Experimental Investigation and Numerical analysis of SO$_2$ Removal Using Polypropylene Membrane Contactor

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Abstract. In this study, polypropylene membrane contactor is designed for SO$_2$ absorption of simulated flue gas. Water and the aqueous solution of Na$_2$SO$_3$ are used as an absorbent. Absorption performance of the longitudinal module is compared to novel transversal membrane contactor module. The result shows that the transversal configuration of hollow fiber module significantly enhances absorption performance over conventional longitudinal configuration. Absorption flux of SO$_2$ in aqueous Na$_2$SO$_3$ is 1.8 times higher than water at optimum Na$_2$SO$_3$ concentration and gas flow rate. According to performance stability test, polypropylene hollow fiber membrane is successfully operated for 15 hours maintaining average absorption flux of $20 \times 10^{-5}$ mol.m$^{-2}$.s$^{-1}$. Numerical simulation shows good agreement with physical absorption data.

1. Introduction
Nowadays, most of the energy production is made up from the combustion of fossil fuels such as oil, gas, and coal. It is the main source for environmental problems of major concern such as acid rain, photochemical smog, and ozone depletion. To cope with these problems, more stringent emission standards on the acid gasses have been introduced. Generally, well-known packed absorption techniques used for flue gas cleaning are characterized by huge space requirements and high capital cost. Furthermore, conventional wet scrubbing techniques suffer from a number of disadvantages such as loading, flooding, and entrainment [1].

Membrane-based processes can be a suitable alternative to traditional processes. Membrane-based acid gas removal processes have become a feasible alternative to conventional gas separation technologies because of its simplicity and effectiveness. A complete review of membrane-based CO$_2$ separation process from the natural gas stream is also available in the literature [2]. More specific review of the application and development of superhydrophobic membrane contactor for acid gas removal has been conducted by Himma and Wenten[3]. Polymeric membranes are widely used in CO$_2$ removal from the natural gas, but few commercially available polymeric membranes which have both high permeability and high selectivity. The effort in enhancing membrane effectiveness has been done in several ways, such as crosslinking, copolymerization, and phase inversion control [4-8]. Molecular engineering of PIM-1/Matrimid blend was carried out by Yong et al.[5] to produce gas separation membranes. Mixed and pure gas permeation tests were performed on these membranes for CH$_4$ and CO$_2$. The obtained ideal selectivity and CO$_2$ permeability were in the range of 4–36 and 12–3355 respectively.
Recently, hybrid process of solvent absorption and membrane filtration process has gained more attention as an alternative to conventional acid gas recovery. The substitution of the contacting equipment by membrane device intensifies the process while increasing process efficiency and reducing solvent loses. Process efficiency is increased due to membrane compactness, higher interfacial area, and membrane modularity. When hollow fiber membrane is used, fluids are contacted on the opposite side of the fiber (lumen and shell) and the gas-liquid interface forms at the entrance of membrane pore. Due to this contacting mechanism the available contact area is independent of flow rate. This type of membrane can be an attractive alternative to conventional packet tower because of the following reasons: flooding and unloading free, no emulsions, and the differen in density between fluids is not required. Some application of membrane contactor for mass transfer operation including CO₂ absorption, SO₂ absorption, palm oil deacidification, and iodide oxidation [9-11]. Initial investigation on the applicability of membrane-based acid gas absorption has been completed by Wickramasingh et al., [12]. Since then, several investigation results have been published with mostly in lab scale. Further, a related study on factor determining mass transfer resistance has been conducted. These factors are module configuration [13], membrane wetting [13-16], and mass transfer enhancement by chemical reaction [17-18].

Studies on SO₂ absorption using hollow fiber membrane have been reported in literature. Park et al. [19] investigated the absorption of SO₂ using synthesized PVDF hollow fiber membrane. The effect of various operating variables on membrane performance was evaluated and the different absorbent was also compared. The PVDF hollow fiber membrane contactors exhibited a quite high removal efficiency (up to 85 %) with 2 M NaOH solution, while the Na₂CO₃ solution is the most promising absorbent. Applying seawater as the absorbent for SO₂ removal from the simulated flue gas through polypropylene hollow fiber membrane contactor was conducted by Sun et al. [20]. Seawater showed superiority compared with aqueous NaOH solution in SO₂ removal especially for high feed gas concentration and large feed gas flux which indicated that seawater has a large capacity on SO₂ absorption. Modeling of SO₂ absorption using hollow fiber ceramic membrane was simulated by Luis et al. [21]. The negative effect of membrane wetting on mass transfer was studied and confirmed in mathematical modeling. In addition, the model also allowed to obtain the best operation condition and solvent selection by incorporating solvent affinity. More recently, experimental study on simultaneous removal of CO₂ and SO₂ was conducted by applying polypropylene hollow fiber membrane as contactor and monoethanolamine (MEA) as absorbent [22]. The experiment results indicated that the membrane could eliminate both components from flue gas. Membrane wetting was also observed during prolonged operation which resulted in the reduction of mass transfer rate. Moreover, increasing gas phase pressure temporarily was found to be effective method to retrieve mass transfer rate. In membrane-based SO₂ absorption, turbulence affects significantly on absorption performance since it controls mass transfer resistance on gas and liquid film. Therefore, flow configuration is an important parameter for gas absorption. Investigation in transversal flow configuration of hollow fiber membrane contactor in CO₂ absorption process was previously reported by Rahmawati et al. [13].

The aim of this study is to investigate the potential of polypropylene membrane contactor in SO₂ removal from synthetic flue gas. In order to get a better understanding of the effect of flow configuration on absorption performance, conventional longitudinal flow membrane contactor is compared to novel transversal flow membrane contactor. In addition, physical and chemical absorption of SO₂ is performed using water and aqueous Na₂SO₃ solution, respectively. Numerical simulation is also conducted to study mass transport inside of membrane module.

2. Numerical Simulation

2.1. Governing equation on gas phase

Differential mass balance equation on gas phase is generated by considering a small cylindrical tube with the porous wall. A mass transfer mechanism is the combination of the convective and diffusive
mechanism. Fig. 1. depict 2-dimensional domain of numerical calculation used in this study.

Fig. 1. The axial and radial coordinate of hollow fiber (a) and free surface approximation (b)

Steady-state mass balance for SO$_2$ on gas phase which consists of the diffusive term and the convective term is described by the following equation:

$$D_{AB} \left[ \frac{\partial C_G}{\partial r^2} + \frac{1}{r} \frac{\partial C_G}{\partial r} \right] - V_Z \frac{\partial C_G}{\partial z} - S_R = 0$$  \hspace{1cm} (1)

Where $D_{AB}$ is binary diffusion coefficient of SO$_2$–N$_2$ in gas phase, $C_G$ is SO$_2$ concentration in the gas phase, $V_Z$ is the linear velocity of gas and $S_R$ is source term for reaction. Mass balance equation should be solved simultaneously with momentum equation for convective effect. With the assumption of fully developed laminar flow, velocity profile on tube side is:

$$V_Z = 2V_{m} \left[ 1 - \left( \frac{r}{R_1} \right)^2 \right]$$  \hspace{1cm} (2)

Where $V_m$ is maximum linear velocity on the tube.

The boundary condition for the system is given by,

at $z = 0$, $r = R_1$, $C_G = C_o$  \hspace{1cm} (3)

at $z = z$, $r = R_1$, $C_G = C_M$  \hspace{1cm} (4)

at $z = z$, $r = 0$, $\frac{\partial C_G}{\partial r} = 0$ (no flux)  \hspace{1cm} (5)

Continuity of flux at the interface between phase and membrane is described by:

at $r = R_1$

$$-D_{AB} \frac{\partial C_G}{\partial r} = -D_M \frac{\partial C_M}{\partial r}$$  \hspace{1cm} (7)

at $r = R_2$

$$-D_M \frac{\partial C_M}{\partial r} = -D_{12} \frac{\partial C_L}{\partial r}$$  \hspace{1cm} (8)
Where $D_M$ is diffusion coefficient of SO$_2$ in membrane pore which is the combination of the binary diffusion coefficient and Knudsen diffusivity and $D_{AB}$ is binary diffusion coefficient for SO$_2$–N$_2$ system.

2.2. Governing equation on the membrane

Naturally, mass transport of SO$_2$ from the gas phase to liquid phase inside membrane pore is mainly due to molecular diffusion. Then the differential mass balance of SO$_2$ is generated by removing convective term and written as:

$$D_M \left[ \frac{\partial C_M}{\partial r^2} + \frac{1}{r} \frac{\partial C_M}{\partial r} \right] = 0$$  \hspace{1cm} (9)

The boundary condition for the system on mass transfer domain is given by:

at $z = z$ and $r = R_1$, $C_M = C_G$ \hspace{1cm} (10)

at $z = z$ and $r = R_2$, $C_M = \frac{C_L}{m}$ \hspace{1cm} (11)

Where $m$ is solubility of SO$_2$ in the aqueous solution of Na$_2$SO$_3$.

2.3. Governing equation on liquid phase

Steady-state mass balance for the differential element in shell side where liquid absorbent flows is given by:

$$D'_L \left[ \frac{\partial C_L}{\partial r^2} + \frac{1}{r} \frac{\partial C_L}{\partial r} \right] - V_z \frac{\partial C_L}{\partial z} - SR = 0$$ \hspace{1cm} (12)

By adopting Happel free surface approximation, the governing velocity profile on shell side is written as:

$$V_z = 2V_{\text{max}} \left[ 1 - \left( \frac{R_2}{R_1} \right)^2 \right] \left[ \frac{\left( R_2/R_3 \right)^2 - \left( R_2/R_3 \right)^2 + 2 \ln \left( R_2/R_3 \right)}{3 + \left( R_2/R_3 \right)^4 - 4 \left( R_2/R_3 \right)^2 + 4 \ln \left( R_2/R_3 \right)^2} \right]$$ \hspace{1cm} (13)

With boundary condition is given by:

at $z = 0$ and $r = r$, $C_L = C_{L_0}$ \hspace{1cm} (14)

at $z = L$ and $r = r$, $\frac{\partial C_L}{\partial r} = 0$ (no flux) \hspace{1cm} (15)

at $z = z$ and $r = R_2$, $C_L = mC_M$ \hspace{1cm} (16)

at $z = z$ and $r = R_3$, $\frac{\partial C_L}{\partial r} = 0$ (no flux)

Where $m$ is solubility of SO$_2$ in the aqueous solution of water.

In this study, two-dimensional analysis of membrane-based SO$_2$ absorption is conducted. Set of differential mass balance equation on liquid phase, membrane and gas phase is solved simultaneously by COMSOL MULTIPHYSICS solver with appropriate boundary condition. COMSOL MULTIPHYSICS solves the partial differential equation by finite element approach.
3. Experimental setup
In this study, hydrophobic polypropylene (GDP Filter Indonesia) membrane is used as contactor. The properties of hydrophobic polypropylene membrane are listed in Table 1. SEM micrograph of hydrophobic polypropylene membrane used in this experiment can be seen in Fig. 2. The configuration of novel transversal membrane contactor is described in Fig. 3. An aqueous solution of Na₂CO₃ (0.18N and 0.25N) and pure water is used as a solvent. Gas-filled pore operation is applied in this experiment since it offers the lowest mass transfer resistance. During experimental test, gas is always flow in lumen side and gas in lumen side since it provides maximum surface area.

Fig. 2. SEM micrograph of hydrophobic polypropylene membrane surface

Fig. 3. The configuration of a) longitudinal and b) transversal membrane
Table 1. Properties of membrane contactor used in this study

| Property                        | Value       |
|---------------------------------|-------------|
| Membrane material               | Polypropylene|
| Pore diameter (µm)              | 0.2         |
| Membrane porosity (%)           | 70          |
| Fiber O.D. (µm)                 | 541         |
| Fiber I.D. (µm)                 | 235         |
| Membrane wall thickness (µm)    | 153         |
| Membrane effective length (mm)  | 180         |
| Number of fiber                 | 145         |
| Module I.D. (mm)                | 12          |

The experimental set up is shown in Fig. 4. A feed gas containing SO₂ diluted with N₂ is introduced to the system from the pressurized gas cylinder. The concentration of SO₂ on gas stream is maintained < 2%. Feed gas flow is controlled and measured with a regulator valve and precision rotameter. Pressure gauges are installed at the contactor inlet and outlet indicates the gas pressure. Liquid absorbent (pure water or aqueous Na₂SO₃ solution) is pumped from the solvent reservoir by using the peristaltic pump. The instrumentation in the liquid line for the control and measurement of flow and pressure is similar to that in the gas line. Liquid pressure is maintained slightly higher than the gas pressure to prevent bubble formation. The system is first operated for at least half an hour to ensure that a steady state has been reached before collecting data. All the experiments are conducted at room temperature.

Iodimetric reversed titration is used to quantify the amount of captured SO₂. In this method, the sample containing SO₂ is reacted with I₂ where I⁻ will be liberated during the reaction. The amount of I⁻ is equivalent to SO₂ in the liquid phase. Determination of I⁻ is conducted by titration of the solution with a standardized Na₂S₂O₃ solution until the equivalent point is reached. Two replication is applied for all of the samples.

4. Result and Discussion

4.1. Effect of operating condition

Turbulence is the most important operating variable in the membrane-based acid gas absorption process since it has an obvious effect on SO₂ absorption flux. Fig. 5 shows the experimental results of SO₂ absorption flux as a function of gas velocity and liquid velocity on chemical absorption where
turbulence parameter is characterized by Reynold number ($N_{Re}$). It is observed that absorption rate of SO$_2$ increases significantly with the increase of Reynold number. This behavior is expected because the boundary layer thickness of the gas phase in lumen side decreases with increase in turbulence, which leads to the decrease in mass transfer resistance on gas boundary layer. Higher gas flowrate also provides higher concentration difference between phase. The same behavior is also observed on the liquid side. This condition indicates that mass transfer process is controlled by both of gas and liquid mass transfer resistance.

![Graph](image1.png)

Fig. 5. Effect of gas flow rate on SO$_2$ absorption flux in various liquid flowrate (Na$_2$SO$_3$ concentration = 0.25 N; longitudinal module)

4.2. Effect of flow configuration

Comparison between two different flow configuration on membrane-based SO$_2$ absorption can be seen in Fig. 6. SO$_2$ Absorption flux in the transversal module and the longitudinal module is compared. The result shows that SO$_2$ absorption flux in the transversal module is higher than the longitudinal module in all absorbent velocity tested indicated by $J_{trans}/J_{long} > 1$. This result is expected since in the transversal module configuration the flow is perpendicular to the fibers cause greater turbulence than the longitudinal module. The perpendicular flow causes a higher degree of circulation eddy. Moreover, the boundary layer is shorter and equivalent to the circumference of membrane fiber resulting lower mass transfer resistance and higher absorption flux. Whereas in the longitudinal module, the flow is parallel to the fiber and hence the boundary layer equivalent to the fiber length.

![Graph](image2.png)

Fig. 6. Performance comparison of longitudinal membrane contactor to novel transversal membrane contactor on physical absorption
4.3. Chemical vs physical absorption

The comparative analysis of absorption performance between chemical and physical absorption is described in Fig. 7. The use of reactive absorbent enhances mass transfer of SO$_2$ and increases its scrubbing capacity. As the result, for equivalent SO$_2$ removal, the required absorbent flowrate can be significantly reduced. The reaction involved in SO$_2$ removal using N$_2$SO$_3$ aqueous solution is shown in equation 16 (Park et al., 2008). According to Fig. 7, absorption flux of SO$_2$ in aqueous Na$_2$SO$_3$ is 1.8 times higher than water at optimum Na$_2$SO$_3$ concentration and gas velocity. It is also observed that SO$_2$ removal of chemically enhanced absorption is initially increased then decrease further after passing optimum point. At high velocity, increase in gas flow rate will reduce gas residence time leading to decrease in removal efficiency. In case of physical absorption, SO$_2$ removal is decreased along with gas flow rate due to its small loading capacity.

$$SO_2 + Na_2SO_3 + H_2O \rightarrow 2NaHSO_3$$

(17)

Fig. 7. Comparison of SO$_2$ absorption flux and % SO$_2$ removal in physical absorption and chemical absorption (liquid flowrate = 0.125 cm$^3$.s$^{-1}$; longitudinal module)

According to Fig. 8 it is observed that SO$_2$ absorption flux increase significantly with the increase of Na$_2$SO$_3$ concentration. This condition is expected since reaction rate between Na$_2$SO$_3$ and SO$_2$ is enhanced by the increase of Na$_2$SO$_3$ concentration lead to higher SO$_2$ flux. In addition, a higher concentration of Na$_2$SO$_3$ will increase the absorption capacity of the absorbent. However, it should be noted that the higher concentration of solvent employed resulting in higher cost of solvent and energy required for regeneration.
Fig. 8. Effect of Na$_2$SO$_3$ concentration on SO$_2$ absorption flux (liquid flowrate = 0.125 cm$^3$.s$^{-1}$; longitudinal module)

4.4. Simulation result

Film theory has been widely used to describe mass transfer on membrane contactor process. In chemically enhanced gas absorption, mass transfer from the gas phase to liquid phase is driven by concentration difference of chemically active component between phase. The film theory assumes that mass transfer resistances are in series and are represented by the sum of the reciprocals product of the individual mass transfer coefficients. The overall mass transfer coefficient may then be written as:

$$\frac{1}{K_{ov}} = \frac{1}{Hk_G \ d_o} + \frac{1}{Hk_M \ d_m} + \frac{1}{E k_L}$$

(15)

Where $H$ represents Henry’s constant, $K_{ov}$ is overall mass transfer coefficient, $k_G$, $k_M$, $k_L$ is individual mass transfer coefficient on gas phase, membrane and liquid phase respectively, $d_i$ is inside diameter of the fiber, $d_o$ is outside diameter of the fiber, $d_m$ is log mean diameter of the fiber and $E$ is enhancement factor due to chemical reaction. Comparison between experimental and calculated value of $K$ is shown in Fig 10. The calculated value is in good agreement with experimental data on physical absorption. On the other side, the calculated value of $K$ is far lower than the experimental value at chemical absorption.
Fig. 9. Comparison overall mass transfer coefficient between experimental and calculated (liquid flow rate = 0.125 cm$^3$.s$^{-1}$; longitudinal module)

The contour of SO$_2$ concentration on the liquid phase is depicted on Fig. 10 with arrow shape indicates total flux. Liquid absorbent (water) flows in the shell side of hollow fiber membrane from $(z/L) = 0$ in the parallel direction to gas phase. SO$_2$ is transported from gas phase to liquid phase due to concentration difference between gas and liquid. The highest concentration of SO$_2$ is found on gas-liquid interface where initial dissolution takes place.

Fig. 10. Concentration of SO$_2$ on liquid phase (liquid flowrate = 0.125 cm$^3$.s$^{-1}$; longitudinal module)

Contour of SO$_2$ concentration on liquid phase at various gas velocity is also presented in Fig. 10. The amount of SO$_2$ captured by water increases along with gas velocity which is expected since it’s provide higher concentration difference. This behavior is also validated by experimental data. Effect of gas inlet velocity and gas initial concentration is shown in Fig. 11. SO$_2$ absorption flux increase along with inlet velocity which is agree with experimental data (Fig. 11.a). Expected behavior is also found for variation of initial SO$_2$ concentration where absorption flux increases due to larger concentration difference between phases (Fig. 11.b).
4.5. Performance stability test

Fig. 12 shows the experimental results of SO$_2$ absorption in membrane contactor over 15 h. The liquid absorbents flowed in the shell side and SO$_2$ in the lumen side of the polypropylene hollow fiber membrane at ambient temperature. During the tests, absorption flux of SO$_2$ is maintained steadily over $20 \times 10^{-5}$ mol.m$^{-2}$.s$^{-1}$. Hydrophobic nature of polypropylene hollow fibers membrane prevents the penetration of liquid absorbent into membrane pores. It prevents additional mass transfer resistance due to entrapment of liquid inside membrane pores.

5. Conclusion

Polypropylene hollow fiber membrane contactor is designed for absorption of SO$_2$ from synthetic flue gas. The effect of mass transfer enhancement by chemical reaction is also investigated by comparing water and the aqueous solution of Na$_2$SO$_3$. The result shows that the absorption performance is controlled by both of gas and liquid flow rate. In comparison with the longitudinal module, transversal module shows potential with higher absorption performance. Significant enhancement of SO$_2$
absorption flux is achieved by chemical absorbent with absorption flux 1.8 times higher than water in optimum gas velocity. The membrane contactor is successfully operated for 15 h maintaining average SO\textsubscript{2} absorption flux of 20 x 10\textsuperscript{-5} mol.m\textsuperscript{-2}.s\textsuperscript{-1}. It is also observed that simulation result on absorption flux is in a good agreement with experimental data.

**Nomenclature**

- \( C_G \): SO\textsubscript{2} concentration in liquid phase (mol.l\textsuperscript{-1})
- \( C_L \): SO\textsubscript{2} concentration in liquid phase (mol.l\textsuperscript{-1})
- \( C_M \): SO\textsubscript{2} concentration in liquid phase (mol.l\textsuperscript{-1})
- \( D_{12} \): diffusion coefficient of SO\textsubscript{2} in Na\textsubscript{2}SO\textsubscript{3} aqueous solution (m\textsuperscript{2}.s\textsuperscript{-1})
- \( D_M \): diffusion coefficient of SO\textsubscript{2} in membrane pore (m\textsuperscript{2}.s\textsuperscript{-1})
- \( D_{AB} \): binary diffusion coefficient for SO\textsubscript{2} – N\textsubscript{2} system (m\textsuperscript{2}.s\textsuperscript{-1})
- \( K_{ov} \): overall mass transfer coefficient (m.s\textsuperscript{-1})
- \( k_G \): individual mass transfer coefficient on gas phase (m.s\textsuperscript{-1})
- \( k_M \): individual mass transfer coefficient on membrane phase (m.s\textsuperscript{-1})
- \( k_L \): individual mass transfer coefficient on liquid phase (m.s\textsuperscript{-1})
- \( m \): solubility of SO\textsubscript{2} in Na\textsubscript{2}SO\textsubscript{3} aqueous solution
- \( N_{Re} \): Reynold number
- \( r \): radius of fiber (m)
- \( S_R \): source term for reaction
- \( V_z \): linear velocity of liquid (m.s\textsuperscript{-1})

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