**Tuning Reverse-Selective Polysulfone Membranes into Molecular-Selective Membranes via Zeolite-13X Incorporation under Low Pressure Operation**

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**Abstract.** The separation of CO₂/CH₄ mixtures is a critical step in biogas upgrading, where high-performance membranes are essential to improve purification efficiency. In this study, a mixed matrix membrane (MMM) was prepared by incorporating a low loading (0.5 wt%) of Zeolite-13X into a polysulfone (PSf) matrix. The structural and chemical properties of the filler and membranes were characterized using FTIR and XRD, while gas separation performance was evaluated through single-gas permeation tests using ultra-high purity CO₂ and CH₄. The addition of Zeolite-13X modified the separation mechanism from reverse-selective to molecular-selective, leading to a maximum ideal selectivity of 3.29—representing a 545.9% improvement compared to the pristine PSf membrane. These findings indicate that even a small fraction of zeolite filler can significantly enhance membrane selectivity, underscoring the potential of MMMs for efficient and sustainable biogas purification.

# Keywords: Gas Separation, Mix Matrix Membrane, PSf Membrane, Reverse-Selective

## INTRODUCTION

The rise of global warming caused the increases of world awareness in environmentally technology utilizations, neither in industrial scale nor in household scale. In industrial scale, gas emission treatment is important to prevent the increases of global warming effect [1,2]. Therefore, it is important to apply the gas separations technology which use the efficient of energy, low-cost production, and long-term utilization. For now, gas separation technology which is popular used in industrial are absorption, adsorption, cryogenic distillation, and membrane [3,4]. Based on these four technologies, membrane is considered is the most efficient.

In general, membrane can be defined by the selective semipermeable layer which can separate two or more mixture in liquid or gas phase [5–8]. Other technologies have the drawbacks in the gas separation process, such as high energy needed for the entire process, difficulty in material regeneration, and used the hazardous chemical on the process [3]. Separation process through the membrane is use the filtering in molecular scale. This process happens when one molecule in the mixture has the appropriate kinetic diameter with membrane pore will be easily pass through the membrane than the other larger particles [9,10]. Membrane performances in gas separation can be improved by modification on membrane pores diameter.

Based on the material, membrane classified to polymer membrane and inorganic membrane. Inorganic membrane has advantages like high of selectivity and good of thermal and mechanical stability. However, inorganic membrane is bad gas permeability, quite complicated on fabrication, and higher cost production compared by polymer membrane. Besides the low-cost production, polymeric membrane has the higher permeability, but polymer membrane has a lack of thermal and mechanical stability which is plasticization in high temperature uses [3]. Moreover, polymer membrane has the lower selectivity value compared by inorganic membrane. This trade-off permeability and selectivity of polymer and inorganic membrane have explained by Robeson Curve 2008. Recently, the mix matrix membrane (MMM) has developed by various researchers, which is modification in polymer membrane matrix by adding inorganic materials as fillers [11–13]. This modification has reported can increase the gas separation performance in membrane.

In fact, filtering in molecular level or also called molecular sieving is not the only mechanism of gas separation process in membrane. Gas separation process in membrane can occur by convective flow, Knudsen diffusion, and solution diffusion, depending on the molecular diameter kinetic and pore size of membrane. Separation on membrane does not always occur based on molecular size. The larger particle can pass through easily than the smaller particle in the mixture under the certain conditions. This phenomenon called ‘reverse-selective’ mechanism. Reverse-selective mechanism can occur due to the high and asymmetric pore size of membrane and under low operating pressure condition. It is having a same mechanism with size exclusion chromatography (SEC). Beads in the column can provide the path for the larger molecule to passes the column, whereas the smaller molecule stuck at the beads [14]. Membranes with this separation mechanism are suitable for specific applications, such as H2/CO2, N2/CO2, or liquid phase like ionic separation with PEG [14–16]. However, reverse-selective mechanism does not appropriate for purifying biogas applications. In the process, CO2 must be the permeate side will passes to container and utilized for other aspects with CCUS scheme. Besides, the retentate side must be CH4 will be utilized as power plant [3]. Various researcher reported synthesized the Polysulfone (PSf) membrane and it show the reverse-selective mechanism in the CO2/CH4 process [17]. This study aims to modify the behaviour of polysulfone (PSf) membranes, which typically exhibit reverse selectivity in CO₂/CH₄ separation, and convert them into molecular sieving membranes. To achieve this, Zeolite-13X (FAU) was incorporated as a filler in the fabrication of mixed matrix membranes (MMMs), with different loadings examined for their effect on separation performance. Zeolites are well-suited for this purpose because of their uniform microporous structures, high surface area, and tunable adsorption properties that can discriminate molecules based on size and affinity [18–20]. In particular, Zeolite-13X, with its relatively large pore openings (≈7.4 Å) and strong affinity for CO₂ due to the presence of cations within its framework, is expected to enhance CO₂ uptake while suppressing CH₄ transport. These characteristics make it an effective additive to shift PSf membranes from reverse-selective toward molecular sieving behavior.

## EXPERIMENTAL

## Materials

Polysulfone (Udel 3600) selected as membrane polymer with N-methyl-2-pyrolidone (NMP, 99%) purchased from Merck. Zeolite 13X FAU as membrane filler purchased from Guangdong Xintao Technology Co., Ltd,. CO2 and CH4 gases (UHP, 99.9%, PT> Samator Indonesia) was used for gas permeation test.

## Membrane Preparation

The pre-treatment of Zeolite-13X was conducted by stirring the material in acetone and followed by washing with aqua DI to remove any impurities during packaging. Next, the zeolite-13X is dried at 80°C for 24h. The dried Zeolite-13X (*x* wt% relative to the Psf mass) was then dispersed in NMP solvent, after which PSf was added to the solution at a PSf/NMP ratio of 20:80 wt%. The resulting dope solution was stirred at room temperature for 24 h and subsequently sonicated for 10 min to remove microbubbles formed during stirring. Membranes were fabricated using the non-solvent induced phase separation (NIPS) method. The dope solution was poured onto a glass plate and spread using a stainless-steel roller. The casted film was immediately immersed in a coagulation bath containing distilled water, where phase separation occurred, resulting in the formation of a solid, glassy polymer membrane. The membranes were kept in the coagulation bath for 24 h at room temperature and then dried at room temperature under ambient air for another 24 h to remove residual water. The detailed compositions of PSf, solvent, and filler are provided in Table 1.

**Table 1. Dope Solution Compositions**

|  |  |  |
| --- | --- | --- |
| **Samples** | **Psf (%wt)** | **Zeolite 13X (%wt)** |
| PSf | 20 | 0 |
| MMM 0.5% | 0.5 |
| MMM 1% | 1 |
| MMM 1.5% | 1.5 |

## Characterization

Zeolite 13X and membranes were characterized by Fourier Transform Infrared (FTIR) to identify the chemical groups existence and X-Ray Diffraction to determine the crystal structures.

## Gas Permeation Measure

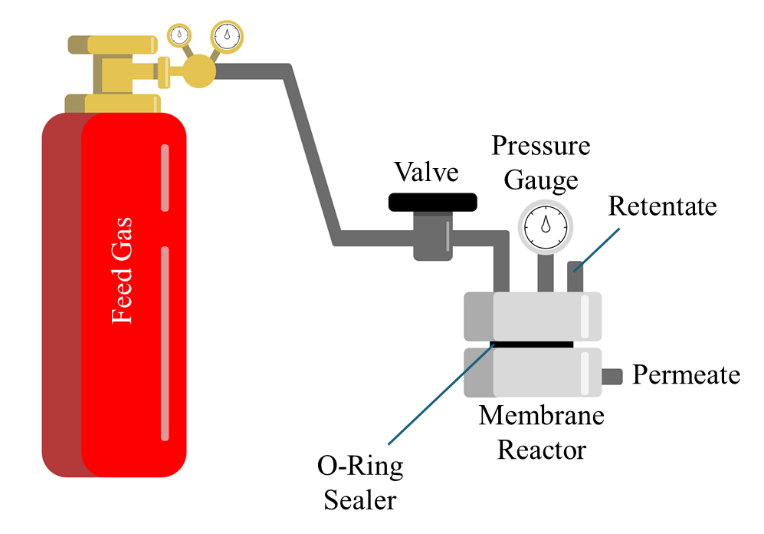


Figure 1 Schematic of Gas Permeation Test Apparatus

The separation performance of membranes identified by single gas permeation test, refers to Li et al. (2022). Membranes cut into circle with diameter of 4.78 cm, then it put into membrane reactor with O – ring rubber as a sealer of membrane. Membrane reactor then connected to gas cylinders. Membranes tested with CO2 and CH4 UHP (99.9%) as feed in 0.1 bar. Specifications and parameters in gas permeation test described in Table 2. Also, the schematic of gas permeation test apparatus shown on Figure 1. The time of gas passed through the membrane recorded with 5 times repetition. Permeability of membranes were calculated based on the equation below.

Pi = (1)

where Pi is the permeability value of gas i (GPU), is pressure change in unit of time (s), A is the area of tested membranes (cm2), P1 is the pressure of feed gas (bar), T is atmosphere temperature (K). The ideal selectivity of membrane (αCO2/CH4) can be calculated by the equation (2).

(2)

**Table 2. Specifications and Parameters of Gas Permeation Test**

|  |  |
| --- | --- |
| Specification | Value |
| Permeate Volume (cm3) | 25 |
| Feed Gas Pressure (bar) | 1,025 |
| Temperature (K) | 298 |
| Area (cm2) | 17,94 (r = 2,39 cm) |
| Gases | H2 (UHP 99,99%); CO2 (UHP 99,99%) |

## RESULTS

## Membrane Morphology

Figure 2 shows the PSf/Zeolite-13X membranes prepared with different filler loadings. At 0.5 wt% (a), the membrane looks smooth and fairly uniform, which suggests that the zeolite is well dispersed in the polymer matrix. At this low concentration, the polymer chains can still accommodate the filler without noticeable aggregation, and this agrees with the higher selectivity measured in the gas permeation tests. When the loading is increased to 1.0 wt% (b), small irregularities begin to appear, likely due to partial particle aggregation or voids at the polymer–filler interface, which reduces separation performance. At 1.5 wt% (c), the surface becomes less uniform and rougher, a sign of poor compatibility between the polymer and zeolite at higher loadings. These defects can act as non-selective pathways, explaining why the separation performance does not continue to improve at higher concentrations.

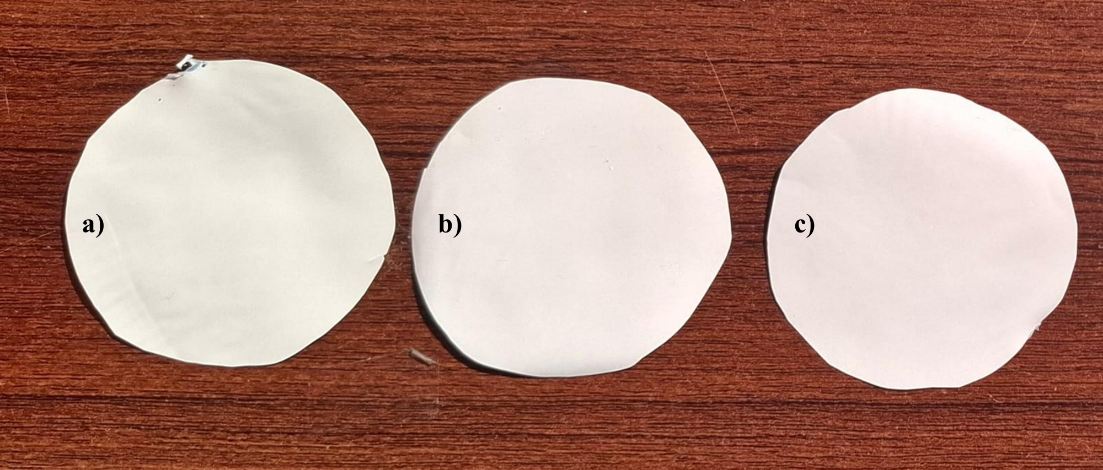
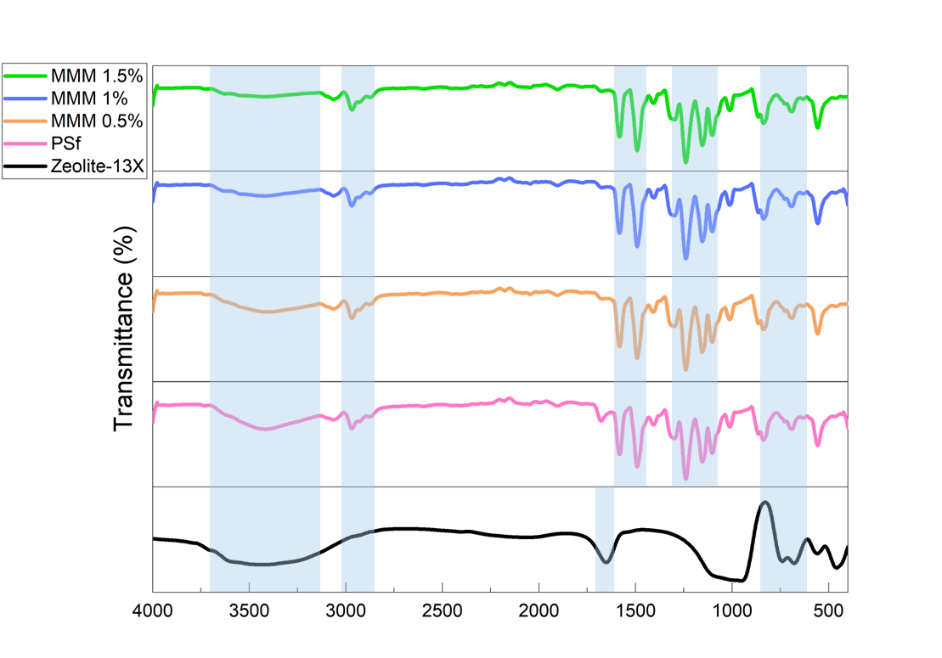


Figure 2 Photo image of the MMMs Psf/Zeolite-13X with filler loading of a) 0.5, b) 1.0 and c) 1.5 wt%

## FTIR Analysis

The FTIR spectra of Zeolite-13X and membranes shown on Figure 3. The Zeolite 13X absorbance peak measured at 974 cm-1 corresponding to vibration T-O-T bond, which T refers to tetrahedrally bonded Si or Al. Peak at 750, 560, and 665 cm-1 are corresponding to T-O-T stretching, double six membered rings of T-O-T stretching, and Si-O-Si stretching. Also, broad peak at 3500 cm-1 and small peak at 1600 cm-1 are corresponding to O-H vibration and stretch, respectively, because of the water molecule adsorption in Zeolite-13X ([21]). Low intensity peak of membranes showed at 2959 cm-1 is corresponding to aromatic C-H group, 1584 cm-1 is corresponding to C=C in polymer chain, 1485 cm-1 is corresponding to aromatic C=C in polymer structure. Asymmetric vibration on O=S=O in PSf polymer showed at 1146 cm-1 and 1102 cm-1. There is no difference in absorbance peak of all membranes. It indicates the interaction between membrane polymer and Zeolite 13X is a physical bond [22].

Figure 3. FTIR Spectra of Zeolite-13X and Membranes

## XRD Analysis

Diffractogram of Zeolite-13X showed diffraction peaks of 2θ = 6.18°, 15.6°, 18.6°, 23.6°, and 31.4° was suitable with JCPDS 12-0288. The diffraction peak in 2θ = 18.52° indicated the distinctive diffraction peak pf PSf. By the loading of Zeolite-13X in membrane matrix showed the slightly shift of PSf diffraction peaks in MMM, corresponding to the higher space results in membrane due to the low interaction between PSf and Zeolite-13X. The small peak showed at 2θ = 6.18° in diffractogram of MMM 1% and 1.5% showed the presence of Zeolite-13X pore (111) as filler in matrix membrane. This research was appropriately with previous paper which adding more the Zeolite-based materials in PSf presence small peak at 2θ ~ 6° [23,24]. So, the absence of peak of Zeolite-13X indicates the good dispersion and compatibility of Zeolite-13X in low concentration [22]. The XRD analysis revealed a noticeable shift in the characteristic diffraction peaks of Zeolite-13X after incorporation into the PSf matrix. Specifically, the peaks were shifted toward lower 2θ values, indicating an increase in the corresponding d-spacing according to Bragg’s law. This shift suggests a lattice expansion, which can be attributed to the interaction between the polymer chains and the zeolite framework. The increase in d-spacing further confirms that the filler was successfully dispersed within the polymer matrix, leading to structural modifications that may influence the transport pathways of gas molecules through the membrane [25].

A graph of different colored lines

AI-generated content may be incorrect.

Figure 4 XRD Diffractogram of Zeolite 13X and Membranes

## Gas Permeance Performance

Gas permeation measured by gas permeation test apparatus showed on Figure 1. CO2 and CH4 used as gas feed to membrane. The gas permeance and ideal selectivity presented at Table 3. The results of gas permeation test showed the increases of CO2/CH4 selectivity from PSf to MMM 0.5% reach 545.09% compared by PSf membrane, following the MMM 1% showed the decreases selectivity of CO2/CH4 reach 33.3% lower than PSf. Then, the unsignificant selectivity increases in MMM 1.5%. By noting the kinetic diameter of CH4 (0.38 nm) is higher than CO2 (0.33 nm), PSf here uses reverse-selectivity in the mechanism of gas separation. This phenomenon may be caused by the large pores generated during the casting process. The non-solvent movements quickly in membranes make the porous structure in matrix membrane. Also, PSf is a glassy polymer that contain the large excess free volume due to the large main chain of monomer [26]. After the loading of Zeolite-13X, the permeability of CH4 decrease following the increases of CO2 permeability. This mechanism followed the molecular sieving in membrane matrix. This would happen because of the decreases of pore size in membrane after Zeolite-13X loading into membrane matrix. There is the competitive sorption between CO2 and CH4 when passes the membrane, which CO2 which has smaller kinetic diameter can passes the membrane easier. Therefore, it makes the increases of CO2/CH4 selectivity. By the adding more of zeolite-13X (MMM 1 and MMM 1.5 wt%) showed the increases of CO2 and CH4 permeability, but the reverse-selectivity on membrane still happened. This might cause by the incompatibility PSf polymer chains with Zeolite-13X causes the void in membrane matrix. The void resulting the larger pore in membrane matrix, so the CH4 permeability increases.

**Table 3. Average of Gas Permeance Performance and Ideal Selectivity of Membranes**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Samples | PCO2 (GPU) | PCH4 (GPU) | αCO2/CH4 | MMM Selectivity Increases (%) |
| PSf | 3.47 | 6.72 | 0.51 | - |
| MMM 0.5% | 8.49 | 2.58 | 3.29 | 545.9 |
| MMM 1% | 3.52 | 10.34 | 0.34 | -33.3 |
| MMM 1.5% | 6.48 | 8.41 | 0.77 | 50.98 |

## CONCLUSION

MMM PSf/Zeolite 13X has successfully fabricated. The structure and performance of membrane have studied. The PSf membrane has the reverse-selective mechanism in separation of CO2/CH4 based on single gas permeation test. The addition low percentage of Zeolite-13X showed the unchanging structure of PSf membrane. Also, by addition low percentage of Zeolite-13X, the reverse-selective behaviour of membrane changes to the selective membrane. The MMM 0.5% showed the selectivity of CO2/CH4 increased reach of 545.9% than PSf membrane

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