

Research Article

Separation of CO₂ from Small Gas Molecules Using Deca-Dodecasil 3 Rhombohedral (DDR3) Membrane Synthesized via Ultrasonically Assisted Hydrothermal Growth Method

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Deca-dodecasil 3 rhombohedral (DDR3) membrane has received much attention in CO₂ separation from small gas molecules because of its molecular sieving property and stable characteristics. Therefore, the present work is focusing on the utilization of previously fabricated membrane (synthesized in 3 days as reported in our previous work) to study the effect of hydrocarbons and its durability at the previously optimized conditions. Subsequently, gas permeation study was conducted on the DDR3 membrane in CO₂ separation from small gas molecules and it was found that the permeance of H₂, CO₂, N₂, and CH₄ decreased in the order of H₂ > CO₂ > N₂ > CH₄, according to the increase in kinetic diameter of these gas molecules. Besides, it was observed that the ideal selectivities of the gas pairs decreased in the sequence of CO₂/CH₄ > CO₂/N₂ > H₂/CO₂. On the other hand, it was found that the presence of hydrocarbon impurities in the gas mixture containing CO₂ and CH₄ has directly affected the performance of DDR3 membrane and contributed to the losses of CO₂ permeability, CH₄ permeability, and CO₂/CH₄ selectivity of 39.1%, 14.8%, and 4.2%, respectively. Consequently, from the stability test, the performance of DDR3 membrane remained stable for 96 h, even after the separation testing using CO₂ and CH₄ gas mixture containing hydrocarbon impurities.

1. Introduction

Over the last few decades, much research has been done on the CO₂ separation especially in syngas production (mainly H₂) and natural gas purification. The presence of CO₂ in the syngas reduces the yield of hydrogen and methane recovery during the Fischer-Tropsch (FT) process [1, 2]. Similarly, the presence of CO₂ decreases the heating value of natural

gas and caused equipment corrosion in the existence of water [3, 4]. Meanwhile, membrane separation technology has received much attention in CO₂ separation mainly due to its advantages compared to the conventional separation technologies [5–11]. Zeolite membrane is favored in the CO₂ separation among the membrane materials over polymeric and mixed matrix membranes due to its characteristics, including well-specified pores, the molecular filtering property, and

TABLE 1: Comparison of different zeolite membranes used for CO₂ separation from CH₄.

Membranes	H ₂ /CO ₂ selectivity	CO ₂ /CH ₄ selectivity	CO ₂ /N ₂ selectivity	CO ₂ permeance (mol/m ² sPa)	Ref.
MFI membrane	70	—	—	0.80×10^{-7}	[14]
AlPO-18 membrane	—	220	45	6.30×10^{-7}	[15]
SAPO-34 membrane	0.44	25	8.30	1.60×10^{-7}	[13]
DD3R membrane	3.70	—	—	1.10×10^{-7}	[17]
DD3R membrane	—	—	200	3.0×10^{-7}	[18]
DD3R membrane	—	—	6.1	8.70×10^{-7}	[19]

strong CO₂ permeation [12, 13]. Table 1 shows the comparison of different zeolite membranes reported in the literature for CO₂ separation from CH₄.

Referring to Table 1, Li et al. [13] reported CO₂ separation from light gases through the SAPO-34 membrane and CO₂ permeance of 1.60×10^{-7} mol/m²sPa was achieved. Meanwhile, H₂/CO₂, CO₂/CH₄, and CO₂/N₂ selectivities of 0.44, 25, and 8.30 were obtained, respectively. Subsequently, Wang and his coworkers [14] reported on the CO₂ removal from small gas molecules at a high temperature of 500°C using the MFI membrane. They found that H₂ and CO₂ permeances as well as H₂/CO₂ selectivity of 1.20×10^{-7} mol/m²sPa, 0.80×10^{-7} mol/m²sPa, and 70, respectively, were achieved. Along with the MFI membrane, they also studied the permeation performance of AlPO-18 membrane for CO₂ separation from small gas molecules at room temperature. H₂, CO₂, and CH₄ permeances of 6.50×10^{-7} mol/m²sPa, 6.30×10^{-7} mol/m²sPa, and 1.0×10^{-7} mol/m²sPa were reported, respectively. On the other hand, CO₂/N₂ and CO₂/CH₄ selectivities of 45 and 220 were attained [15, 16].

Recently, the DDR3 zeolite membrane has attracted various researchers due to its aperture size of 0.36 nm × 0.44 nm which resulted in a remarkable performance in the separation of CO₂ from CH₄ [17–21]. In 27 to 31 days, many scientists used a secondary hydrothermal growth process and evaluated membrane efficiency in CO₂ separation from CH₄. DDR3 membrane is synthesized by different researchers. Tomita et al. documented CO₂ permeance over a membrane of DDR3 of 3.0×10^{-7} mol/m²sPa and selectivity CO₂/CH₄ of 200. CO₂ permeance was then obtained at 7.80×10^{-9} mol/m²sPa and selectivity at CO₂/CH₄ at 6.1 obtained by Nakayama et al. [19]. Recently, Bose et al. [17] investigated the fabrication of DDR3 membrane in 7 days and they found that CO₂ permeance of 1.1×10^{-7} mol/m²sPa and H₂/CO₂ selectivity of 3.70 was achieved at ambient conditions.

Although several studies have reported the gas permeation of DDR3 membrane in CO₂ separation from CH₄, researchers rarely reported on the membrane performance in CO₂ separation from other small gas molecules including H₂ and N₂. Therefore, in the present work, we focus on the performance of the DDR3 membrane synthesized in our previous work [22] for CO₂ separation from small gas molecules including H₂, CH₄, and N₂. This research also explores the impact of the impurities of hydrocarbons on the efficiency of CO₂ distinguishing the DDR3 membrane from CH₄.

2. Materials and Methods

Chemicals and the procedure used for the synthesis of DDR3 membrane have been reported in detail in our previous work [10, 22]. Firstly, the mixture solution was prepared using 1-adamantaneamine, tetramethoxy silane, ethylenediamine, and water. Subsequently, the mixture solution was undergoing ultrasonic-irradiation pretreatment for 1 h prior to hydrothermal growth for 1 day. Then, the seeds were recovered, washed with deionized water, and attached on α-alumina support via a vacuum-assisted seeding method. In the typical synthesis of DDR3 membrane, the seeded support was placed in an autoclave filled with solution mixture prepared using a similar recipe and procedure as described in our previous work [10]. After that, the membrane was grown in 2 days under hydrothermal heating. Consequently, the resultant membrane was preceded for calcination and characterization. The crystallinity and morphology of the membrane were characterized using an X-ray diffractometer (Bruker D8 Advance) and field emission scanning electron microscopy (Zeiss Supra 55 VP).

2.1. Permeation Study

2.1.1. Pure Gas Permeability. The performance of the resultant DDR3 membrane was investigated using the permeation test rig as shown in Figure 1. The membrane was sealed in the permeation cell, and the flow rate of the targeted gases including, H₂, CO₂, N₂, and CH₄ was fixed at 200 ml/min while the temperature (MKS instrument) and feed pressure were maintained at room temperature and 2 bar, respectively. Temperature and feed pressure were controlled with an error of ±0.1°C and 0.1 bar using MKS instrument temperature and feed pressure controllers, respectively. The gas permeation was calculated using equation (1), while ideal selectivity was calculated using equation (2) as follows [22, 23].

$$P_i = \frac{J_i}{\Delta P}, \quad (1)$$

$$\alpha_{i,j} = \frac{P_i}{P_j}, \quad (2)$$

where J_i is the flux (mol/m²s) and ΔP is the pressure drop (Pa) while $\alpha_{i,j}$ is the gas pair selectivity.

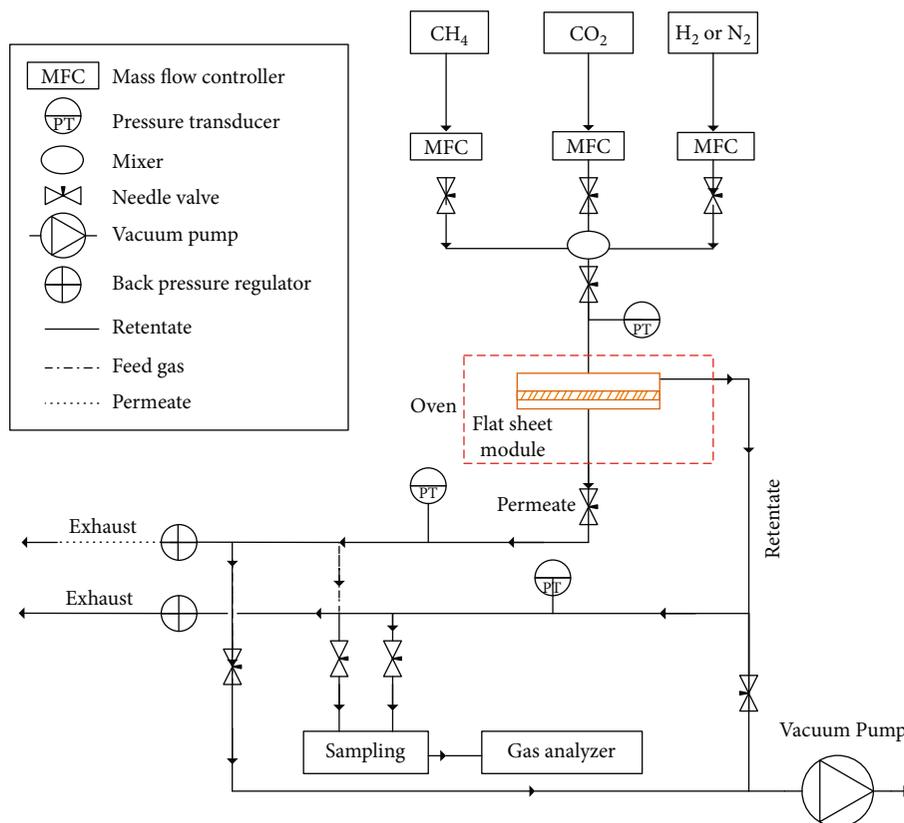


FIGURE 1: Schematic of permeation test rig used in the present work.

2.1.2. Effect of Hydrocarbon Impurities and Stability Test on DDR3 Membrane. The effect of hydrocarbon impurities and stability test over the resultant membrane in CO₂ separation from CH₄ was also conducted using the same permeation test rig. The experiments were conducted in three consecutive steps in which the first and third steps were used to investigate the stability of DDR3 membrane. Meanwhile, the second step of the experiment was carried out to study the effect of hydrocarbon impurities on the performance of the resultant membrane in CO₂ separation from CH₄.

During the first step of the experiment, temperature, inlet pressure, and CO₂ inlet composition were maintained at 30.1°C, 1 bar, and 10 vol% (90 vol% of CH₄), respectively, because this experimental condition was obtained as the optimum CO₂/CH₄ separation condition as reported in our previous work [10]. After 24 h, the second step of the testing was conducted by feeding the CO₂ and CH₄ gas mixture containing hydrocarbon impurities with the compositions of 35.84% CO₂, 44.50% CH₄, 6.27% C₃H₈, 3.44% C₄H₁₀, and 9.94% C₂H₆ into the system. This composition resembled the natural gas composition presence in North Sumatra Basin, Indonesia. After 48 h of testing, the feed gas was cut off and the experiment was continued using the feed gas composition applied in the first step of the testing. After each step, prior to the measurement of gas permeances, the system was allowed to stabilize for 30 min.

Gas compositions were calculated using gas chromatographs in feed, permeate, and retention streams (PerkinElmer Clarus 580). The CO₂ and CH₄ permeations were

determined with equation (1) with the amount of pressure decrease obtained by equation (3) [24]. The selectivity of the composition of CO₂/CH₄ was determined subsequently from the mole fractions as defined in equation (4).

$$\Delta P_i = (P_f - P_p), \quad (3)$$

where P_f and P_p are partial pressures in feed and permeate, respectively.

$$\text{Composition selectivity} = \frac{(X_i/X_j)_P}{(X_i/X_j)_F}, \quad (4)$$

where X_i and X_j are mole fractions of the i and j elements of the binary mixture. P and F subscriptions reflect both permeate and feed.

3. Results and Discussion

3.1. Characterization. The details on XRD, FESEM, FTIR, and EDX analysis on DDR3 membrane have been described in our previous work [10]. The significant XRD peaks and FESEM images obtained from the resultant membrane showed comparable crystallinity and hexagonal morphology which are analogous with those findings documented in the researches for DDR3 membrane [9, 10, 25, 26]. The surface and cross-sectional view of the membrane showed that a uniform, flat, and fully intergrown membrane layer was formed.

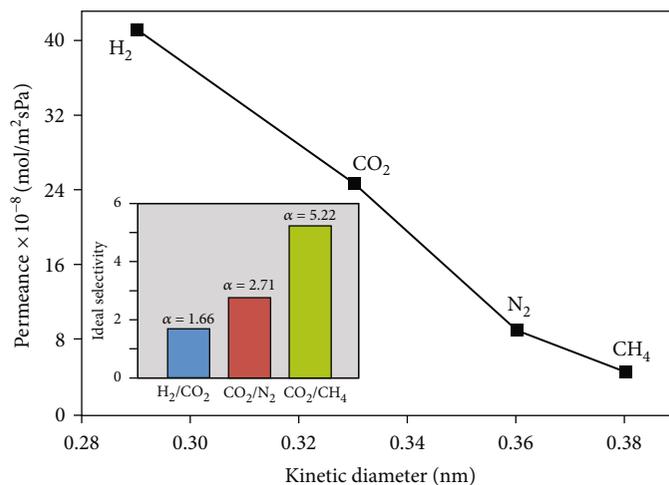


FIGURE 2: Single gas permeation results of small molecular gas over DDR3 membrane at room temperature and feed pressure of 2 bar (inset: ideal selectivities for H₂/CO₂, CO₂/N₂, and CO₂/CH₄).

3.2. Gas Permeation. Figure 2 shows the performance of the membrane at ambient temperature and feed pressure of 2 bar. Referring to Figure 2, H₂, CO₂, N₂, and CH₄ permeances of 4.10×10^{-7} mol/m²sPa, 2.46×10^{-7} mol/m²sPa, 9.11×10^{-8} mol/m²sPa, and 4.73×10^{-8} mol/m²sPa are obtained, respectively. It can be observed from Figure 2 that H₂ permeance is higher than that of those values obtained for CO₂, N₂, and CH₄ gases. This could be due to the significant difference in the Knudsen diffusion value of the gases through the membrane. H₂ with a molecular diameter of 0.29 nm is smaller than of the molecular diameter of CO₂, N₂, and CH₄, as well as the DDR3 pore channel. Meanwhile, the molecular diameter of CO₂, N₂, and CH₄ of 0.33 nm, 0.36 nm, and 0.38 nm, respectively, is closer to the DDR3 pore channel. Thus, H₂ can diffuse faster compared to CO₂, N₂, and CH₄ gases.

From Figure 2, it is found that the gas permeances are decreased in the order of H₂ > CO₂ > N₂ > CH₄. This decreasing trend is mainly attributed to the molecular sieving behaviour of DDR3 membrane and Knudsen diffusion mechanism of the gases [25–28]. The diffusion rate of these gases through the DDR3 membrane decreases when the kinetic diameter of the gases increases, and thus, decreasing order of gas permeance has been observed [17]. Moreover, H₂, CO₂, N₂, and CH₄ permeances obtained from the membrane were comparable with those results reported in the literature [17, 20, 25].

As shown in Figure 2 also, the ideal selectivities of CO₂/CH₄, CO₂/N₂, and H₂/CO₂ of 5.22, 2.27, and 1.66, respectively, are obtained. It is interesting to note that the ideal selectivities of the gas pairs decreased in the sequence of CO₂/CH₄ > CO₂/N₂ > H₂/CO₂. This result is attributed to the CH₄ permeance, which is much smaller than the permeance of H₂ and N₂ as shown in Figure 3. Overall, although the gas permeance and ideal selectivity results obtained in the present work are comparable with those results reported in the literature [25], the relatively low performance of DDR3 membrane could be due to the presence of minor defects or intercrystalline pores which have reduced the performance of the membrane. Thus, it is suggested that the membrane

should be modified with a TEOS solution to minimized intercrystalline defects to enhance its performance in gas separation [24]. TEOS could penetrate into intercrystalline defects and seal it through the formation of hydrogen bonding between membrane zeolite materials and TEOS. In addition, it also slows down the CH₄ permeation by providing extra resistance due to its higher kinetic diameter [25].

3.3. Effect of Hydrocarbon Impurities on the Performance of DDR3 Membrane. Figure 4 shows the stability test and effect of hydrocarbon impurities on the performance of the membrane in CO₂ separation from CH₄ for the testing duration of 96 h. Sections I and III shown in Figure 4 display the stability results of DDR3 membrane in CO₂ separation from a binary gas mixture of CO₂ and CH₄ at a temperature of 30.1°C, inlet pressure of 1 bar, and CO₂ inlet composition of 10 vol% because this experimental condition was obtained as the optimum CO₂/CH₄ separation condition which provided higher permeation performance of DDR3 membrane as reported in our previous work [10].

Meanwhile, section II in Figure 4 exhibits the effect of hydrocarbon impurities on the performance of DDR3 membrane in CO₂ separation from CH₄ at a temperature of 30.1°C and feed pressure of 1 bar. Referring to Figure 4, at points C and E, CO₂ and CH₄ permeances of 3.13×10^{-8} mol/m²sPa and 2.81×10^{-8} mol/m²sPa are obtained, respectively. Meanwhile, CO₂/CH₄ selectivity of 2.59 was obtained at point G. Results at points C*, E*, and G* were obtained when the feed gas was changed from a pure binary mixture of CO₂/CH₄ to CO₂/CH₄ gas mixture containing hydrocarbon impurities. At points C* and E*, CO₂ and CH₄ permeances of 3.11×10^{-8} mol/m²sPa and 2.83×10^{-8} mol/m²sPa are obtained, respectively. On the other hand, CO₂/CH₄ selectivity of 2.59 was obtained at point G*.

It can be seen from Figure 4 that the performance of the membrane slightly decreases when the gas mixture containing hydrocarbon impurities is fed into the system. Besides, from Figure 4, it is found that CO₂ and CH₄ permeances decrease from point E* to F and C* to D, respectively, after

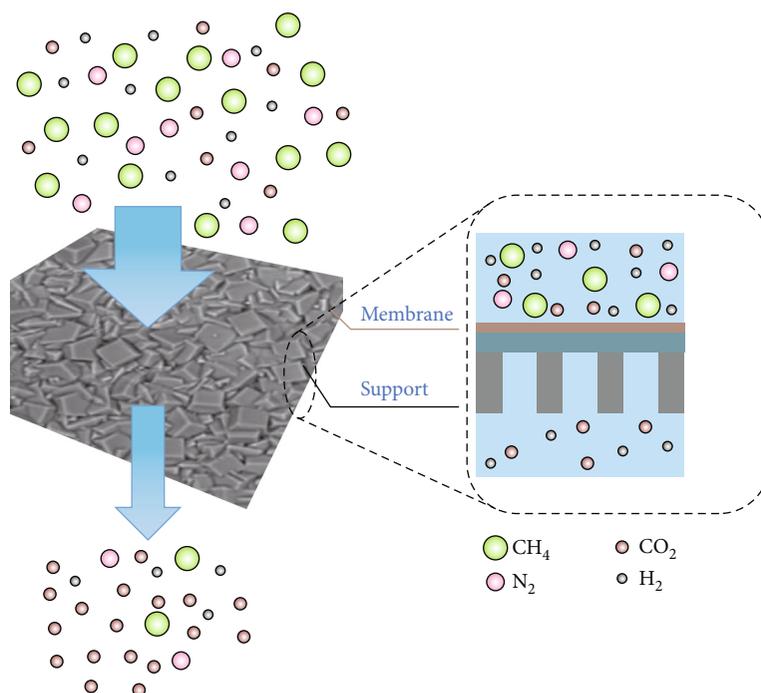


FIGURE 3: Mechanism of small molecular gas transport over DDR3 membrane.

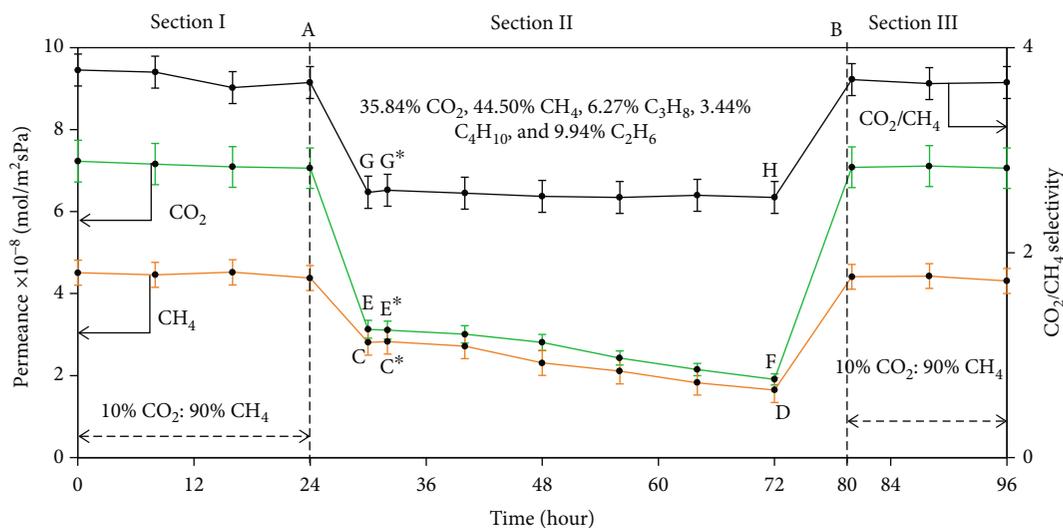


FIGURE 4: Effect of hydrocarbon impurities on the performance of DDR3 membrane and its durability test (separation results points C, E, and G were determined at a temperature of 30.1°C, feed pressure of 1 bar, with the CO₂ composition of 35.84% (CH₄ composition of 64.16%).

testing duration of 40 h (from 32 h to 72 h). Meanwhile, CO₂ and CH₄ permeances of 1.91×10^{-8} mol/m²sPa and 1.65×10^{-8} mol/m²sPa were obtained at point F and point D, respectively. This result shows that CO₂ permeance is gradually dropped by about 39.1% with respect to the value of CO₂ permeance obtained at point E*. Similarly, CH₄ permeance decreased by about 14.8% with respect to the initial value of CH₄ permeance obtained at point C*. The decrease in CO₂ and CH₄ permeances could be due to the adsorption of hydrocarbon impurities in DDR3 pores which inhibited the diffusion of CO₂ and CH₄ molecules [3]. Adsorption of

hydrocarbon impurities into the pores of DDR3 pore reduced its capacity for the diffusion of CO₂ and CH₄ molecules. Subsequently, adsorption of hydrocarbon impurities may also cause saturation for adsorption of CO₂ and CH₄ molecules which may also resulted to drop the performance of the membrane. In addition, a competitive sorption effect has been reported previously that mixed gas performance is always lower than pure gas due to a competitive sorption mechanism [4, 16]. However, CO₂/CH₄ selectivity of 2.54 was found at point H, which is slightly lower than the CO₂/CH₄ selectivity obtained at point G*. It is found that

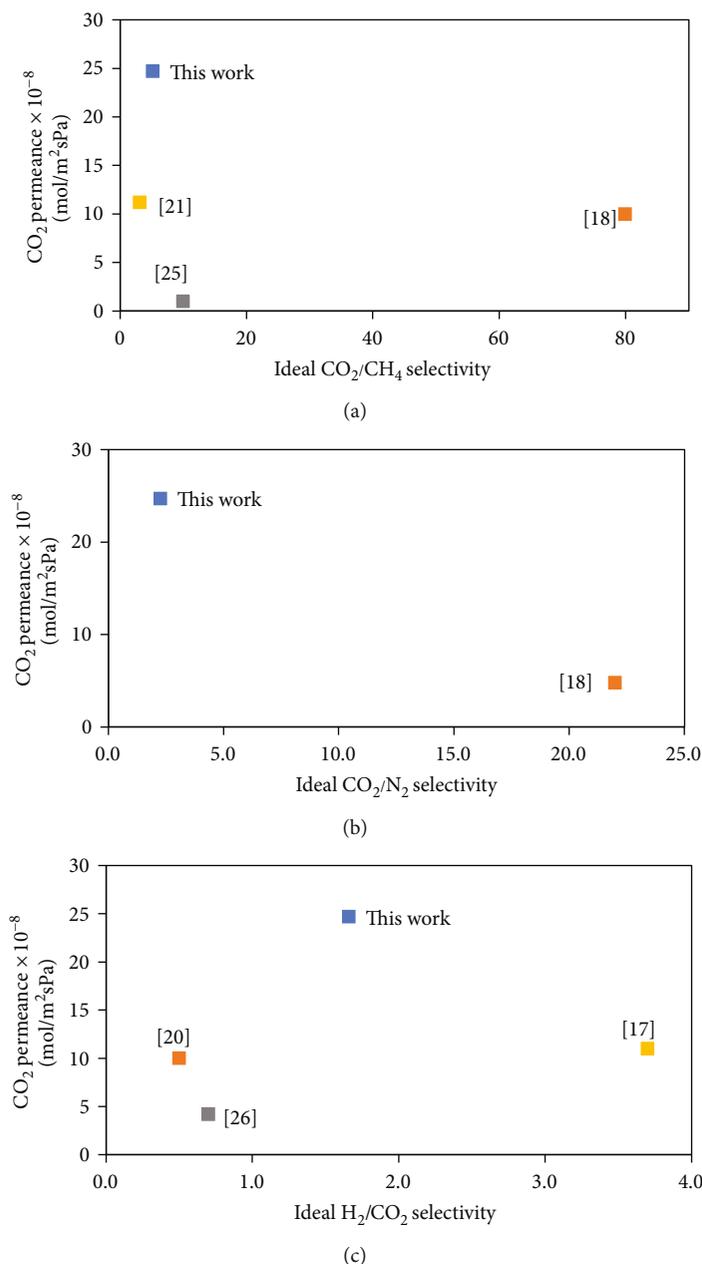


FIGURE 5: Comparison of gas pair ideal selectivities with reported literature ((a) CO₂/CH₄, (b) CO₂/N₂, and (c) H₂/CO₂).

the loss of CO₂/CH₄ selectivity at point H was only about 4.2% compared to the CO₂/CH₄ selectivity which was initially obtained at point G*.

Furthermore, it can be seen from Figure 4 (section I) that, when the hydrocarbon impurities are not presented in the feed gas, the performance of DDR3 membrane remains stable. After 24 h, nearly constant CO₂ and CH₄ permeances of 7.23×10^{-8} mol/m²sPa and 4.51×10^{-8} mol/m²sPa were obtained, respectively, while CO₂/CH₄ selectivity of 3.66 was achieved (line A). These results are similar to those values obtained for CO₂ permeance, CH₄ permeance, and CO₂/CH₄ selectivity when the feed gas was changed to the binary gas mixture of CO₂ and CH₄ at the testing duration from 80 h to 96 h. In section III, CO₂ and CH₄ permeances of 7.08×10^{-8}

mol/m²sPa and 4.41×10^{-8} mol/m²sPa were obtained (line B), while CO₂/CH₄ selectivity of 3.69 was achieved.

Therefore, it can be concluded that the presence of hydrocarbon impurities in CO₂ and CH₄ gas mixture has affected the performance of DDR3 membrane and caused a slight reduction of CO₂ and CH₄ gas over a testing duration of 40 h (from 32 h to 72 h). Consequently, CO₂/CH₄ selectivity of DDR3 membrane remained stable at testing condition of temperature of 30.1°C and feed pressure of 1 bar and CO₂ feed composition of 10 vol%.

3.4. Stability of DDR3 Membrane. Figure 4 also shows the stability of DDR3 membrane in CO₂ separation from CH₄. It can be seen from Figure 4 that CO₂ permeability, CH₄

permeability, and CO_2/CH_4 selectivity ($7.06 \times 10^{-8} \text{ mol/m}^2\text{sPa}$, $4.38 \times 10^{-8} \text{ mol/m}^2\text{sPa}$, and 3.66, respectively) obtained over DDR3 membrane after 24 h are comparable with those values achieved after 96 h (CO_2 permeance = $7.08 \times 10^{-8} \text{ mol/m}^2\text{sPa}$, CH_4 permeance = $4.41 \times 10^{-8} \text{ mol/m}^2\text{sPa}$, and CO_2/CH_4 selectivity = 3.69). Overall, after 96 h of testing duration, CO_2 permeability, CH_4 permeability, and CO_2/CH_4 selectivity were dropped by about 0.28%, 0.68%, and 0.81% compared to the original values obtained at line A shown in Figure 4. From these results, it has been found that membrane performance was dropped in small quantity and thus, it was decided that the membrane was stable in the presence of hydrocarbons.

In conclusion, the permeation performance of DDR3 membrane in the separation of CO_2/CH_4 remained steady up to 96 h with small losses of CO_2 permeability, CH_4 permeability, and CO_2/CH_4 selectivity. Thus, the separation performance of DDR3 membrane is durable and stable in the presence of hydrocarbon impurities.

The findings seen in the literature under the same temperature and pressure condition assess the efficiency of DDR3 membrane in CO_2/CH_4 , H_2/CO_2 , and CO_2/N_2 separations. With respect to Figure 5(a), the ideal CO_2/CH_4 selectivity obtained in this analysis is comparable to the published findings by Yajima and Nakayama [21]. However, this value is below the published results by Himeno et al. [18] and van den Bergh et al. [25]. Relatively low CO_2/CH_4 ideal selectivity of the DDR3 membrane obtained during the present research may be triggered by the existence of slight membrane defects which may influence CO_2 or CH_4 transport pathways via the membrane.

On the other hand, the performance of DDR3 membrane in CO_2/N_2 and H_2/CO_2 separation is shown in Figures 5(b) and 5(c). From Figure 5(b), it is found that the CO_2/N_2 selectivity obtained in the present work is lower than those results reported by Nakayama et al. [19]. Referring to Figure 5(c), the ideal selectivity of H_2/CO_2 obtained in the present study is slightly higher than those results reported by Tomita et al. [20] and Zheng et al. [26]. However, these values were lower than those results reported by Bose et al. [17]. Low ideal selectivity values of CO_2/N_2 and H_2/CO_2 obtained in the present work could be because of the similar reason as described previously for the performance of the membrane in CO_2/CH_4 separation. The presence of defects on the membrane affected the transport mechanisms of N_2 , H_2 , and CO_2 through the membrane and lowered the selectivity values of CO_2/N_2 and H_2/CO_2 .

4. Conclusions

In the present work, the performance of DDR3 membrane for the CO_2 separation from gas molecules exhibiting comparatively smaller kinetic diameters, i.e., N_2 and CH_4 , has been successfully conducted. The permeances of the gases obtained over the membrane decreased in the order of $\text{H}_2 > \text{CO}_2 > \text{N}_2 > \text{CH}_4$. Subsequently, it was also found that the DDR3 membrane demonstrated an ideal selectivity of 5.22 for CO_2/CH_4 which was higher than the ideal selectivity of H_2/CO_2 and CO_2/N_2 gas pairs. Besides, ideal selectivities

obtained from DDR3 membrane decreased in the sequence of $\text{CO}_2/\text{CH}_4 > \text{CO}_2/\text{N}_2 > \text{H}_2/\text{CO}_2$. Furthermore, it was observed that the presence of hydrocarbon impurities in the CO_2 and CH_4 gas mixture directly affects the performance of DDR3 membrane and contributed to the losses of CO_2 permeability, CH_4 permeability, and CO_2/CH_4 selectivity of 39.1%, 14.8%, and 4.2%, respectively. Consequently, from the stability test, it can be suggested that the permeation performance of DDR3 membrane remained stable for 96 h, even after the separation testing of a gas mixture containing hydrocarbon impurities, with losses of CO_2 permeability, CH_4 permeability, and CO_2/CH_4 selectivity of 2.35%, 4.43%, and 0.55%, respectively. From the comparison study, it was concluded that the permeation performance of DDR3 membrane in CO_2 separation from H_2 , CH_4 , and N_2 still needs to be improved mainly due to the presence of defects in the membrane.

Data Availability

The datasets generated and/or analyzed during the current study are available from the corresponding author on reasonable request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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