

Study of CO₂ and CH₄ Permeation Properties through Prepared and Characterized Blended Pebax-2533/PEG-200 Membranes

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Abstract This study demonstrates how incorporation of polyethylene glycol (PEG-200) into poly (ether-*block*-amide) (Pebax-2533) can improve the prepared membrane performance in separating CO₂ from CH₄. Additionally, the effect of various PEG-200 loadings on CH₄ and CO₂ permeability and CO₂/CH₄ selectivity values was investigated. The prepared membranes were examined using Fourier transform infrared (FTIR), X-ray diffraction (XRD) and scanning electron microscopy (SEM) analyses. Permeation experiments of the gasses through the neat Pebax and the blended (Pebax-2533/PEG-200) membranes were carried out at a temperature of 25 °C and pressure range of 2 to 10 bar. The gas permeation experiments indicated that the performance of blended membranes is better than that of the neat membrane. As an example, CO₂ permeability and ideal CO₂/CH₄ selectivity values for the blended membrane with 40 wt.% of PEG-200 loading are 351.65 and 9.17 Barrer, while those values for the neat membrane are 187.54 and 7.28 Barrer, respectively.

Keywords CO₂/CH₄ separation · Pebax-2533 · PEG-200 · Blended membranes

1 Introduction

Removal of carbon dioxide and hydrogen sulfide from natural gas streams has been essential in gas processing over the past few decades. Several technologies such as membrane processes, adsorption, chemical and physical absorption are employed for this purpose. However, gas separation based on membrane technologies has recently attracted much attention in chemical industries due to its economical and environmental benefits such as low operating and fixed costs, safety, compatibility with the environment as well as lower energy consumption [1–4]. One of the suitable polymers that can be considered as a good candidate for CO₂ separation from natural, flue and synthesis gasses is polyether-*block*-amide (Pebax) [5, 6]. Pebax involves linear chains of soft polyether (PE) and hard polyamide (PA) with rubbery and glassy structures, respectively. In fact, the PA block plays a role as an impermeable phase due to its low chain mobility and the PE block acts as a permeable phase. Various grades of Pebax differ in the type and content of PA and PE segments. In the present work, Pebax with a high content of PE (Pebax-2533) was utilized as the base polymer for membrane fabrication. It contains 80 wt.% of poly(tetramethylene oxide) and 20 wt.% of polyamide 12 [7, 8]. There are various modification methods to promote gas transport performances of polymers. One of the effective techniques for this purpose is incorporation of a proper polymer as a filler into the basic polymer and accordingly preparation of the blended membrane. In this research, physical blending of a suitable filler (PEG-200) with the Pebax-2533 matrix has been carried out to prepare the blended membranes and consequently improve their gas transport performance [9]. Several researches have investi-

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gated the preparation of different Pebax/PEG membranes for gas separation, as summarized below:

Car et al. [1] prepared Pebax-1657/PEG-200 blended membranes for separation of CO₂ from gas mixtures containing N₂, H₂, and CH₄. Their results showed that enhancing PEG-200 content up to 50 wt.% in the Pebax-1657 matrix increases permeability values of all the gasses and accordingly CO₂/H₂, CO₂/N₂ and CO₂/CH₄ selectivity values.

Yave et al. [10] synthesized blended membranes via blending PEG-200 with Pebax-1657 matrix to investigate the gasses (CO₂, C₄H₁₀, H₂ and CH₄) permeability and selectivity values. The gas permeation results showed that permeability values of all single gasses (particularly CO₂) and accordingly CO₂/CH₄ and CO₂/H₂ selectivity values improve with increasing the PEG content.

Feng et al. [11] fabricated Pebax-1074/PEG-1500 blended membranes to study permeation values of various gasses (CO₂, N₂, H₂ and CH₄). They measured the gas permeation properties of the prepared membranes at 35 °C and 60 °C. Obtained results revealed that at 35 °C, the CO₂ permeability along with CO₂/N₂, CO₂/H₂ and CO₂/CH₄ selectivity values of the blended membranes (containing 10, 20 and 30 wt.% of PEG-1500 content) are lower than those of the neat membrane. In contrast, at 60 °C, CO₂ permeability and consequently the CO₂/light gasses selectivity values increase with enhancing the PEG loading.

Ghadimi et al. [12] investigated gas transport properties of Pebax-1657/polyethylene glycol diacrylate (PEGDA) blended membranes. They found that with addition of PEGDA into the Pebax-1657 matrix, CO₂ permeability through the blended membranes increases, whereas CH₄ and N₂ permeability values decrease. Additionally, H₂ permeability exhibited a nearly constant trend.

Rabiee et al. [13] applied poly(tetramethylene ether) glycol (PTMEG) as a filler to prepare Pebax-1657/PTMEG blended membranes for CO₂ separation from H₂, N₂ and CH₄ gasses. Permeation results exhibited that the permeability values of all gasses are enhanced with increasing PTMEG loading. Besides, CO₂/H₂ selectivity increases, while CO₂/N₂, CO₂/CH₄ selectivity values decrease slightly.

The literature review reveals that PEG with a low molecular weight as an appropriate plasticizer can lead the gas diffusivity and solubility values to increase when it blends with polymers. Ethylene oxide (EO) units in PEG increase the solubility of CO₂ molecules and accordingly CO₂/CH₄ selectivity. On the other hand, PEG with a lower molecular weight has more interactions between end-groups of its chains and CO₂ molecules which in turn enhance CO₂ solubility and consequently CO₂/CH₄ selectivity in the blended membranes [11, 14, 15]. Therefore, PEG-200 was selected to prepare the blended membranes. The physical

characteristics of Pebax-2533 and PEG-200 are shown in Table 1 [16, 17].

Over the last decade, various researches have been conducted on Pebax based membranes for gas separation [18, 16, 19–22] but any application of the Pebax-2533/PEG-200 membrane for CO₂/CH₄ separation has not yet been reported. In this work, a solution blending technique was used to prepare the blended membranes containing various loadings of PEG (10, 20, 30 and 40 wt.%). Finally, the membranes were characterized using SEM, XRD, and FTIR and evaluated for CO₂ and CH₄ permeation.

2 Materials and Methods

2.1 Materials

Pebax-2533 as the base polymer, PEG-200 as the filler and dimethylformamide (DMF) (solvent) were supplied by Arkema, Sigma-Aldrich Inc. and Merck Company, respectively. Also, CH₄ and CO₂ gasses with 99.995% purity were obtained from Farafan Gas Tehran Company.

2.2 Membrane Preparation

A solution casting-solvent evaporation technique was applied in this study to prepare the neat and blended membranes. For preparation of the neat membrane, the moisture of the Pebax-2533 granules was first removed by placing them in a typical oven at 75 °C for 12 h and then 2.5 wt.% of the polymer was stirred magnetically in DMF at 100 °C for 3 h. The prepared solution was cast in a glass Petri-dish and then heated in an oven at 75 °C for 24 h. Thereafter, the prepared neat membrane was cautiously removed from the Petri-dish surface and again placed in a vacuum oven at 50 °C for 5 h to remove the residual solvent [23]. For

Table 1 Physical properties of Pebax-2533 and PEG-200

Property	Content	Unit
Pebax-2533		
Polyether content	80	wt.%
Density	1	g.cm ⁻³
Water absorption at 23 °C and 24 h in water	1.2	wt.%
Melting point	133.5	°C
Glass transition temperature	−65	°C
Stress at break	32	MPa
PEG-200		
Molecular weight	190 – 210	g.mol ⁻¹
Density at 20 °C	1.124	g.cm ⁻³
Viscosity at 20 °C	60	MPa.s

preparation of the blended membranes, certain amounts of PEG-200 (10, 20, 30 and 40% based on Pebax weight) were poured into DMF and stirred for 1 h at room temperature. 2.5 wt.% of Pebax granules were added to the prepared mixture and stirred using a magnetic stirrer for 3 h at 100 °C. The prepared solutions were poured into Petri-dishes and dried with the same method applied to the neat membranes.

2.3 Characterization

The X-ray diffraction (XRD) patterns of the prepared membranes were obtained using a JEOL, JDX-8030 instrument with a Cu K_{α} X-ray source and the wavelength of 1.54 Å to certify crystal structure and intermolecular distances between the intersegment chains. For this purpose, the diffraction angle (2θ) was changed between 5° and 75°. For obtaining the FTIR spectra of the prepared membranes, a Shimadzu 8400S spectrometer was employed and the spectra recorded from 4000 to 400 cm^{-1} with a resolution of 2 cm^{-1} . A Sigma scanning electron microscope (Zeiss, Germany) was applied to examine the prepared membranes' morphologies and also the PEG distribution in the Pebax matrix. The polymeric films were fractured in liquid nitrogen and then coated with Au before FESEM analysis.

2.4 Gas Permeation Measurement

As shown in Fig. 1, pure gasses (CH_4 and CO_2) permeation values of the prepared membranes were measured via a constant pressure (variable volume) setup at 25 °C and pressures of 2, 4, 6, 8 and 10 bar. It should be mentioned that the permeate side is at atmospheric pressure. Permeability in terms of Barrer (1 Barrer = $10^{-10} \text{ cm}^3 \text{ (STP).cm.cm}^{-2}.\text{s}^{-1}.\text{cmHg}^{-1}$) for the gasses was calculated

after the measurement of volumetric flow rate of each gas at steady state using Eq. (1):

$$P_i = 10^{10} \times \frac{273.15}{76} \times \frac{p}{T} \times \frac{Q_i \cdot l}{\Delta p \cdot A} \quad (1)$$

where, P_i , p , T , Q_i , l , Δp and A are permeability of the gas i (Barrer), permeate side pressure (cm Hg), temperature (K), the gas i volumetric flow rate ($\text{cm}^3.\text{s}^{-1}$), the membrane thickness (cm), the pressure difference between feed and permeate sides (cm Hg) and the membrane area (cm^2), respectively. Additionally, the ideal CO_2/CH_4 selectivity was calculated using the following equation:

$$\alpha_{\text{CO}_2/\text{CH}_4} = \frac{P_{\text{CO}_2}}{P_{\text{CH}_4}} \quad (2)$$

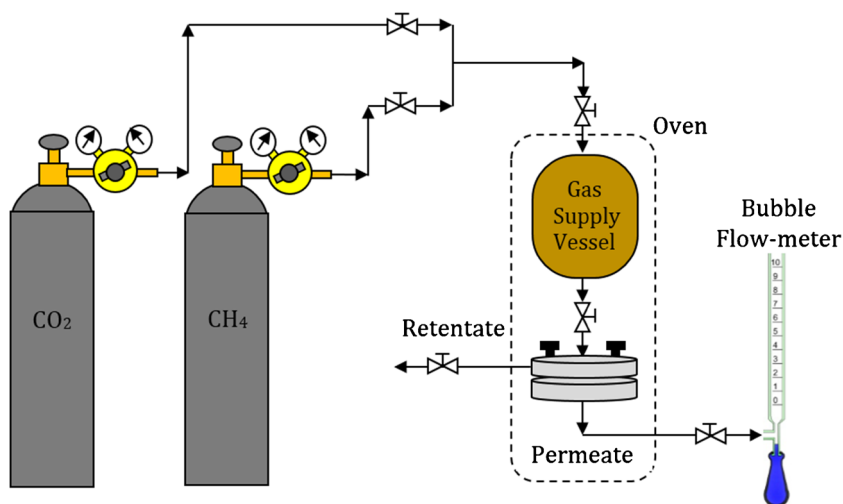
where P_{CO_2} and P_{CH_4} are permeability values of CH_4 and CO_2 , respectively. Note that three samples for each membrane were synthesized and accordingly all the gas permeation measurements were repeated three times for each membrane to minimize the possible errors of the experiments. Eventually, the average values were recorded based on the standard deviations.

3 Results and Discussions

3.1 Characterization

The crystalline structure of the prepared membranes which gives worthwhile information about permeability of the gasses through the membranes was investigated via XRD analysis [24, 25]. In Fig. 2, XRD patterns of the neat Pebax, blended Pebax/PEG (20 wt.% of PEG) and blended Pebax/PEG (40 wt.% of PEG) membranes are displayed. The pattern of the neat Pebax membrane demonstrates the crystalline zone of the Pebax matrix at 20.2°. As expected, the XRD patterns of the blended Pebax/PEG membranes

Fig. 1 Schematic of the gas permeation setup



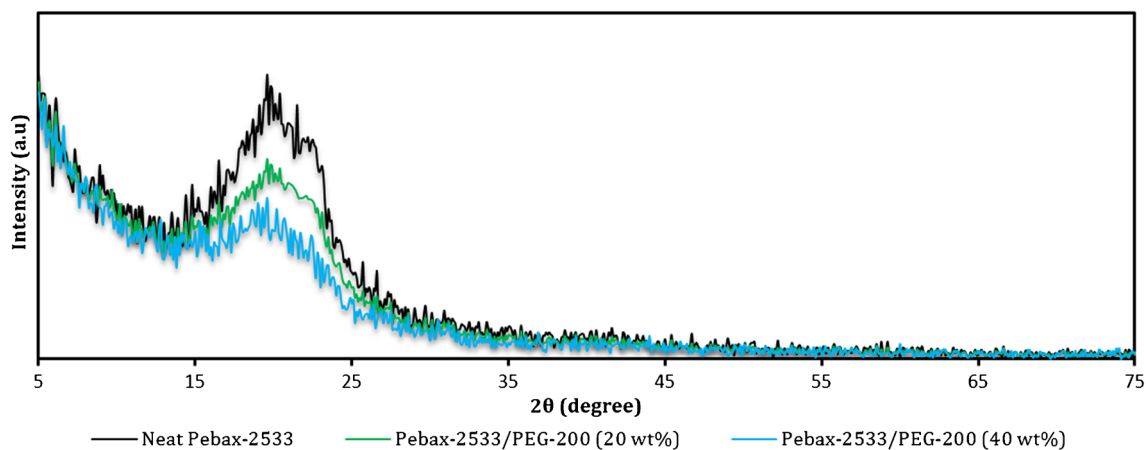


Fig. 2 XRD patterns of the neat Pebax-2533 and the blended Pebax/PEG membranes

show that the incorporation of PEG-200 into the Pebax-2533 matrix reduces the intensity of the peak at the crystalline region. Therefore, the crystallinity degree decreases with the addition of the PEG into the Pebax matrix. The hydrogen bonding disruption between PA segments of the Pebax and accordingly the cohesive energy reduction in the blended membrane are the main reasons for this behavior. It means that the hydrogen bonds are not created due to a positive interaction between PEG and Pebax. Since gas permeability is affected by the variation in the membrane crystallinity, it is expected that the gas permeability enhances through the blended membranes owing to the reduced crystallinity and also increased fractional free volume with enhancing PEG loading in the Pebax matrix. The measurement of gas permeability through the membranes absolutely proves this expectation [26]. The effects of PEG incorporation into the Pebax matrix were also investigated using a comparison between FTIR spectra of PEG-200, the neat Pebax-2533, the blended Pebax/PEG (20 wt.% of PEG) and the blended Pebax/PEG (40 wt.% of PEG) membranes

as shown in Fig. 3. The FTIR spectrum of the neat Pebax-2533 membrane shows two characteristic peaks at 1106 and 1640 cm^{-1} which correspond to the symmetric stretching vibration of ether (-C-O-C-) and amide (H-N-C=O) groups, respectively. Additionally, a peak at 3300 cm^{-1} is attributed to the N-H stretching in the PA part. Furthermore, two observed peaks around 2850–2950 cm^{-1} indicate the bending vibration of C-H. As shown in Fig 3, the absorption peaks at 2868 and 2932 cm^{-1} are assigned to stretching vibration of -CH₂ (asymmetric) and -CH₂ (symmetric) in alkyl chains, respectively and the characteristic peak at 1109 cm^{-1} corresponds to vibration of the -C-O-C- group. Comparison of the spectrum of the blended Pebax/PEG membranes and that of the neat membrane reveals that the -N-H- and the H-N-C=O peaks in the FTIR spectra of the blended membranes are moved to lower wavenumbers. These changes can be the proof of hydrogen bonding rupture and accordingly the crystallinity decrements in the blended membranes. Moreover, the absorption peak at 1106 cm^{-1} which is assigned to the ether group is intensified with the

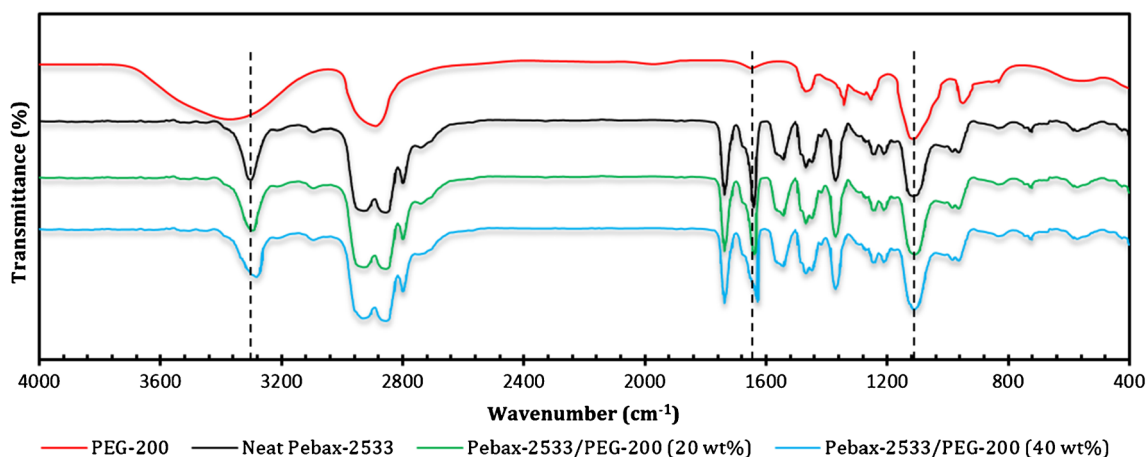


Fig. 3 FTIR spectra of PEG-200, the neat Pebax-2533 and the blended Pebax/PEG membranes

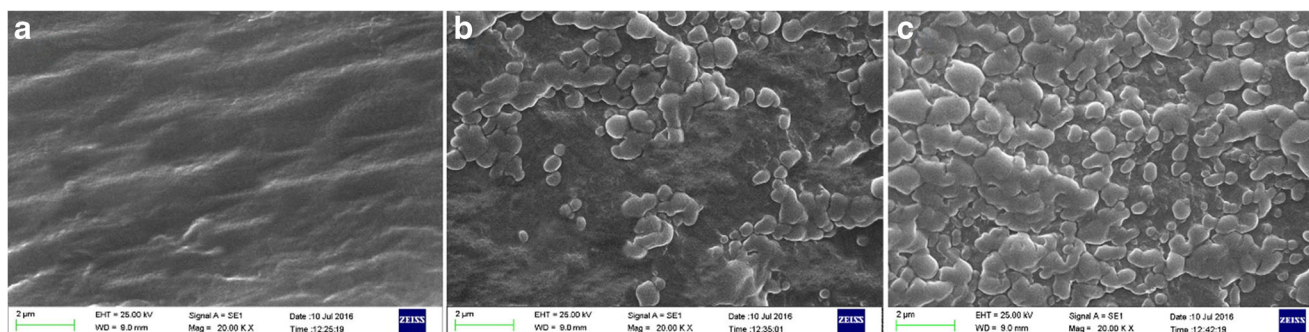


Fig. 4 Cross-sectional FESEM images of **a**) the neat Pebax-2533 membrane, **b**) the blended Pebax/PEG (20 wt.%) membrane, **c**) the blended Pebax/PEG (40 wt.%) membrane

incorporation of the PEG. This can be due to the presence of EO units in PEG-200. The results show that the FTIR spectra and the XRD patterns are in good agreement. As shown in Fig. 4, the morphology of the neat and the blended Pebax/PEG-200 membrane with PEG loadings of 20 and 40 wt.% was investigated via cross-sectional FESEM images. The FESEM images show that all the membranes have the dense structures and the neat membrane structure is more regular than the blended Pebax/PEG-200 membranes. The XRD results show that addition of the PEG-200 into the Pebax-2533 matrix results in a reduction in the membrane crystallinity via the disturbance of intermolecular hydrogen bonding which consequently leads to a non-uniformity in the structural body of Pebax and this irregularity is higher for the blended Pebax/PEG (40 wt.% of PEG) membrane [18]. Note that the fabricated membranes' thicknesses were measured from 40 to 60 μm .

3.2 Gas Permeation Properties

3.2.1 Effect of PEG Addition

Pure gasses permeation values through the neat and the blended membranes were measured at feed pressure of 2 bar and temperature of 25 °C to investigate the influence of various PEG-200 loadings in the Pebax-2533 matrix. As seen in Table 2, as the PEG content enhances in the Pebax matrix, CO₂ and CH₄ permeability values increase;

however, CH₄ permeability does not increase considerably. The principal reasons for the increment in the permeability values are: (1) Reduced crystallinity and accordingly increased free volume of the blended membranes which increase the tendency of gasses to diffuse more through the membranes and consequently the gasses permeability values. In other words, PEG-200 as a plasticizer inserts its molecules among the chain parts of Pebax-2533, and make extra intermolecular space for small Brownian movement and accordingly causes the polymer chains flexibility to increase [27]. Equation (2) Addition of the EO units in the blended membranes due to PEG incorporation into the Pebax matrix makes CO₂ permeability increase more than CH₄ through the blended membranes owing to the increased CO₂ solubility resulting from the more interaction of CO₂ with the EO units in the blended membranes. Therefore, solubility and permeability of CO₂ are enhanced more than those of CH₄ with enhancing PEG content and consequently the ideal CO₂/CH₄ selectivity of the blended membranes has an ascending trend as the PEG-200 loading increases (see Table 2). As a result, the efficiency of the blended Pebax-2533/PEG-200 membranes for CO₂/CH₄ separation is higher than that of the neat Pebax-2533 membrane and the Pebax-2533/PEG-200 membrane with 40 wt.% of PEG loading has the best performance among all the prepared membranes (It shows CO₂ permeability of 351.65 Barrer and ideal selectivity of 9.17 for CO₂/CH₄ separation, at 2 bar and 25 °C). Additionally, a comparison between

Table 2 Influence of PEG-200 contents on gas permeation performance of the prepared membranes at 2 bar and 25 °C

Prepared membranes	Permeability (Barrer)		Selectivity CO ₂ /CH ₄
	CO ₂	CH ₄	
Neat Pebax-2533	187.54 ± 5.65	25.77 ± 0.77	7.28 ± 0.44
Pebax-2533/PEG-200 (10 wt.%)	243.61 ± 7.31	32.21 ± 0.97	7.56 ± 0.45
Pebax-2533/PEG-200 (20 wt.%)	282.82 ± 8.42	36.15 ± 1.10	7.82 ± 0.47
Pebax-2533/PEG-200 (30 wt.%)	320.44 ± 9.63	37.09 ± 1.12	8.64 ± 0.52
Pebax-2533/PEG-200 (40 wt.%)	351.65 ± 10.62	38.36 ± 1.14	9.17 ± 0.55

Table 3 Comparison of CO₂/CH₄ separation performance of the best blended membrane prepared in the current study with reported results in the literature

Membrane type	Filler content (wt.%)	Pressure (bar)	Temperature (°C)	P _{CO₂} (Barrer)	α _{CO₂/CH₄}	Reference
Pebax-1657/PEG-400/ZnO	PEG-400:40 ZnO:4	3	25	94.5	24.2	[15]
Pebax-1657/ PEG-200	50	0.6	30	151	15.5	[18]
Pebax-1657/ PEG-200	50	0.6	30	152	15.2	[10]
Pebax-1657/ PEGDA	40	8	25	160	15.9	[12]
Pebax-1074/ PEG-1500	50	5	35	36.4	26.0	[11]
Pebax-1657/ PTMEG	30	4	25	230	14	[13]
Pebax-2533/ PEG-200	40	2	25	351.6	9.2	This study

CO₂/CH₄ separation efficiency of the best prepared Pebax-2533/PEG-200 membrane (loaded with 40 wt.% of PEG) in this study and those of other prepared Pebax based blended membranes presented in previous researches is listed in Table 3.

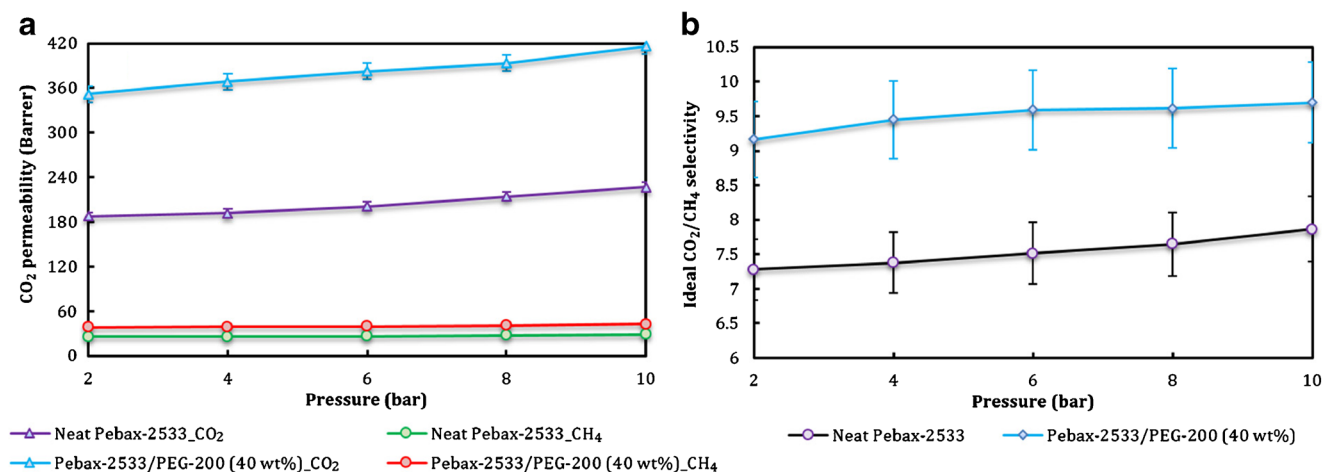
3.2.2 Effect of Feed Pressure

To investigate the effect of feed pressure on the performance of the prepared membranes, pure CH₄ and CO₂ permeability values through the neat Pebax and the blended Pebax/PEG (40 wt.% of PEG) membranes were measured at pressures of 2, 4, 6, 8 and 10 bar and temperature of 25 °C. As seen in Fig. 5a, the gas permeability values through the prepared membranes increase by increasing the pressure owing to enhancement of the driving force for mass transfer and solubility of the gasses. With increasing the pressure from 2 to 10 bar, the permeability of CH₄ through the neat Pebax membrane increases from 25.77 to 28.86 Barrer whereas, that of CO₂ increases from 187.54 to 226.92 Barrer. As seen in Fig. 5a, the enhancement of CO₂ permeability is more than CH₄ due to greater affinity of CO₂ with the Pebax polar ether groups and consequently its higher

solubility in the polymer matrix. Therefore, as shown in Fig. 5b, the ideal CO₂/CH₄ selectivity of the neat Pebax increases with enhancing the feed pressure. Figure 5a also shows that as the feed pressure increases from 2 to 10 bar, the permeability of CH₄ through the blended Pebax/PEG (40 wt.% of PEG) membrane increases from 38.36 to 42.93 Barrer whereas, that of CO₂ increases from 351.65 to 416.34 Barrer. As observed, the permeability increment for CO₂ is comparatively greater than that for CH₄ due to higher solubility of CO₂ in the blended membrane resulting from the added EO units into the Pebax matrix and accordingly rubbery section which determines the gas transport. Consequently, as displayed in Fig. 5b, the ideal CO₂/CH₄ selectivity of the blended membrane increases as the pressure increases [28, 29].

4 Conclusion

In this research, Pebax-2533 and PEG-200 were used as the base polymer and the filler of the prepared membranes for CO₂/CH₄ separation, respectively. The FTIR and XRD results revealed that with addition of the PEG into the

**Fig. 5** Influence of feed pressure on a) CO₂ and CH₄ permeability values, b) ideal CO₂/CH₄ selectivity values

Pebax matrix, the hydrogen bonding between the PA parts is disrupted. Additionally, regarding the FESEM images, increasing PEG-200 content causes some irregularity in the Pebax/PEG membranes' structures in comparison with the neat Pebax membrane. The obtained results of the gas permeation experiments showed that gas separation efficiency of the blended Pebax/PEG membranes is better than that of the neat Pebax membrane due to the increased CO₂ solubility and the reduced crystallinity of the blended membranes resulting from the increased EO unit in the Pebax matrix and rupture of the hydrogen bonding between the PA blocks, respectively. Consequently, the blended membrane containing 40 wt.% of PEG-200 loading exhibited the best performance (highest CO₂ permeability and CO₂/CH₄ selectivity) compared to the neat and other blended membranes.

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