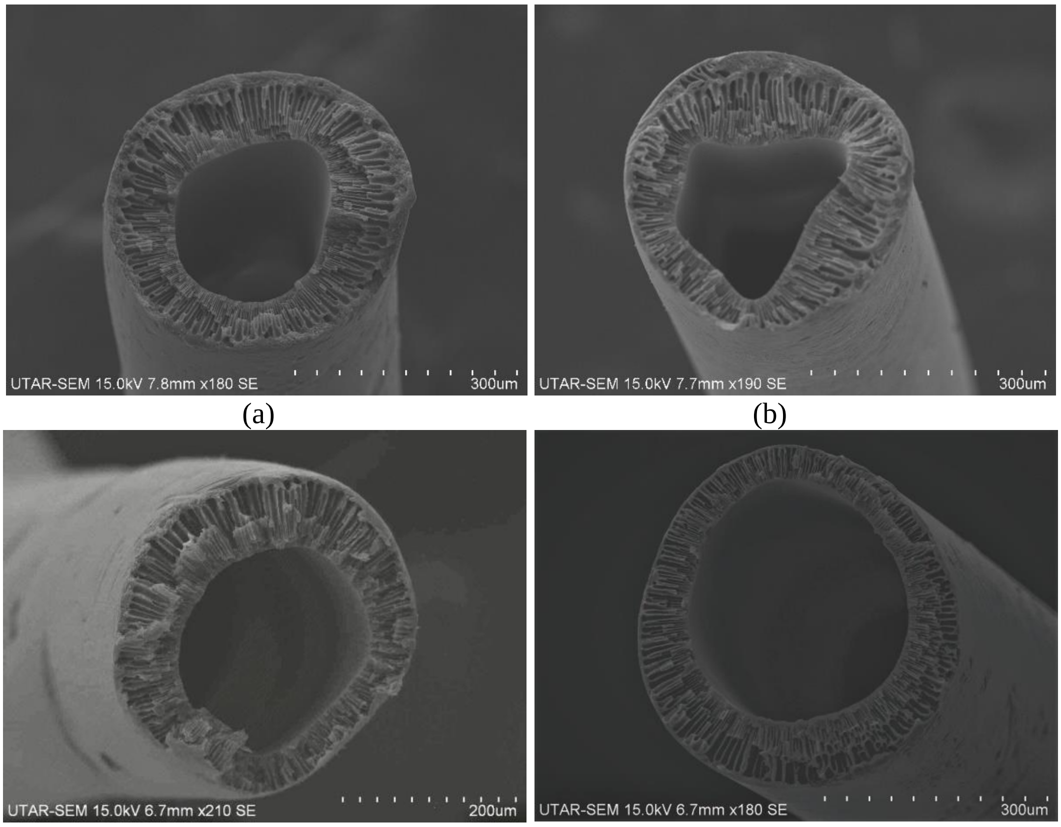

Figure 3 SEM image of cross-sectional pristine PEI membrane under different spinning parameter (a) PEI-1; (b) PEI-2; (c) PEI-3; (d) PEI-4

It is noteworthy to mention the effect of the bore liquid flow rate on the morphology where the increase of bore flow rate lead to the decreases of the spongy structure as the exchange between solvent/non-solvent occur more rapidly [21]. Additionally, the membrane lumen geometry also been affected by the bore fluid flow rate where the higher the bore fluid flow rate, the thinner the membrane thickness (Table 2). As the flowrate of the bore fluid increases, the pressure of the bore fluid exerted on the inner layer of the hollow fiber membrane increases, lead to the reduction in the thickness and the inner diameter of the hollow fiber membrane [22]. The porosity of the fabricated hollow fiber membranes suggested that there is no significant effect of the bore fluid flow rate as the porosity are in the range of 21.24 to 22.70% which is similar to the report by previous literature studies [15].

Table 2 Inner diameter, thickness and porosity of PEI hollow fiber membrane

Membrane	Inner Diameter (μm)	Membrane Thickness (μm)	Porosity, ε (%)
PEI-1	218 ± 15	87 ± 3	22.70 ± 1.4
PEI-2	233 ± 21	68 ± 4	21.57 ± 2.5
PEI-3	311 ± 18	63 ± 3	21.24 ± 1.7
PEI-4	223 ± 24	57 ± 5	21.65 ± 1.8

3.2 Effect of the Bore Fluid Flow Rate on the Gas Separation Performance

The O_2/N_2 gas separation performance recorded by the PEI hollow fiber membrane under different bore fluid flow rate was tabulated in Table 3. The gas permeance of oxygen gas is found to be in the range of 33.57 to 47.15 GPU whereas the gas pemeance of nitrogen gas is yield at the range of 10.79 to 12.03 GPU. Additionally, the selectivity of the membranes was in the range of 3.11 to 3.92. The separation performance of the PEI hollow increases with the increase of the bore fluid flow rate of the membrane where the oxygen and nitrogen gas permeance improved by 40% and 11%, respectively whereas the selectivity increases by 26%. The improvement of the gas permeance of the PEI membrane might attributed to the thickness of the membrane as the increase of bore fluid flow rate during the spinning of the hollow fiber membrane will decreases the membrane thickness as discussed in the previous section. The reduction of the thickness of the membrane lead to the reduction in mass transfer resistance as reported by Kamaruddin and Koros [23]. Nevertheless, the thickness of the membrane should be well control and to be optimized as low membrane thickness will lead to the reduction of overall mechanical strength of the membrane. Membranes with low mechanical strength is generally unsuitable in the gas separation application where the membrane might be prone to leaking and rupture attributed to the high-pressure application [24, 25]. However, no leaking or rupture of the membrane were observed in this study through the sampling time.

Table 3 Permeance and selectivity of PEI hollow fiber membrane in O_2/N_2 gas separation

Polymer	**Oxygen**	**Nitrogen**	**O_2/N_2 Selectivity (α)**
	x 10^{-6} $cm^3{}_{STP}/cm^2$ s cm Hg (GPU)		
PEI-1	33.57 ± 1.37	10.79 ± 2.47	3.11
PEI-2	42.79 ± 2.13	12.26 ± 1.95	3.49
PEI-3	45.56 ± 1.89	12.08 ± 2.31	3.77
PEI-4	47.15 ± 2.09	12.03 ± 2.29	3.92

3.3 Evaluation of Gas Separation Performance in Robeson Upper Bound

The oxygen permeance and selectivity of the fabricated PEI hollow fiber membranes were plotted in the 2008 Robeson upper bound for comparison (Figure 4). The selectivity of the membrane recorded in the upper bound with the oxygen permeance range of 33.57 to 47.15 GPU (3.36 to 4.71 Barrer) whereas the highest selectivity of the membrane in this study is 3.92 by PEI-4. The comparison of the Robeson upper bound with the top performance membrane (PEI-4) in this study suggested that there are room for improvement in both selectivity and permeance in order to allow the membrane to be commercially feasible.

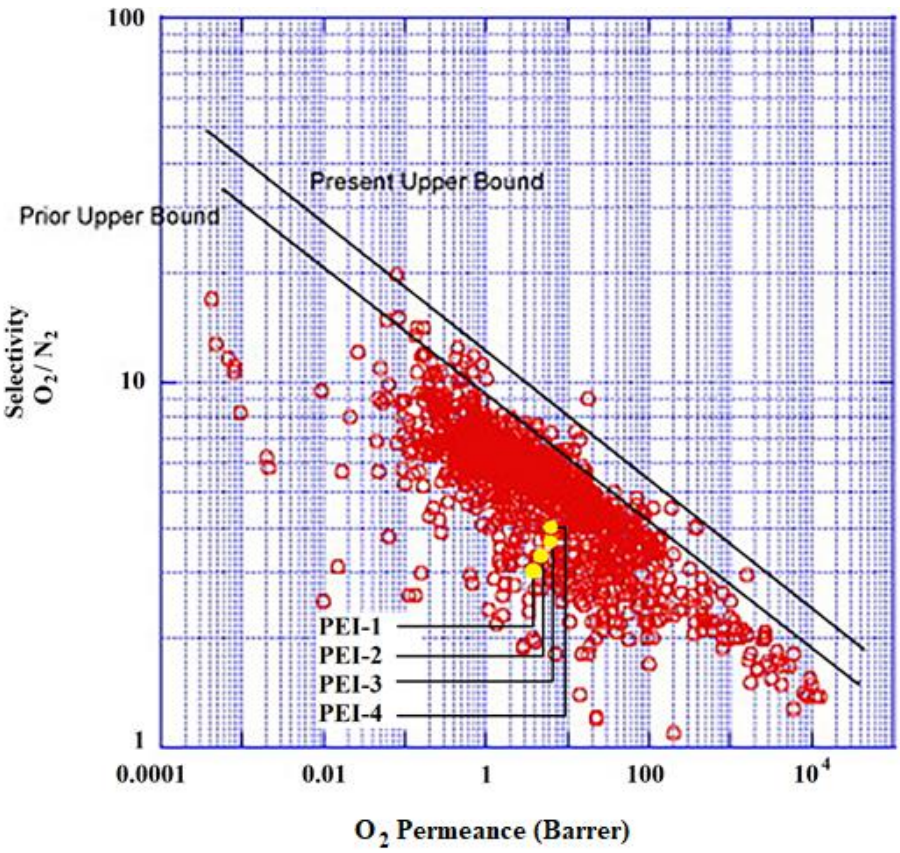

Figure 4 Comparison of O_2/N_2 gas separation performance in Robeson upper bound

4.0 CONCLUSION

The PEI hollow fiber membranes were fabricated by dry-jet wet phase inversion with the bore fluid flow rate of 0.1 mL/min, 0.3 mL/min, 0.7 mL/min and 1.0 mL/min. Generally, all the fabricated PEI hollow fiber membranes possesses asymmetric thin and dense structure supported by finger like structure. The membrane fabricated with highest bore fluid rate at 1.0 mL/min recorded the lowest thickness attributed to the pressure of the bore fluid exerted in the spinneret during membrane spinning. PEI-4 (PEI hollow fiber membrane with 1.0 mL/min bore fluid flow rate) yield the highest selectivity, gas permeance for both oxygen and nitrogen gas. This phenomenon was attributed to the reduction of mass transfer resistance arise from the lower membrane thickness. The comparison of the membrane separation performance with respect to the Robeson upper bound depicted that the performance of the membranes in this studies were not closed to the bound, suggesting further improvement such as coating or surface modification are essential to unleash the potential of this PEI hollow fiber membrane in O_2/N_2 gas separation

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