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Enhanced CO_2 Separation Using Pebax Membrane Modified with Ethylene Glycol Monophenyl Ether

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ABSTRACT

CO_2 separation is one of the main challenges of today's world due to the growth of industries. Membrane separation method is a promising method for CO_2 separation, and poly(ether-block-amide) (Pebax) polymer membrane is one of the industrial membranes in this regard. However, this membrane is limited by the permeability-selectivity trade-off, which restricts its wider industrial application. This study aimed to address this limitation by incorporating ethylene glycol monophenyl ether (EGME) as filler in the Pebax membrane. The investigation focused on the impact of EGME on the chemical structure, morphology, physical and thermal properties, as well as the separation characteristics of the polymer. The results showed that by adding EGME, the prepared membrane became more brittle, with increased stiffness, and the tensile strength and Young's modulus of the Pebax/EGME membrane increased by 53 and 99.5 percent, respectively, compared to the pure membrane. Furthermore, the permeability of CO_2 and the CO_2/N_2 selectivity improved by 247% and 49%, respectively, attributed to the interactions between EGME and CO_2 molecules, including Lewis acid-base, dipole-quadrupole, and π-quadrupole interactions. This performance improvement allowed the Pebax/EGME membrane to surpass the Robeson upper bound, overcoming the permeability-selectivity trade-off, indicating the key role of EGME in improving the separation performance of Pebax. Moreover, The separation efficiency of the developed membranes was on par with, and in many cases superior to, the majority of membranes created by other researchers.

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1. Introduction

Nowadays, the pollution caused by greenhouse gases represents a significant challenge for nations worldwide. Among these gases, carbon dioxide (CO_2) is particularly detrimental, playing a crucial role in the greenhouse influence and contributing to worldwide warming (Sun et al., 2024). Flue gases are a major source of CO_2 emissions, necessitating the implementation of various removal techniques. Membrane separation technology has developed as a prominent and effective technique in this domain, gaining considerable attention in recent years. The majority of membranes utilized for CO_2 separation are polymer-based, offering numerous advantages such as flexibility and ease of production and modification (Khosravi, 2021). Membranes constructed from the poly(ether-block-amide) (Pebax) copolymer have proven to be particularly effective for CO_2 separation, attracting significant interest from researchers (Afshoun et al., 2017). Pebax is a copolymer composed of polyethylene and polyamide segments, which endows it with mechanical strength and favorable permeability for CO_2 separation due to its distinctive two-part structure. Nonetheless, membranes derived from this copolymer often face challenges related to the trade-off between enhancing selectivity and permeability simultaneously (Kojabad et al., 2024a). To address this issue, researchers have discovered the incorporation of organic or inorganic fillers, amine compounds, carbon-based materials, zeolites, metal oxides, and metal-organic frameworks into the polymer matrix (Elyasi and Norouzi, 2025; Kojabad et al., 2024b; Zhang et al., 2025). This approach improves the interaction between the polymer and gas molecules, so improving both gas separation performance and membrane stability. The collaboration between polymers and fillers leads to the development of membranes characterized by high efficiency, selectivity, and permeability, offering effective solutions for a variety of applications (Narkkun et al., 2023).

The selection of an appropriate compound to incorporate into the polymer matrix is crucial for the development of membranes that exhibit effective separation capabilities. Given that Pebax interacts with CO_2 via its ether groups, the introduction of a compound featuring functional groups with a strong affinity for CO_2 can significantly enhance the permeability (Hosseinkhani et al., 2024). While employing various nanoparticles within the polymer matrix is a viable approach, it presents the challenge of particle aggregation, which can adversely affect the membrane's separation efficiency. Furthermore, although high molecular weight compounds, such as polymers, integrate well with the polymer matrix, their positioning among the polymer chains tends to diminish the fractional free volume, thereby substantially lowering the gas diffusion coefficient. In contrast, low molecular weight compounds that possess functional groups favoring CO_2, when incorporated into the polymer matrix, achieve a favorable distribution and exhibit enhanced mobility, ultimately contributing positively to the increase in gas permeability (Setiawan and Chiang, 2023).

In recent years, the incorporation of low molecular weight compounds into polymer matrices has garnered significant interest for enhancing membrane separation performance. Meshkat et al. (2019) introduced benzoic acid and isophthalic acid, both of which contain carboxylic acid groups, into the Pebax matrix. Their findings indicated that benzoic acid was more effective in enhancing the performance of Pebax, achieving improvements in CO_2/N_2 selectivity and CO_2 permeability of 55% and 120%, respectively, compared to the pure Pebax membrane. Sanaeipour et al. (2019) incorporated glycerol, a compound with hydroxyl groups, into the Pebax matrix. While the addition of glycerol led to a 172% increase in CO_2/N_2 selectivity, it also led to a 23% reduction in CO_2 permeability relative to the pure Pebax membrane. Elyasi et al. (2021a) utilized aniline, which contains an amino group, in the Pebax matrix. The presence of aniline created a facilitated transport mechanism, resulting in increases of 101% in CO_2/N_2 selectivity and 76% in CO_2 permeability compared to the pure Pebax membrane. Hassanzadeh et al. (2022) added sorbitol, characterized by its six hydroxyl groups, to the Pebax matrix. This modification yielded a modest 0.5% increase in CO_2 permeability, while CO_2/N_2 selectivity improved by 29% compared to the pure Pebax membrane. Given these studies, researchers are actively seeking to identify an optimal compound that can further enhance the performance of Pebax. This compound should ideally feature appropriate functional groups to reinforce the interaction between the CO_2 molecules and polymer matrix, while also ensuring compatibility with the polymer matrix to prevent defects at the membrane structure, which could otherwise diminish selectivity. Choosing a compound that can interact with CO_2 faster and easier is essential. As mentioned, various compounds containing hydroxyl, carboxylic, amine, and ether groups have been used extensively as CO_2 carriers in recent years. The amino group necessitates the availability of water to engage in reactions with CO_2, thereby restricting its applicability. In contrast, ether, hydroxyl, and carboxyl groups can interact with CO_2 under various conditions, making them more versatile. Selecting a material that possesses at least two of these functional groups can prove advantageous. Ethylene glycol monophenyl ether (EGME) is a compound that contains both an ether group and a hydroxyl group.

In this research, EGME was identified to enhance the separation efficiency of Pebax. The study examined its influence on the chemical structure, morphology, physical and thermal characteristics, as well as the CO_2 separation capabilities of this polymer. EGME can interact with CO_2 by Lewis acid-base interactions via its hydroxyl group and can engage in dipole-quadrupole interactions with CO_2 through its ether group. Furthermore, the π electrons present in the aromatic ring of the EGME structure can facilitate π-quadrupole interactions with CO_2.

2. Experimental

2.1. Materials

CO_2 and N_2 gases with 99.9% purity were provided by Araz Oxygen Co. Pebax®1657 was purchased from Arkema. EGME and ethanol were obtained from Sigma-Aldrich.

2.2. Membrane Preparation

To prepare the membranes, first 0.58 g of Pebax was dissolved in 22.4 cc of a ethanol/water solution (70/30 wt%), which was refluxed at 85 °C for a duration of 2 hours, leading to a 3 wt% solution. Once the solution reached room temperature, 0.145 g of EGME (20 wt%) was added to the Pebax solution and stirred for 30 min. After that, the prepared solution was bath sonicated for 20 min and then was subsequently stirred for 2 hour. The resultant solution was then poured into a designated Teflon mold and placed in an oven set at 50°C for 24 hours. Following this period, during which membrane films were formed, the membranes were transferred to an oven at 110°C for an additional 6 hours to ensure complete drying.

2.3. Characterizations

The physical properties and thermal characteristics of the membranes were assessed through differential scanning calorimetry (DSC) using a Netzsch instrument (Netzsch DSC-200 F3, Germany). Fourier transform infrared spectroscopy (FTIR) was conducted utilizing an alpha-p spectrometer (Bruker, UK) to analyze the chemical composition of EGME and the prepared membranes. The morphology of the membranes was also investigated via field emission scanning electron microscopy (FESEM) with a TESCAN BRNO-Mira3 LMU apparatus, while the mechanical strength of the membranes was determined by a Zwick/Roell tensile testing machine (Z010, Germany).

3.4. Gas Permeation Tests

The gas separation performance of prepared membranes was examined under conditions of varying volume and constant pressure, which is shown schematically in (Figure 1). In this regard, The prepared membranes were cut into the round shape disc with the diameter of 6 cm. These experiments were carried out at a pressure of 7 bar and temperature of 25 °C. The gas permeability (P) was calculated using Eq. 1 (Amini & Asghari, 2018).

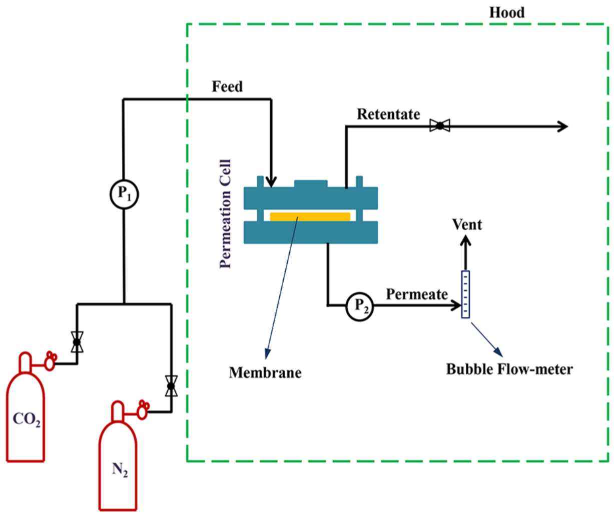

Figure 1. Schematic of the Experimental Setup for Gas Permeation Measurement

In this equation, Q denotes the volumetric flow rate of the gas flowing through, measured in cm^3/s. A represents the effective membrane area in cm^2, Δp signifies the pressure difference across the membrane in cmHg, and L indicates the thickness of the membrane in cm. The permeability derived from this equation is quantified in Barrer, where 1 Barrer is equivalent to 10^{-10} cm^3(STP) cm/(cm^2cmHg). Moreover, the selectivity ($\alpha_{i/j}$) for the gas pair i and j is determined by the ratio of their permeability, as outlined in Eq. 2 (Momeni et al., 2019).

$$\alpha_{i/j}=\frac{P_i}{P_j} \tag{1}$$

3. Experimental

3.1. Characterizations

(Figure 2) shows the findings from the FTIR analysis conducted on EGME and the prepared membranes. The FTIR spectrum of EGME reveals distinct peaks at 1230 cm^{-1} and 1088 cm^{-1}, which correspond to the C–O and C–O–C bonds, respectively. Furthermore, while C–H groups are identified at 2880 cm^{-1}, hydroxyl groups are observed in the region of 3500 cm^{-1}. The FTIR analysis of both the pure Pebax membrane and the Pebax/EGME membrane reveals distinct peaks associated with the N–H group of the polyamide component of Pebax at 3296 cm^{-1} (Kojabad et al., 2021b). Peaks at 2900 cm^{-1} and 1734 cm^{-1} correspond to the CH_2 group and the C = O ester bond, respectively, while the peak for the H-N-C = O bond is observed at 1650 cm^{-1}. Furthermore, the peak at 1090 cm^{-1} is attributed to the C–O–C bond present in the polyether segment of Pebax (Mahdavi et al., 2017). A comparison of the FTIR spectra between the pure membrane and the EGME contained membrane indicates that the addition of EGME does not result in any significant alterations to the FTIR spectrum of the Pebax polymer matrix. This suggests that the interactions between EGME and the polymer matrix are predominantly physical and hydrogen bonds.

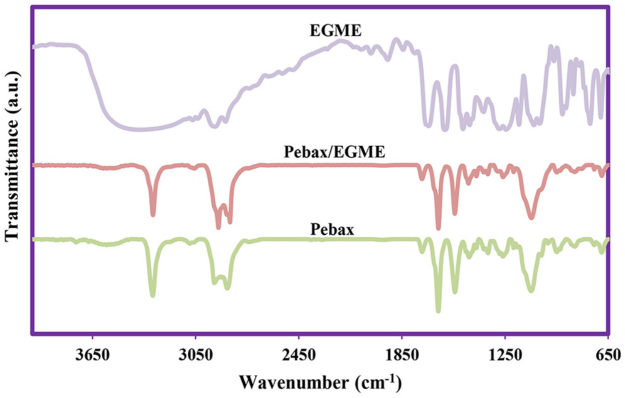

Figure 2. FTIR Spectra of EGME and the Prepared Membranes

To investigate the morphology of the prepared membranes, FESEM images was employed, with results presented at two distinct magnifications in (Figure 3). The figure indicates that the prepared membranes are devoid of any structural imperfections and exhibit a compact, non-porous configuration. It is evident that the incorporation of EGME into Pebax has resulted in a final membrane that is more brittle and textured. This change may be due to the formation of hydrogen bonds between the Pebax chains and the functional groups of EGME. In essence, as EGME molecules interpose between the polymer chains and establishing hydrogen bonds with various segments, the polymer matrix become brittle.

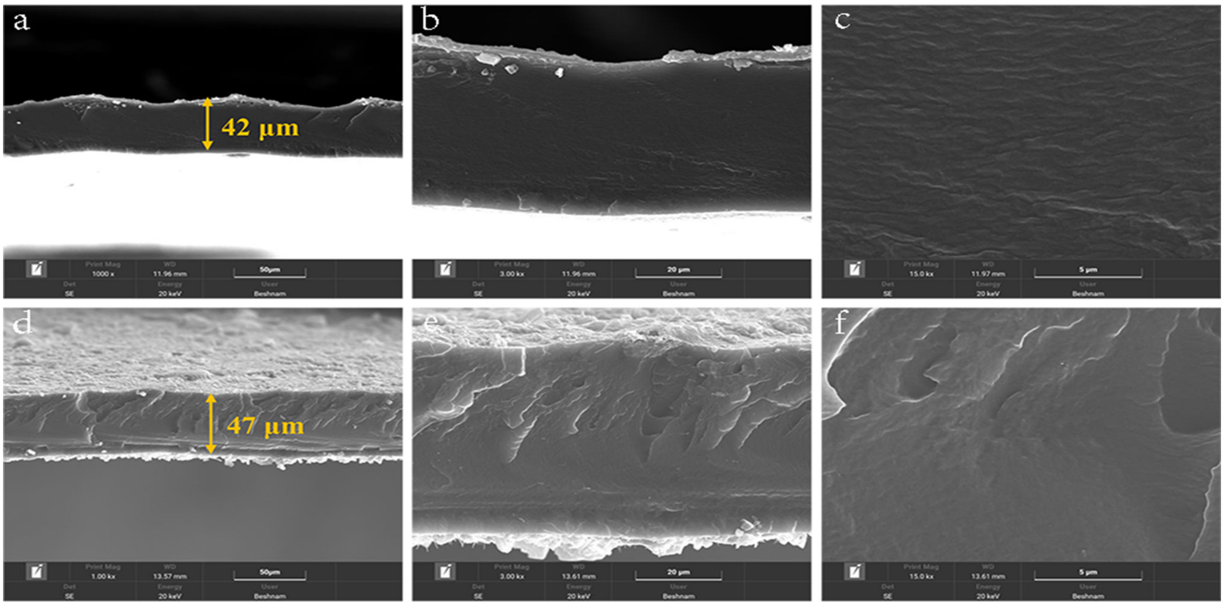

Figure 3. Cross-sectional FESEM Images of the Prepared Membranes with Two Different Magnifications: (a, b, and c) Pebax; (d, e, and f) Pebax/EGME

(Figure 4) and (Table 1) present the findings from the DSC analysis. The glass transition temperature (T_g) of the Pebax/EGME membrane is elevated compared to that of the Pebax membrane. This indicates that the Pebax/EGME mebrane become more glassy due to hydrogen bonds between EGME and polymer chins. The prepared membranes display two distinct melting points: one associated with the polyamide component ($T_{m,PA}$) and the other with the polyether component ($T_{m,PE}$) of the Pebax. A comparison of the melting point temperatures of Pebax/EGME, as opposed to the pure membrane, reveals that the addition of EGME has raised the $T_{m,PE}$ while lowering that of the $T_{m,PA}$. The presence of EGME molecules within the polymer chains facilitates the formation of hydrogen bonds with both the polyether and polyamide segments of the Pebax chain, leading to a partial loss of microphase separation between these two phases (Lotfi Mayan Sofla et al., 2019). This interaction results in the amide component melting at higher temperatures due to its integration within the polyether structure, while the polyether component freezes at lower temperatures as it is incorporated within the polyamide structure. As indicated in the table, the blending of the polyamide and polyether components, facilitated by EGME, has resulted in a reduction in the crystallinity of both components. The polyether segment of Pebax plays a crucial role in the transport of CO_2 molecules due to its inherent flexibility. Therefore, decreasing the crystallinity of polyether segment ($X_{c,PE}$) will enhance both its flexibility and CO_2 permeability. Furthermore, a reduction in the crystallinity of the polyamide segment ($X_{c,PA}$) may also create a synergistic effect, contributing to an increase in gas permeability.

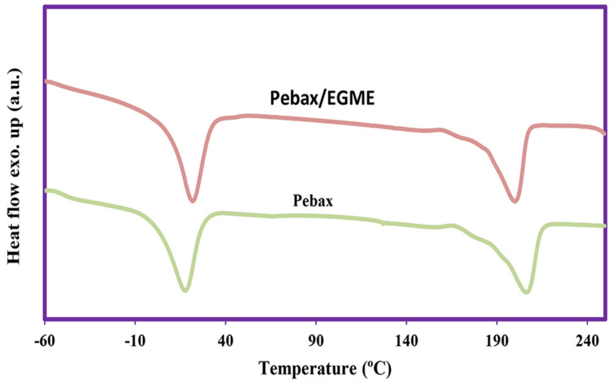

Figure 4. DSC Thermograms of the Prepared Membranes

Table 1. The Physical and Thermal Characteristics of the Prepared Membranes

Membranes	T_g (°C)	$T_{m,PE}$ (°C)	$T_{m,PA}$ (°C)	$\Delta H_{m,PE}$ (J/g)	$\Delta H_{m,PA}$ (J/g)	$X_{c,PE}$ (%)	$X_{c,PA}$ (%)
Pebax	-53.6	17.7	206.4	20.0	21.2	20.0	23.1
Pebax/EGME	-50.2	21.8	199.6	18.1	19.6	18.1	21.3

The mechanical characteristics of both Pebax and Pebax/EGME membranes were examined utilizing a tensile testing apparatus, with the findings presented in (Figure 5) and (Table 2). The results indicate that the Young's modulus of the membrane was enhanced twofold with the incorporation of EGME in comparison to the pure membrane. Furthermore, the tensile strength of the Pebax/EGME membrane exhibited a notable increase relative to that of the pure membrane. This substantial enhancement in tensile strength and Young's modulus attributed to the addition of EGME may be linked to the intercalation of its molecules within the Pebax chains, facilitating the creation of multiple hydrogen bonds. The molecules of EGME have contributed to the rigidity and strength of the membrane by creating hydrogen bonds with both the polyether and polyamide segments of Pebax. It is evident that the elongation at break of the Pebax/EGME has experienced a slight reduction compared to the pure membrane, likely due to the increased stiffness of the polymer matrix resulting from the aforementioned hydrogen bonding. This behavior suggests a trade-off between tensile strength and Young's modulus in relation to the elongation at break, which needs to be optimized based on the specific application. In membrane separation applications, a key characteristic of the membrane is its high mechanical strength and resistance to abrupt deformation. Therefore, both tensile strength and Young's modulus are critical; however, it is also essential for the membrane to possess sufficient elasticity to avoid brittleness.

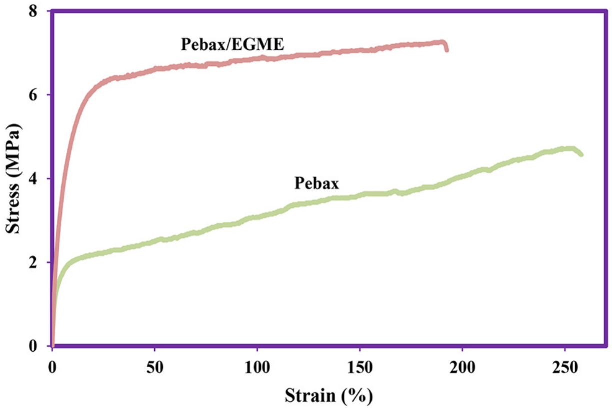

Figure 5. Stress-strain Curves of the Prepared Membranes

Table 2. Mechanical Properties of the Prepared Membranes

Membranes	Young's modulus (MPa)	Tensile strength (MPa)	Elongation at break (MPa)
Pebax	87.5	4.7	253.2
Pebax/EGME	174.6	7.2	191.2

3.2. Gas Permeation Properties

The CO_2 permeability of the Pebax and Pebax/EGME membranes is illustrated in (Figure 6). The results reveals a significant enhancement in CO_2 permeability by incorparation EGME to Pebax. Specifically, the incorporation of EGME to pebax matrix leads to increase in CO_2 permeability from 83 Barrer to 287 Barrer, representing an approximate increase of 3.5 times. (Figure 7) shows a schematic representation of the performance of the Pebax incorporating EGME. In the pure Pebax membrane, a dipole-quadrupole interaction occurs between the CO_2 molecules and ether groups of the Pebax chain. EGME creates three distinct types of interactions, which significantly enhance CO_2 permeability. 1. A Lewis acid-base interaction between $CO_2$and the hydroxyl group of EGME; 2. A dipole-quadrupole interaction between the ether group in the structure of EGME and CO_2 molecules; 3. A π-quadrupole interaction involving the π electrons of the aromatic ring in EGME and CO_2 molecules (Meshkat et al., 2019; Sanaeepur et al., 2019). Due to the lower electronegativity of carbon atoms compared to oxygen, the CO_2 molecules exist as transient quadrupolar, allowing them to interact with dipolar ether groups. This transient quadrupole can also interact with the π bond present in the aromatic ring of EGME. Moreover, oxygen atoms of hydroxyl groups of EGME have negative charges, while the corresponding hydrogen atoms are positively charged and can act as acidic hydrogens. CO_2 has two couples of carbonyl groups with a positively charged center carbon that can be attracted by the electron withdrawing oxygen atoms of EGME. This Lewis acid-base interaction has the potential to help a better transport of CO_2 through the membranes. The addition of EGME into the polymer matrix leads to remarkable increase in CO_2 permeability by 247%, driven by the synergistic effects of these three interactions. As illustrated in (Figure 6), the permeability of N_2 exhibited a moderate increase with the incorporation of EGME. This enhancement in N_2 permeability can be attributed to the liquid state of EGME. Due to its liquid nature, this substance offers limited resistance to N_2 diffusion when situated between polymer chains, allowing N_2 gas to infiltrate the Pebax/EGME membrane more readily than in the pure membrane. Notably, the increase in CO_2 permeability was substantial in comparison to the reduction in N_2 permeability, resulting in an overall enhancement of CO_2/N_2 selectivity from 45.8 to 68.1 with the addition of EGME to the Pebax membrane, representing a 49% increase. (Figure 8) shows the long-term stability of Pebax/EGME separation performance. It is clear that the CO_2 permeability and CO_2/N_2 selectivity had a limited decline in the initial times, but subsequently had a relatively stable trend, which enhances the membrane's operability in industrial applications.

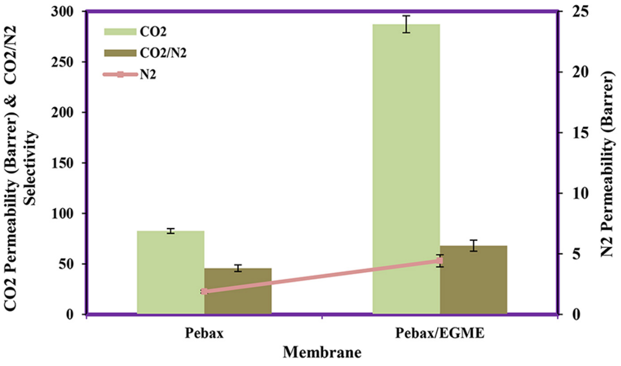

Figure 6. Effect of EGME on CO_2 Separation Properties

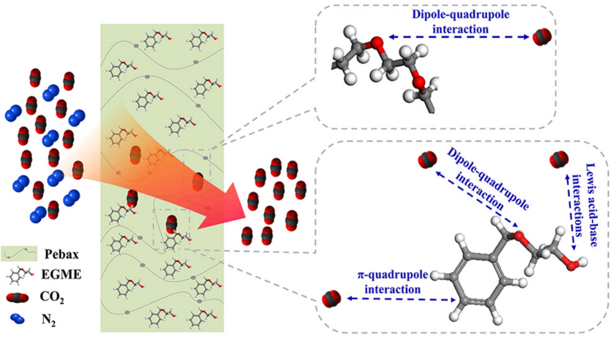

Figure 7. Schematic of CO_2 Separation Mechanism in Pebax/EGME Membrane

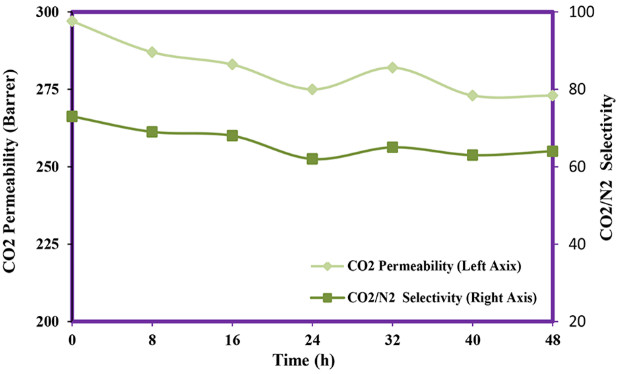

Figure 8. The Long-term Stability of Pebax/EGME Separation Performance

3.3. Comparison with other Works

(Figure 9) shows the positioning of the membrane developed in this study in comparison to the pure Pebax membrane, as well as several membranes created in recent years for CO_2 separation. The figure indicates that the separation performance of the pure membrane falls short of the Robeson upper bound line. However, the incorporation of EGME enhances the separation performance of the membranes, placing them above the Robeson upper bound line. This indicates that simultaneous increase in both CO_2/N_2 selectivity and CO_2 permeability created by addition of EGME, effectively surpassing the Trade-off limitation. Furthermore, when evaluating the performance of the membrane developed in this study against that of other membranes prepared in recent years, it can be concluded that the performance of the membranes produced here is comparable to that of the majority of membranes created by researchers. This suggests that EGME, as an easily obtainable compound, has significantly enhanced the separation efficiency of the Pebax polymer membrane.

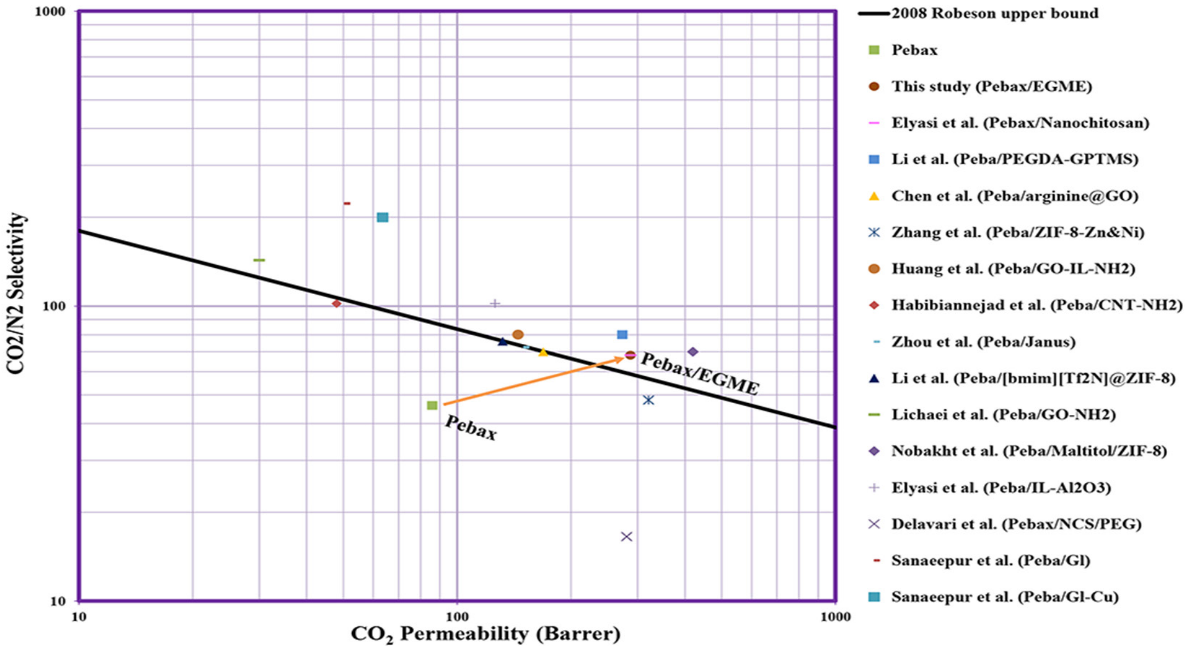

Figure 9. Separation Performance of Prepared Membranes Compared to Some Recent Studies (Chen et al., 2022; Delavari et al., 2024; Elyasi and Norouzi, 2025; Habibiannejad et al., 2016; Huang et al., 2018; Kojabad et al., 2021c; Li et al., 2016; Li et al., 2023; Lichaei et al., 2022; Nobakht and Abedini, 2023; Sanaeepur et al., 2019; Zhang et al., 2018; Zhou et al., 2015)

4. Conclusions

In this study, Pebax/EGME polymer membranes were prepared for CO_2/N_2 separation by incorporating EGME into the Pebax matrix. Following this, an examination of the chemical structure, morphology, physical and thermal properties, tensile strength, and separation performance of the resulting membrane was conducted, with the findings being thoroughly analyzed. The FTIR analysis revealed that the interactions between the Pebax and EGME polymer chains were predominantly of a physical type. The FESEM images of the membranes illustrated that the addition of EGME resulted in a rougher and more brittle membrane texture. The results of the DSC analysis showed that the crystallinity of the polyamide and polyether components of Pebax was diminished due to the incorporation of EGME between the polymer chains, which facilitated the formation of multiple hydrogen bonds between these components. Furthermore, the EGME-modified membrane exhibited significantly increased Young's modulus and tensile strength compared to the pure Pebax membrane. The gas separation performance assessment of the prepared membranes demonstrated that the inclusion of EGME in the Pebax matrix resulted in 247% increase in CO_2 permeability, driven by interactions such as dipole-quadrupole, Lewis acid-base, and π-quadrupole interactions. Moreover, the CO_2/N_2 selectivity of the Pebax/EGME membrane improved by 49% compared to that of the pure membrane. This enhanced performance allowed the Pebax/EGME membrane to surpass the Robeson upper bound, demonstrating the significant potential of EGME for improving Pebax-based CO_2 separation. The elevated CO_2 permeability of the membranes developed in this research diminishes the surface area necessary for application in the CO_2 separation application, thereby offering membrane systems that are optimally sized and reasonably priced.

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جداسازی CO_2 ارتقا یافته با استفاده از غشای Pebax اصلاح شده با اتیلن گلایکول مونو فنیل اتر

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چکیده

جداسازی CO_2 یکی از چالش‌های اساسی دنیای امروز می‌باشد که به دلیل رشد صنایع اتفاق افتاده است. روش جداسازی غشایی روشی نوین برای جداسازی CO_2 می‌باشد که غشای پلیمری پلی اتر بلاک آمید (Pebax) یکی از غشاهای صنعتی در این راستا می‌باشد. این نوع غشا محدودیتی به نام محدودیت Trade-off دارد که مانع از کاربرد گسترده آن در صنعت می‌شود. در این مطالعه به‌منظور غلبه بر این محدودیت، از اتیلن گلایکول مونو فنیل اتر (EGME) به‌عنوان پرکننده در زمینه Pebax استفاده شده و تأثیر آن بر روی ساختار شیمیایی، موفولوژی، خواص فیزیکی و حرارتی و نیز خواص جداسازی این پلیمر مورد بررسی قرار گرفت. نتایج نشان داد با افزودن EGME غشای تهیه شده تردتر و سفت‌تر شده و استحکام کششی و مدول یانگ برای غشای حاوی EGME نسبت به غشای خالص به ترتیب ۵۳ و ۹۹/۵ درصد افزایش داشت. علاوه بر این عبور دهی CO_2 و انتخاب‌گری CO_2/N_2 به دلیل برهمکنش‌های اسید-باز لوئیس، دوقطبی- چهارقطبی و π-چهارقطبی برقرار شده بین مولکول‌های EGME و CO_2 به ترتیب ۲۴۷ و ۴۹ درصد ارتقا یافت به‌طوری که با این بهبود عملکرد، غشای Pebax حاوی EGME توانست از مرز بالایی رابسون عبور کرده و بر محدودیت Trade-off غلبه کند که نشانی بر نقش کلیدی EGME در بهبود عملکرد جداسازی Pebax بود. علاوه بر این، عملکرد جداسازی غشاهای تهیه شده با غشاهای ساخته شده توسط دیگر محققان قابل مقایسه بود و از اکثرشان بهتر بود.

واژگان کلیدی: جداسازی CO_2، غشای پلیمری، پلی اتر بلاک آمید، اتیلن گلایکول مونو فنیل اتر، محدودیت Trade-off