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# Hydrophobic Modification of Polyetherimide Hollow Fiber Membrane Contactor by 2-(Perfluoroalkyl) Ethanol Coating for Carbon Dioxide Absorption

M. R. Ahmadpour & A. Mansourizadeh*

Department of Chemical Engineering, Membrane Science and Technology Research Center (MSTRC), Gachsaran Branch, Islamic Azad University, Gachsaran, Iran

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## ABSTRACT

Highly porous polyether imide (PEI) hollow fiber membranes were fabricated through a phase-inversion process. Surface modification of the membranes was performed by coating an ultra-thin layer of 2-(perfluoroalkyl) ethanol (ZONYL). The properties of the prepared membranes were evaluated through field emission scanning electronic microscopy (FESEM), water contact angle, N2 permeation, overall porosity, critical water entry pressure ($CEP_w$) and collapsing pressure experiments. From FESEM, the PEI membrane showed an open structure with large finger-likes cavities. By coating the ZONYL layer, the modified membrane presented improved outer surface water contact angle (105 °), $CEP_w$ (450 kPa), and collapsing pressure (750 kPa). The improved PEI-ZONYL membrane presented an almost stable $CO_2$ absorption flux ($6.8\times10^{-4}$ mol/m$^2$ s) during 72 h of the gas-liquid membrane contactor operation while a significant flux reduction was observed for the plain PEI membrane. Therefore, the improved membrane with high surface hydrophobicity and porosity can be a promising candidate for high performance gas-liquid membrane contactors.

*Keywords*: PEI hollow fiber membrane, 2-(Perfluoroalkyl) ethanol, coating, membrane contactor, $CO_2$ absorption

## 1.0 INTRODUCTION

Carbon Dioxide ($CO_2$) as one of the main greenhouse gases has been responsible for global climate change. Due to the importance of greenhouse gases emission, the Kyoto Protocol was adopted since 1997 (Kyoto, Japan) in order to control global warming by reducing greenhouse gas concentrations in the atmosphere [1]. Since the world economic is related to the combustion of fossil fuels as the main source of $CO_2$ emission, $CO_2$ separation from the gas streams by developed technologies has been the leading research focus.

Membrane-based gas separation technologies have shown several advantages such as high-efficiency, simple process, and low energy consumption [2, 3]. Usually, selective dense membranes that are available for $CO_2$ separation cannot provide the necessary purity, in which an advanced membrane process such as gas-liquid membrane contactor has been applied [4, 5]. A gas-liquid contactor process is a hybrid performance which provides membrane separation and solvent absorption together [6]. The gas and liquid phases are separated by the membrane interface which provides the mass transfer contact area for $CO_2$ absorption. Therefore, the gas-liquid membrane contactor has proposed several advantages over the traditional contacting devices for gas absorption

* Corresponding to: A. Mansourizadeh (email: a.mansourizadeh@yahoo.com)
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[7, 8]. However, membrane wetting is the main drawback that can significantly affect the mass transfer process [9]. It should be noted that the membrane is the key factor in a membrane contactor process for $CO_2$ absorption which is responsible for higher removal efficiency and the stability of the process. So far, several works have been conducted on development of the membrane structure for $CO_2$ absorption.

Li *et al*. [10] fabricated super-hydrophobic poly ether ether keton (PEEK) hollow fiber membranes by a melt extrusion process for $CO_2$ absorption by diethanolamine (DEA). They reported a stable $CO_2$ removal efficiency of about 90% for over 100 h of the gas-liquid membrane contactor operation. Lin *et al*. [11] grafted fluorinated $SiO_2$ and $TiO_2$ on the surface of polyether sulfone (PES) by hydrolyzing silicon precursors and titanium precursors at high temperature in order to improve anti-corrosion and hydrophobicity of the flat-sheet PES membranes. The $CO_2$ absorption flux of $10.11 \times 10^{-3}$ mol/m$^2$s and the $CO_2/CH_4$ separation factor of 554.89 were found for the improved fluorinated OH/$SiO_2$-$TiO_2$/PES membrane. In another study, hybrid polyvinylidene fluoride-hexadecyltrimethoxysilane (PVDF–HDTMS) membranes were fabricated via the non-solvent induced phase-inversion method for $CO_2$ absorption through a gas-liquid membrane contactor system [12]. The largest water contact angle on the membrane outer surface was found to be 150°. By adding 1.5 wt% of HDTMS in the dope, the developed membrane showed the maximum $CO_2$ flux of $2.23 \times 10^{-3}$ mol/m$^2$s. After 17 days of the membrane contactor operation, the membrane $CO_2$ flux declined by 17% and then remained stable. Rosli *et al*. [13] applied silica nanoparticles (SiNPs) modified with hexamethyldisilazane (HMDS), dimethyldichlorosilane (DMDCS) and polydimethylsiloxane (PDMS) to produce mixed matrix membranes (MMMs) for $CO_2$ absorption. The modified PVDF/TS-530 MMM showed significant improvements in pore size and $LEP_w$ from 114.45 nm to 80 nm and from 4.75 bar to 7.51 bar, respectively. The $CO_2$ absorption flux of $1.91 \times 10^{-4}$ mol/m$^2$s and the $CO_2/N_2$ selectivity of 22.5 were found for the modified membrane which was higher that the values for the neat PVDF membrane.

It is worth mentioning that surface coating as an easy method has been used to improve surface properties of the hollow fiber membranes for various applications. The fluorinated compounds are favorable to improve surface hydrophobicity of the polymeric membranes and minimize the membrane wetting. In this study, for the first time, 2-(perfluoroalkyl) ethanol as a fluorohydrocarbon macromolecules was coated on the outer surface of the highly porous polyether imide (PEI) hollow fiber membranes for $CO_2$ absorption through a gas-liquid membrane contactor process. The prepared membranes were characterized by field emission scanning electronic microscopy (FESEM), $N_2$ permeation, collapsing pressure, overall porosity, critical water entry pressure ($CEP_w$), and water contact angle experiments. A prolonged $CO_2$ absorption experiment was conducted to evaluate the membranes performance during the membrane contactor operation.

## 2.0 EXPERIMENTAL

### 2.1 Materials

PEI pellets (Ultem®1000) were

supplied by General Electric Co to fabricate hollow fiber membranes. 1-Methyl-2-pyrrolidone (NMP) and glycerol were provided by MERCK and used as the solvent and non-solvent in the polymer dope, respectively. 2-(Perfluoroalkyl) ethanol (Zonyl® BA) and acetone was purchased from Sigma-Aldrich for preparation of the coating solution. Figure 1 represents the molecular structure of hydrophobic Zonyl® BA. Ethanol (99.8%, Sigma-Aldrich) was used for post-treatment of the PEI membranes. Distilled water was used as the physical absorbent in the membrane contactor experiments.

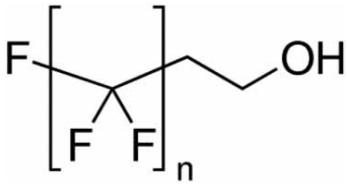


**Figure 1** Molecular structure of 2-(Perfluoroalkyl) ethanol

## 2.2 Fabrication of Hollow Fiber Membranes

In order to prepare a highly porous PEI hollow fiber membrane, composition of the polymer dope was adjusted as PEI/glycerol/NMP 14/4/82 wt% according to our previous study [14]. The homogeneous PEI dope was prepared under a continuous stirring at 60 °C for 12 h. Prior to spinning hollow fiber membranes, the polymer dope was de-bubbled by an ultrasonic bath (15 min) at temperature of 60 °C. The hollow fiber membranes were fabricated by a dry-wet spinning process using constant spinning conditions [14] and then they were immersed in distilled water for 3 days to remove residual NMP and glycerol. The membranes were immersed in pure ethanol for 15 min to minimize shrinkage and pores collapse during drying at room temperature.

A dip-coating process was applied for surface modification of the dry hollow fiber membranes. The coating solution was prepared by dissolution of 2 wt% Zonyl in acetone according to our previous study [14]. Then, the dead ends hollow fibers were immersed in the coating solution for 30 min to form an ultra-thin layer on the outer surface. The hollow fibers were cured by keeping them in a vacuum oven at 70 °C for 12 h.

## 2.3 Characterization of the Membranes

Field emission scanning electronic microscopy (Hitachi S-4700 FESEM) was applied to study cross-section, inner surface and outer surface morphology of the plain and surface coated PEI membranes.

$N_2$ permeation experiment was performed to estimate mean pore size and surface porosity over pore length of the membranes [15]. For this propose, the membrane module was prepared by sealing two dead-end hollow fibers with the length of 10 cm in a stainless tube (ID = 1.4 cm). $N_2$ from a high pressure cylinder was supplied to the shell side of the module and its pressure was adjusted from 100 kPa to 200 kPa at 20 kPa intervals by a gas pressure regulator. The $N_2$ permeation rate from the lumen side of the hollow fibers was measured by a soap bubble flowmeter. $N_2$ permeance (mol/m$^2$sPa) was calculated as [16]:

$$J = \frac{40.87\ Q}{A_o \times \Delta P} \quad (1)$$

Where $Q$ is $N_2$ permeation rate (m$^3$/s); $A_o$ is outer surface area of the hollow fibers (m$^2$); and $\Delta P$ is pressure difference (Pa) across the membrane. Assuming cylinder-shaped pores on

the outer skin layer of the membrane, $N_2$ permeance can be calculated by summation of Poiseuille and Knudsen flows as [17]:

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

$$J = K_0 + P_0\bar{P} \tag{2}$$

Where $L_p$ and $r_p$ are pore length and mean pore radius (m), respectively; $\varepsilon_s$ is surface porosity (-); $R$ is universal gas constant 8.314 (J/mol K); $\mu$ is $N_2$ viscosity (Kg/m s); $M$ is $N_2$ molecular weight (kg/mol); $T$ is gas temperature (K); and $\bar{P}$ is mean pressure (Pa). The intercept ($K_0$) and slope ($P_0$) of the linear equation (2) can be measured by plotting $J$ versus $\bar{P}$ and they were used to estimate mean pore radius and surface porosity over pore length as:

$$r_p = 5.33\left(\frac{P_0}{K_0}\right)\left(\frac{8RT}{\pi\mu}\right)^{0.5} \tag{3}$$

$$\frac{\varepsilon}{L_p} = \frac{8\mu RTP_0}{r_p^2} \tag{4}$$

The overall porosity of the hollow fiber membranes was measured by density measurement. Five membrane samples with the length of 50 cm were totally dried at 105 °C for about 2 h in a vacuum oven and then they weighted by a digital scale to calculate density. The overall porosity ($\varepsilon_o$) can be calculated as [18]:

$$\varepsilon_o(\%) = \left(1 - \frac{\rho_f}{\rho_p}\right) \times 100 \tag{5}$$

Where $\rho_p$ and $\rho_f$ are the polymer and hollow fiber densities (kg/m$^3$), respectively. The density of hollow fiber membrane is calculated as:

$$\rho_f = \frac{4w}{\pi(d_o^2 - d_i^2)L} \tag{6}$$

Where $d_i$ and $d_o$ are the inner and outer diameter of the hollow fibers (m), respectively. $W$ is mass of the hollow fibers (kg); and $L$ is the hollow fiber length (m). The density of PEI is 1270 kg/m$^3$.

Wetting resistance of the hollow fiber membranes was evaluated by the critical water entry pressure ($CEP_w$) test. Distilled water was pressurized into the lumen side of the dead-end hollow fiber membranes by a diaphragm pump and the pressure was increased step by step in 50 kPa intervals. At each constant pressure, the membrane was kept for 10 min to check the presence of water droplet on the membrane surface. $CEP_w$ was reported as the pressure when the first water droplet observed.

Surface hydrophobicity of the PEI hollow fiber membranes was inspected by water contact angle measurement. A contact angle goniometer (DSA20E, KRUSS) was used to measure the contact angle of the outer surface of the hollow fiber membranes. An average value of contact angle was reported at ten different locations of the membrane surface.

Collapsing pressure test was applied to evaluate mechanical stability of the PEI hollow fiber membranes. Using the same membrane module for $N_2$ permeation test, $N_2$ pressure in the shell side of the module was improved at 50 kPa intervals. At each constant pressure, the module was maintained for 5 min to check a rapid decrease/increase in the permeate flow which represents collapsing of the hollow fibers. Collapsing of the fibers was visually confirmed by opening the module.

### 2.4 $CO_2$ Absorption Experiment

The membrane contactor module was prepared by packing 20 hollow fibers and used in the gas-liquid membrane

contactor system to measure the $CO_2$ absorption flux. The characteristics of the module and flow diagram of the experimental membrane contactor system are shown in Table 1 and Figure 2, respectively.

**Table 1** Characteristics of the membrane contactor module

| Parameter | Value |
|---|---|
| Module o.d. (mm) | 14 |
| Module length (mm) | 300 |
| Effective fibers length (mm) | 200 |
| Fiber o.d. (mm) | 0.6 |
| Fiber i.d. (mm) | 0.4 |
| Number of fibers | 20 |
| Effective membrane area ($cm^2$) | 75 |

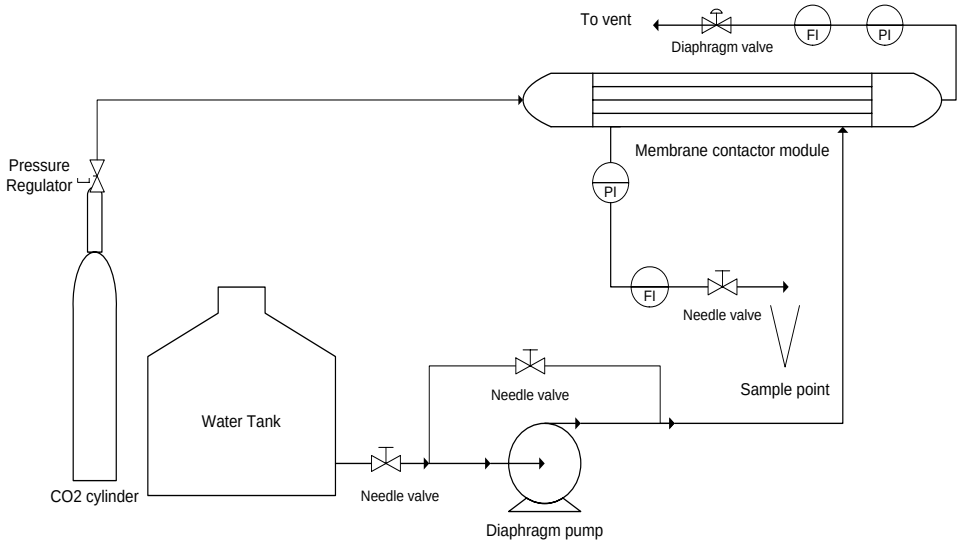


**Figure 2** Flow diagram of the experimental membrane contactor system

Pure $CO_2$ as the feed gas was flowed in the lumen side of the hollow fiber membranes. Distilled water as the liquid absorbent was flowed through shell side of the module in contact with the outer skin layer of fibers. A counter-current mode of operation was used and the operating conditions are given in Table 2.

**Table 2** Operating condition of the gas-liquid membrane contactor system

| Parameter (unit) | Value |
|---|---|
| Liquid pressure (bar) | 1.2 |
| Gas pressure (bar) | 1 |
| Liquid flowrate (ml/min) | 50 - 300 |
| Gas flowrate (ml/min) | 100 |
| Temperature (°C) | 25 |

In order to obtain a steady state condition, the water sample outlet of the module was taken after 30 min of the experiment. Using a chemical titration method, the $CO_2$ concentration in the liquid outflow was measured to calculate the $CO_2$ absorption flux as the below equation:

$$J_{CO2} = \frac{Q_l \times C_{lo}}{A_o} \quad (7)$$

Where $J_{CO2}$ is the $CO_2$ flux (mol/m$^2$ s); $C_{lo}$ is the $CO_2$ concentration (mol/m$^3$) in the liquid outlet of the module; and $A_o$ is the outer surface area of the membrane.

## 3.0 RESULTS AND DISCUSSION

### 3.1 Morphology and Structural Properties of the PEI Hollow Fiber Membranes

Figure 3 shows cross section, outer surface and inner surface morphology of the prepared PEI hollow fiber membranes. The membranes obtained outer diameter, inner diameter and thickness of about 600 µm, 400 µm and 120 µm, respectively. Using a low PEI concentration (14%) and a strong non-solvent additive in the spinning dope resulted in decrease of thermodynamic stability which improved liquid-liquid phase separation and subsequent production of a finger-like structure. It can be said that the kinetic effect of the polymer dope viscosity was overtaken by the thermodynamic effect that formed large finger-likes beneath the outer and inner surfaces. From the outer surface image, the plain PEI membrane showed a good porosity which could be related to the extension of the finger-likes to the surface. In addition, an open microporous inner surface was generated due to using a mixture of NMP/water 70/30 as the bore fluid.

As for the coated PEI membrane, there is no significant change in the cross-section morphology. It seems that an ultra-thin coated layer with thickness of about 1 µm was generated. The modified membrane showed a lower surface porosity with smaller pore sizes which can be attributed to the formation of coated layer. Due to using a low concentration coating solution and short coating time, the formation of an ultra-thin coated layer can be expected. The main objective of this study was to enhance surface hydrophobicity by coating without a significant effect on the surface porosity. Indeed, the high hydrophobicity and surface porosity are favorable factors for gas-liquid membrane contactor since they provide high gas-liquid contact and wetting resistance.

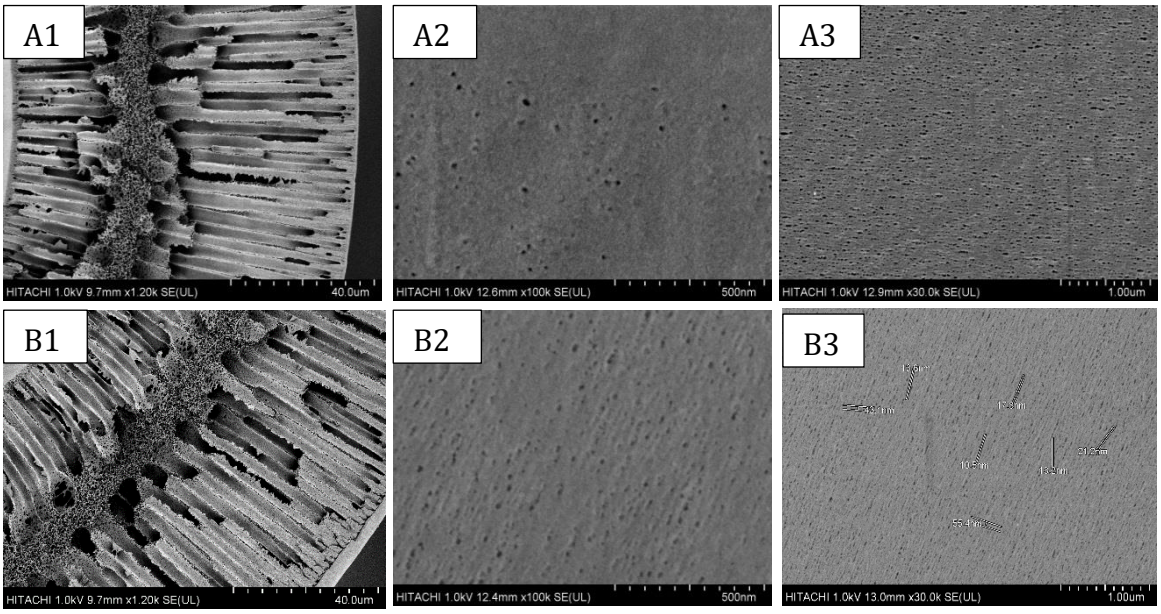


**Figure 3** FESEM images of the PEI hollow fiber membranes: (A) plain membrane; (B) coated membrane; (1) cross section; (2) outer surface; and (3) inner surface

**Table 3** Properties of the plain and coated PEI hollow fiber membranes

| Characteristic | Value | |
|---|---|---|
| | **PEI** | **PEI-ZONYL** |
| Mean pore size (nm) | 32 | 27 |
| Surface porosity over pore lenght ($\varepsilon/l_p$) ($m^{-1}$) | 996 | 581 |
| Outer surface water contact angle (º) | 81.3±1.64 | 105.4±2.55 |
| Overall porosity (%) | 81.2 | 79.8 |
| CEPw (kPa) | 250 | 450 |
| Collapsing pressure (kPa) | 650 | 750 |

Wetting resistance of the membranes was examined through the $CEP_w$ experiment and the results are shown in Table 3. By coating of an ultra-thin layer of ZONYL on the outer surface of the PEI hollow fiber membrane, the $CEP_w$ increased from 250 kPa to 450 kPa which can be associated to the improved hydrophobicity and the smaller pore sizes. According to Laplace-Young equation, pore size, surface hydrophobicity, surface tension and operating pressure can affect the membrane pores wetting [19].

Using $N_2$ permeation test, the mean pore size and surface porosity of the membranes were calculated, as shown in Table 3. The $N_2$ permeance of the membranes as a function of mean pressure was plotted (Figure 4), where the slope ($P_0$) and intercept ($K_0$) of the permeance lines were used for calculation of the mean pore size and surface porosity based on Eqs. (3) and (4). The smaller pore sizes and surface porosity of the coated membrane can be correlated to the formation of an ultra-thin layer. A similar trend was also reported for the PEI hollow fiber membrane coated by silicon rubber used in a gas-liquid membrane contactor system for $CO_2$ removal [20]. It should be noted that although the pore size and surface porosity of the modified membrane slightly decreased, the improved surface hydrophobicity can minimize wetting and enhance the stability of $CO_2$ capture operation.

The prepared porous PEI hollow fiber membrane demonstrated a high overall porosity of about 81% due to the generated open structure with large finger-likes. In general, a higher void fraction in the membrane matrix can be related to the formation of large finger-likes which can provides a higher overall porosity compared to a sponge-like structure [21]. A slightly lower porosity was found for the coated PEI membrane which may be related to the decrease of pores volume due to penetration of the coated solution in the membrane matrix. It can be said that the membranes with high porosity can provide a low mass transfer resistance for a definite application.

Mechanical stability of the prepared PEI hollow fiber membranes was evaluated by collapsing pressure test and the results are given in Table 3. The plain and coated PEI membrane presented collapsing pressure of 650 kPa and 750 kPa, respectively. Both membranes showed a good collapsing pressure which can be related to the molecular structure of PEI with high $T_g$ of about 215 °C. About 100 kPa increase in the collapsing pressure of the coated membrane can be correlated to the formation of ultra-thin ZONYL coated layer.

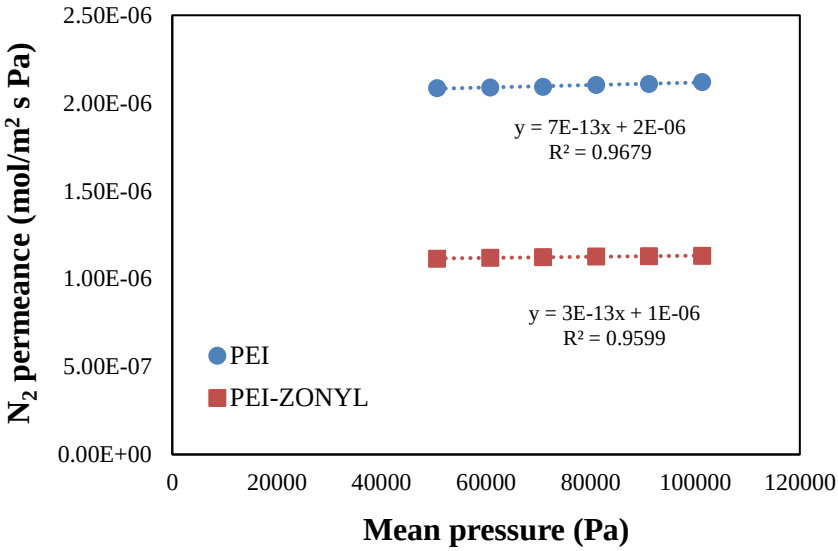


**Figure 4** $N_2$ permeance of the PEI membranes as a function of mean pressure

### 3.2 $CO_2$ Absorption Performance of the PEI Hollow Fiber Membranes

In the present study, an ultra-thin layer of ZONYL was coated on the outer surface of the highly porous PEI hollow fiber membrane in order to enhance the $CO_2$ absorption flux. Figure 5 shows $CO_2$ absorption flux of the membranes as a function of water flowrate in the module. As can be seen, the $CO_2$ flux of both membranes improved when water flowrate in the shell side of the module increased. Since the gas phase mass transfer resistance was minor because of applying pure $CO_2$, the dominant resistance of the liquid phase can be confirmed by the effect of the liquid velocity on the $CO_2$ flux. It can be said that an increase in the liquid velocity can increase mass transfer coefficient for $CO_2$ absorption due to decreasing the boundary layer thickness. The plain PEI membrane showed a slightly higher $CO_2$ flux compared to the coated PEI membrane. This can be related to the formation of coating layer which could reduce the pore size and surface porosity. In fact, a decrease in pore size and surface porosity can reduce the gas-liquid contact area which could decline the $CO_2$ flux. However, the effect of coating layer on the flux reduction was insignificant.

A prolonged gas-liquid membrane contactor operation was performed to evaluate stability of the PEI hollow fiber membranes for $CO_2$ absorption and the results are shown in Figure 6. The plain PEI membrane presented a gradual $CO_2$ flux reduction during over 70 h of the operation, where the flux decreased from $8.1\times10^{-4}$ s to about $4.5\times10^{-4}$ mol/m$^2$ s. This significant flux reduction of about 44% can be correlated to the low surface hydrophobicity and membrane wetting. On the other hand, the coated PEI membrane presented about 12% flux reduction during initial 10 h of the operation and then the flux remained almost stable. The improved properties of the coated membrane with mean pore size of 27 nm, $CEP_w$ of 450 kPa and water contact angle of 105° could considerably reduce pores wetting. The performance of the coated PEI membrane for $CO_2$ absorption by water was compared with the literature and the results are given in Table 4. At the same operating condition, the prepared PEI-ZONYL membrane showed a

higher $CO_2$ flux than the hydrophobic PVDF membrane. The modified PEI membrane [23] showed a higher flux compared to the PEI-ZONYL membrane which can be related to the reported higher absorbent velocity. Therefore, the improved PEI-ZONYL membrane with high hydrophobicity and surface porosity has shown reasonable $CO_2$ flux compared to the literature.

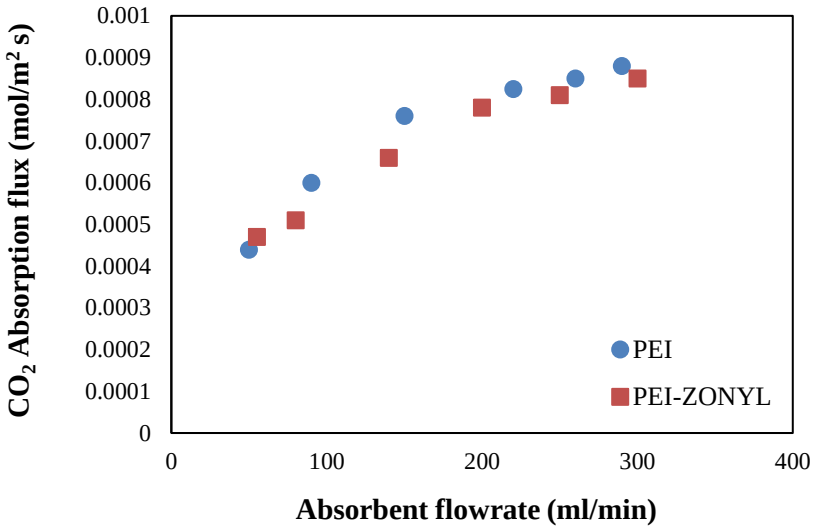


**Figure 5** $CO_2$ absorption flux of the PEI membranes as a function of absorbent flowrate

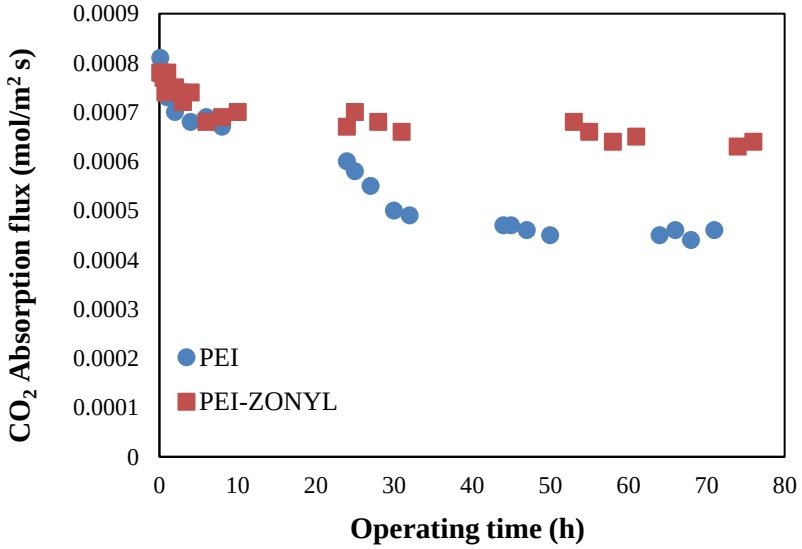


**Figure 6** Long-term $CO_2$ absorption performance of the PEI hollow fiber membranes

**Table 4** Performance comparison of different hollow fiber membranes for $CO_2$ absorption by water

| Membrane | Pore size (nm) | Absorbent velocity (m/s) | $CO_2$ flux (mol/m$^2$ s) | Ref. |
|---|---|---|---|---|
| PVDF | 10 | 0.03 | $7.8\times10^{-4}$ | [22] |
| PEI | 140 | 0.012 | $6.4\times10^{-4}$ | [23] |
| Modified PEI | - | 0.1 | $1.7\times10^{-3}$ | [24] |
| PEI-ZONYL | 27 | 0.03 | $8.6\times10^{-4}$ | Present work |

## 4.0 CONCLUSION

In this work, highly porous PEI hollow fiber membranes were fabricated by a phase inversion process. An ultra-thin ZONYL layer was coated on the outer surface of the PEI hollow fiber membrane in order to enhance hydrophobicity. The prepared membranes were applied in a gas-liquid membrane contactor system for $CO_2$ absorption by distilled water. The PEI-ZONYL membrane presented mean pore size, porosity and contact angle of 27 nm, 81% and 105°, respectively. The plain PEI membrane presented a significant $CO_2$ flux reduction of about 44% during 72 h of the membrane contactor operation. However, the modified PEI-ZONYL membrane showed the flux reduction of about 12%. An almost stable $CO_2$ absorption flux of about $6.8\times10^{-4}$ mol/m$^2$ s was found for the modified PEI membrane during the long-term membrane contactor operation. Therefore, improvement of the membrane properties such as hydrophobicity and surface porosity play important roles in the performance of gas-liquid membrane contactors.

## ACKNOWLEDGEMENT

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