CO₂ Separation by Using a Three-stage Membrane Process

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ABSTRACT

This work proposed and optimized a three-stage membrane process for CO₂ separation. The results of this study revealed that the membrane technology is a suitable process for the CO₂ separation in a higher concentration. In addition, the MATLAB was used to simulate and obtain the optimal operational parameters for a three-stage membrane process. This work established a partial cycle and recovered the CO₂ from the permeation side of second-stage membrane that enhance a higher purity CO₂ gas stream. The results of this study indicated that when the CO₂ concentration was higher than 50% and at a flow rate of 100000 Nm³ d⁻¹, the CO₂ separation could be achieved at the optimal operation condition. Under the conditions that the membrane areas were 2400, 3800, and 1800 m² for the first-, second-, and third-stage membrane, respectively and the operational pressure at first- and third stage membrane were 3.0 and 2.5 MPa, respectively, the CO₂ separation fraction was higher than 90% and CH₄ loss rate was lower than 5%. The results of this study have a high potential for the practical application.

Keywords: Optimal design; CO₂-EOR extraction gas; CO₂ capture; Multi-stage membrane separator.

INTRODUCTION

As the pace of oil exploitation was accelerated due to the importance of oil, more and more oilfields have the characteristics of lower permeability because of mining and geological reasons. Generally, the recovery efficiency of low permeability reservoirs is only 20 to 25% in the oilfield, the proportion of low permeability reservoirs in proving oil reserves increases year by year (Wei et al., 2018). There are a lot of flooding patterns, of which CO₂-EOR is more available than water flooding, nitrogen, air and flue gas, which has the advantages of low cost and high natural gas quality (Booran et al., 2016; Wang et al., 2017; Bender and Akin, 2017; Wang et al., 2018). However, CO₂-EOR flooding will lead to a large number of CO₂ (about 40 to 60% of the injected gas) spilling out of the ground along with the gas produced during oil recovery (hereinafter referred to as the extraction gas). Meanwhile, with continuous exploration, a number of oilfields with a higher CO₂ content are presented. For example, the CO₂ concentration in extraction gas fields in Malaysia ranges from 28% to 87% (Tan et al., 2012b; Jean et al., 2016; Xie et al., 2017; Yang et al., 2019). If large amount of CO₂ will be discharged directly into the atmosphere, it will not only cause serious climate problems, but also can be harmful to human health (Ping et al., 2018; Shiue et al., 2018; Tsai et al., 2018). Therefore, it is serious issue for CO₂ capture from extraction gas.

There has been developed several CO₂ capture technologies, such as absorption, adsorption, membranes, cryogenic in the last decades (Sreedhar et al., 2017; Vinoba et al., 2017). The chemical absorption method is to remove the CO₂ from the extraction gas through the convection contact between the feed gas and the chemical solvent in the packing column with the usual solvent being monoethanolamine (MEA), diethanolamine (DEA), methyldiethanolamine (MDEA), etc. (Tan et al., 2012a; Li et al., 2016). This method, however, gradually reveals a number of drawbacks of large volume occupancy and some operational problems such as flooding, channeling, entrainment and foaming (Ghasem et al., 2012a). Chemical absorption technology is usually used to process extraction gas within a relatively narrow range in feed (Fu et al., 2012). So in the case of high CO₂ concentration (about 60% or more) at 0.3–0.6 MPa, the chemical method may not be applicable due to excessive circulating amount of poor absorption effect. Holmes and Ryan (1982) first invented cryogenic distillation for natural gas purification, and it also uses for CO₂ capture. However, due to the high energy consumption of this technology, which accounts for about 50% of the total energy, this technology has not been widely used in CO₂ capture from extraction gas (Ebrahimzadeh et al., 2016).

In recent years, membranes for CO₂ separation are receiving growing attention for application in the field of...
CO₂ capture and storage (CCS) (Boot-Handford M et al., 2014; Roussanaly S et al., 2016). Compared with chemical absorption, membrane-based separation is more attractive for its easy installation, minimal influence of SO₂ and NOₓ on membrane materials and avoidance of regeneration energy consumption (Merkel et al., 2010; Rufford et al., 2012). In order to have a good development prospect, the membrane-based method must develop “good” membrane modules and membrane materials. Robeson (2008) has identified the upper bound on membrane material selectivity and permeability in many gas separation systems, including CO₂/N₂, O₂/N₂, CO₂/CH₄ and H₂/CH₄ etc. In the case of the membrane separation process, the membrane material is the first element because the selectivity and permeability of the membrane directly determine the separation efficiency. It is well known that performance of polymeric membranes is characterized by an ‘upper bound’ that correlates permeability and selectivity (Robeson, 2008). However, in recent years, many new materials, such as MOFs and ssz-13, have also been applied in the membrane separation field, with good performance in separation performance and permeability (Rodenas, 2015; Chisholm et al., 2018). The gas permeability through the polymer membrane depends on the solution-diffusion mechanism in which the rate of gas movement correlates with the ratio between the gas molecules and the polymer material and the diffusion rate (Rufford et al., 2012). Polyimide is a very attractive polymer that can be used in membrane technology, having high selectivity and high permeability, as well as various applications such as gas and liquid separation (Favvas et al. 2017).

In order to obtain high quality product features, the multi-stage membrane separation device is proposed to multi-separate the product gas to improve the purity of the product instead of single-stage membrane device which has low cost but poor performance. The experiments of CO₂ capture from natural gas in single-stage membrane device were conducted under high pressure conditions or efficient absorbents, studying the influence of membrane area, membrane pressure and feed gas flow rate on CO₂ removal rate (Kang et al., 2017). Few studies have been done on multi-stage membrane devices, for example, O₂ from air, CH₄ from biogases and landfill, CO₂ from coal flue gases and H₂ from H₂/CO mixtures (Rautenbach et al., 1987; Bhide et al., 1991; Xu et al., 1996; Zhao et al., 2011). Ohs et al. (2016) recently applied superstructure method to N₂/CH₄ from natural gas to identify the optimal process and structure parameters. Not only did they not fully consider the membrane separation structure and overall operation parameter optimization, but they also did not have the overall process design of CO₂/CH₄ system. Chong (2017) proposed a polymeric membranes for O₂/N₂ gas separation and through using N,N-dimethylacetamide (DMAc) and tetrahydrofuran (THF) and ethanol as additive, a Polysulfone (PSF) hollow fiber membrane was created. This membrane can achieve better O₂/N₂ separation rate.

With reference to single-stage membranes and literature, we propose a three-stage membrane separation process based on this. In this study, CO₂ capture performance of membrane separator was investigated using extraction gas with high concentration CO₂ (60%) as feed. A mathematical model was established and introduced into Matlab for numerical simulation of the membrane separation. The effect of operating pressure and membrane area on CO₂ recovery fraction and CH₄ loss rate were discussed for optimal parameters. It will provide guidance for the application of multi-stage membrane separator in the CO₂ capture of extraction gas.

**METHODOLOGY**

**Extraction Gas**

We have injected CO₂ into the underground oil layer since 2012. In the next six years, the change of CO₂ concentration over time in Shengli oilfield and CO₂ concentration were sostenuto monitored in the extraction gas which is shown in Fig. 1. As showed in Fig. 1, in the whole process of oil recovery after CO₂ injection, the CO₂ content in the extracted gas increased from the original 1.5% to above 90%. CO₂ content in Well89 1–7 increased sharply in the initial phase and quickly rose to the second phase, while Well89 S1 remained stable at low content for a long time in the initial phase. But after a period of time, the CO₂ concentration in both wells rose rapidly to the third phase, where the concentration range was stable at 60%–90%. After a few years, the total gas volume may increase tenfold compared with the gas injection cause of large fluctuations of CO₂ gas, which will bring technical difficulties to separation and further treatment.

Results related to heavy hydrocarbon components are demonstrated in Fig. 2. Heavy hydrocarbon content of C₅⁺ is relatively stable, at about 2%–3%, but the content of C₇⁺ is volatile, as high as 15%. Therefore, it is necessary to design a special pretreatment module to remove these. The content of C₅⁺ may contaminate membrane devices which cause membrane material poisoning. Therefore, a special pretreatment module should be designed to remove heavy hydrocarbons, which will be carried out in the future work.

According to the content of CO₂ in Fig. 1, we divided the produced gas into I area (CO₂ concentration< 30 mol%), II area (30 mol% < CO₂ concentration < 60 mol%), III area (CO₂ concentration > 60 mol%). In conclusion, the extraction gas in III area has the following characteristics: large gas flow; high partial pressure of CO₂; high CO₂ concentration (60% or greater); the main components are CO₂ and CH₄. For CO₂ concentration above 60%, our primary work and Kang, G (2017) recommend membrane separation, which is also the key part of this work.

**Process Description**

The process of membrane-based separation was displayed in Fig. 3. The extraction gas is first processed through the pretreatment module, in which the liquid water, heavy hydrocarbons and solid particles carried in the EOR extraction gas are removed. Otherwise, the membrane components will be blocked and the membrane materials will be contaminated, which will affect the normal operation of the membrane system. Since the research on the pretreatment process was not mature, the design was not discussed in this paper. The gas (material 1) that is passed through the pretreatment system was then compressed into the first-stage membrane.
device for gas separation. Furthermore, the gas was split into two streams, one is permeate gas loaded with CO$_2$ (material 2), and the other is entrapped gas loaded with CH$_4$ (material 3). The former enters the third-stage membrane separator after being pressurized in compressor for further purification, the latter directly enters the second-stage membrane for purification of CH$_4$ gas. The permeate gas stream treated by the third-stage membrane separator was the CO$_2$ product gas (material 4) while the stream generated by the second-stage membrane was CH$_4$ product gas (material 5). However, the permeate gas of the secondary membrane contained in CO$_2$ (material 6) was designed to mix with the inlet gas to form a partial circulation, because of the concentration of CO$_2$ was similar to that of the feed gas. The entrapment side of

*Fig. 1.* CO$_2$ concentration of extraction gas in Well-89 in Shengli oilfield.

*Fig. 2.* Pie diagrams of extraction gas component.
the third-stage membrane was discharged as exhaust gas (material 7). The module used in the design is hollow fiber which is equivalent to the mass transfer of a shell and tube heat exchanger (Mat et al., 2014). Such an assembly can significantly increase the performance of the membrane by increasing the chemical potential difference across the membrane. Polyimide membrane was selected as the membrane material, and the separator parameters were referred to Robeson (2008). The design parameters were selected as shown in Table 1.

For the membrane-based capture, the main factors that affect the CO₂ recovery fraction and CH₄ loss rate in the whole process are two, one is the membrane area, the other is the membrane pressure, which are also the two objects we discussed. The membrane area includes first-, second- and third-stage membrane area. The CO₂ concentration in the purified gas is mainly affected by the area of second-stage membrane, while that in the captured gas is mainly affected by the area of tertiary membrane. Therefore, the area of the second- and third-stage membranes is tentatively determined to optimize the area of the primary membrane. After analyzing the influence of first-stage membrane area on the CO₂ concentration in purified gas and the concentration of CO₂ product gas, the first-stage membrane area was obtained, and then the second-stage membrane area and the third-stage membrane area were optimized. The operating pressure is the outlet pressure of the compressor, which directly affects the pressure of the feed gas. If the pressure of the feed gas is increased, the faster the gas passes through the membrane, the higher the CO₂ recovery fraction and the smaller the membrane area.

**Process Simulation**

**Membrane Unit Model**

As shown in Fig. 4, the counter-current flow pattern was developed in this study with the concept of tank-in-series (Huang et al., 2018; Lee et al., 2018). This ideal model does not take into account the pressure drop, concentration polarization and scaling on the residual side. Simulation optimization conditions are achieved by simulink component optimization in Matlab, which is a reliable calculation method.

Mass differential equation:

\[ dU = dV \]  \hspace{1cm} (1)

Differential equation of flow:

\[ d(Ux) = d(V_y) \]  \hspace{1cm} (2)

Differential equation for change of CO₂ concentration:

\[ \frac{d(U_x)}{dl} = -J_{CO_2} \frac{A}{L} (P_x - P_y) \]  \hspace{1cm} (3)
The separation module with counter-current flow pattern.

Differential equation for change of CH$_4$ concentration:

$$\frac{d[U(1-x)]}{dl} = -J_{CH4} A \left[ p(1-x) - p(1-y) \right]$$

(4)

where $U$ is the flow of feed gas, Nm$^3$ h$^{-1}$; $V$ is flow of permeate gas, Nm$^3$ h$^{-1}$; $X$ is the concentration of CO$_2$ in the feed gas; $Y$ is the concentration of CO$_2$ in permeate gas; $P_h$ is the pressure of feed gas, MPa; $P_l$ is permeate pressure, MPa; $A$ is membrane area, m$^2$.

With boundary conditions: $l = 0$; $U = U_0$; $x = x_0$; $I = L$; $V = 0$; $x_L = y_L$.

The following was the integral from the entry ($l = 0$) of the separator to any section of Eq. (3):

$$y = \frac{-U_0x_0 + V_0y_0 + Ux}{V}$$

(5)

Using Eq. (3), the permeate gas concentration of each cross section in fiber bundles ($l \neq L$) can be obtained. Because when $L = 1$, $V = 0$, the permeation gas concentration of $y_L$ cannot be directly obtained from Eq. (3). Therefore, $y_L$ is now defined as:

$$y_L = \lim_{l \to L} y = \lim_{l \to L} \frac{-U_0x_0 + V_0y_0 + Ux}{V}$$

(6)

When $l \to L$, $y_L$ is defined as:

$$y_L = \frac{J_{HI} \left( P_Lx_L - p_Ly_L \right)}{J_{HI} \left( P_Lx_L - p_Ly_1 \right) + J_{L1} \left( P_L(1-x_L) - p_L(1-y_L) \right)}$$

(7)

The simulation results can be obtained by means of differential equations and boundary conditions through MATLAB.

Mass Balance Equations for the Three-stage Membrane Process

Binary variables are used in this study to represent the presence or absence of any structural options in this optimization. Additionally, Genetic algorithm (GA) is applied in the simulation, which can well obtain the results of variable calculation in the simulation environment of MATLAB (Lee et al., 2018). The mass equilibrium and composition equilibrium based on binary variables are defined as follows:

Flow rate mass balance for the first membrane stage is:

$$F_i = F_{feed} + \sum_{s=1}^{N} \sum_{n=1}^{N} \left( S_{x,s;n} F_{x,s,n} + S_{p,s;n} F_{p,s,n} \right)$$

(8)

Flow rate mass balance for the second- and third-stage membrane stage is:

$$F_a = \sum_{s=1}^{N} \sum_{n=1}^{N} \left( S_{x,s;n} F_{x,s,n} + S_{p,s;n} F_{p,s,n} \right)$$

(9)

where $F_i$ is the flow rate, Nm$^3$ h$^{-1}$; $F_{feed}$ is the flow rate of feed gas, Nm$^3$ h$^{-1}$; $F_e$ is the flow rate of entrapped side, Nm$^3$ h$^{-1}$; $F_p$ is the flow rate of permeate side, Nm$^3$ h$^{-1}$; $s$ is splitter numbers; $n$ is the stage number of membrane; $S_p$ is the binary variable for flow connection in permeate side; $S_e$ is the binary variable for flow connection in entrapped side; $N$ is the overall number of membrane stage.

Composition balance for the first membrane stage is:

$$\sum_{i=1}^{N} \sum_{n=1}^{N} \left( S_{x,s;n} f_{x,s,n} + S_{p,s;n} f_{p,s,n} \right) = \frac{f_{i}}{F_i}$$

(10)

Composition balance for the second and tertiary membrane stage is:

$$\sum_{s=1}^{N} \sum_{n=1}^{N} \left( S_{x,s;n} f_{x,s,n} + S_{p,s;n} f_{p,s,n} \right) = \frac{f_{e}}{F_a}$$

(11)

where $f_i$ is the mole fraction of component; $f_{feed}$ is the mole fraction of each component at overall feed flow rate; $f_e$ is the mole fraction of component of entrapped side; $f_p$ is the mole fraction of component of permeate side.

Power Consumption

Power consumption is an important parameter for evaluating CO$_2$ capture performance. However, the power requirement of the three-stage membrane process is due to compressor, so the calculation of power consumption is shown as follows (Song et al., 2017):

$$W_{compressor} = E_{membrane}$$

(12)

RESULTS AND DISCUSSION

Effects of Membrane Area

First-stage Membrane Area

The optimal first-stage membrane area was obtained by
changing the membrane area from 2300 to 2700 m², getting the CO₂ concentration in outlet gas stream, and measuring the CO₂ recovery fraction and the CH₄ loss rate. On the experience of previous studies, the membrane area of second- and third-stage were 3500 and 2000 m², respectively.

In Figs. 5 and 6, an increase of first-stage membrane area did affect the separation efficiency of CO₂/CH₄. It was negatively correlated with the CO₂ recovery fraction and CO₂ concentration, and positively correlated with the CH₄ loss rate. The above results indicated that an increase of membrane area led to hinder the separation of CO₂ and CH₄.

When the first-stage membrane area was greater than 2500 m², the CH₄ loss rate was higher up to 5%. In order to lower the CH₄ loss rate, the first-stage membrane area must be less than 2500 m². However, if considering both CO₂ recovery fraction and CH₄ loss rate, the first-stage membrane area must be selected as 2400 m², which has a 98.6% CO₂ recovery efficiency and 4.2% CH₄ loss rate.

**Second-stage Membrane Area**

After the first-stage membrane, the CO₂ concentration needs to be further purified. If the first- and third-stage membrane area were selected as 2400 and 3000 m², respectively and the second-stage membrane area was varied from 2000 to 4000 m² for obtaining an optimal membrane area of second-stage.

The main purpose of second-stage membrane is to obtain a higher concentration of CH₄ gas. Therefore, the lower the CO₂ permeate concentration is the better. In Fig. 8, it showed that a higher second-stage membrane area did decrease the permeate CO₂ concentration from 3% to less than 0.5%. In order to ensure that permeate CO₂ concentration be less than 2%, the second-stage membrane area was set to be 3800 m².

![Fig. 5. CO₂ concentration along with the change of first-stage membrane area.](image1)

![Fig. 6. CO₂ recovery fraction and CH₄ loss rate along with the change of first-stage membrane area.](image2)
Third-stage Membrane Area

When the first- and second-stage membrane area were selected to be 2400 and 3800 m², respectively, the optimal membrane area of the third-stage was determined by looking the data of CO₂ recovery fraction and CH₄ loss rate.

In Fig. 8, it showed that an increase of third-stage membrane area resulted in a CO₂ recovery fraction increased, which was rising from 50% to higher than 95% and finally tended to be stable.

When the third-stage membrane area was higher than 2000 m², the CO₂ recovery fraction and CH₄ loss rate were both stable and at around 97% and 4.5%, respectively. However, when the membrane area was greater than 2000 m², the CH₄ loss rate declined linearly and rapidly. Under the condition of third-stage membrane area was 1800 m², CO₂ recovery fraction and CH₄ loss rate can reach 90.5% and 4.45%, respectively.

Effects of Operating Pressure

Operational Pressure at First-stage Membrane

When the CO₂ concentration in inlet gas stream were 0.5, 0.6, 0.7 and 0.8 and the operating pressure at first-stage membrane was set at 1.5, 2.0, 2.5, 3.0 and 3.5 MPa, respectively. The variation of CO₂ recovery fraction and CH₄ loss rate by the first-stage membrane was calculated and analyzed.

In Fig. 9, it showed that as the operational pressure of first-stage membrane continued to rise, both the CO₂ recovery fraction and CH₄ loss rate were increased. When the operational pressure of first-stage membrane pressure was 3 MPa, the CO₂ recovery fraction was over 90% and CH₄ loss rate was less than 5%. Obviously, at a high pressure did increase the flux of CO₂ gas through the membrane. According to the curve in Fig. 10, it can be seen that excessive pressure would lead to the reduction of CO₂ recovery fraction.
A proper membrane operational pressure is not only achieving a good gas separation effect but also improving CO$_2$ recovery efficiency. Therefore, the operational pressure was setting from 2 to 4 MPa. According to Fig. 10, when the optimal pressure is 3 MPa, the power consumption was 115.4 kW.
Operational Pressure at Third-stage Membrane Pressure

The operating pressure of first-stage membrane was fixed at 3 MPa and those of third-stage membrane were varied and set at 1.0, 1.5, 2.0, and 3.0 MPa, respectively. Then, the optimal pressure of third-stage membrane can be obtained.

As shown in Fig. 11, both CO2 recovery fraction and CH4 loss rate increased with an increase of operational pressure. When CO2 concentration in the input gas stream was 0.5 or 0.6, the operational pressure at third-stage membrane was 2.0 MPa, the CO2 recovery fraction was higher than 90% and CH4 loss rate was less than 5%. When the CO2 concentration in the input gas stream was 0.7 or 0.8, the operational pressure at the third-stage membrane was 2.5 MPa, both a high CO2 recovery fraction and a low CH4 loss rate can also be obtained. The driving force required for a low CO2 concentration is lower than that of a high one. Therefore, in the point of energy saving, a different operating pressure could be adopted for CO2 separation in different CO2 input concentrations. By analyzing the data, the operational pressure at third-stage membrane was set at 2.5 MPa.

Under the conditions that the first-, second- and third-stage membrane area were 2400, 3800 and 1800 m2, respectively and the operational pressure at first- and third-stage membrane were 3.0 and 2.5 MPa respectively; the simulation results were shown in Table. 2, which indicated that the CO2 recovery fraction be over 90%, CH4 loss rate be less than 5% and the power required be 203.4 kW (Fig. 12). Compared to that of three-stage membrane process by Song (2017), their results displayed that the CO2 recovery fraction was 84.6% and power required was 2.8 MJ kg–1. The three-stage membrane process of this study can save approximately 4% power consumption and can achieve a higher CO2 recovery fraction.

CONCLUSIONS

1. The membrane technology is more suitable for the CO2 separation in a higher concentration.
2. In this study, the MATLAB is used to simulate and obtain the optimal operational parameters for the three-stage membrane process. This work established a partial cycle and recovered the CO2 from the permeation side of second-stage membrane that enhance a higher purity CO2 gas stream.
3. The results of this study indicated that when the CO2 concentration was higher than 50% and at a flow rate of 100000 Nm3 d–1, the CO2 separation could be achieved at the optimal operation condition. Under the conditions that the membrane areas were 2400, 3800, and 1800 m2 for the first-, second-, and third-stage membrane, respectively.
Table 2. Summary of three-stage membrane process.

| Results parameters                        | Numerical value |
|-------------------------------------------|-----------------|
| First-stage membrane area (m²)            | 2400            |
| Second-stage membrane area (m²)           | 3800            |
| Third-stage membrane area (m²)            | 1800            |
| First-stage membrane pressure (MPa)       | 3.0             |
| Third-stage membrane pressure (MPa)       | 2.5             |
| Power consumption (kW)                    | 203.4           |

Fig. 12. Power consumption with the change of third-stage pressure.

first-, second-, and third-stage membrane, respectively and the operational pressure at first- and third stage membrane were 3.0 and 2.5 MPa, respectively, the CO₂ separation fraction was higher than 90% and CH₄ loss rate was lower than 5%.

4. The results of this study can be applied on the practical engineering application.

ACKNOWLEDGMENTS

This work was supported by Department of Science & Technology of Shandong Province (No. ZR2018LB025).

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Received for review, October 21, 2019
Revised, November 4, 2019
Accepted, November 8, 2019