Optimization of Polysulfone (PSf) Membrane Concentration for Enhanced H2/CO2 Gas Separation Performance

Kavitaningrum1, Nurul Widiastuti1, Ardi Nugroho Yulianto2, Triyanda Gunawan1,a)

Author Affiliations

1Department of Chemistry, Faculty of Science and Data Analytics, Sepuluh Nopember Institute of Technology Surabaya, Indonesia

2Department of Naval Architect, Faculty of Marine Engineering, Institut Teknologi Sepuluh Nopember; Sukolilo, Surabaya 60111, Indonesia

Author Emails

a) Corresponding author: triyanda@its.ac.id

**Abstract.** The increasing global demand for clean and renewable energy has encouraged the development of hydrogen (H₂) as a promising alternative fuel. Membrane-based gas separation has emerged as an efficient and environmentally friendly technology for H₂/CO₂ separation due to its low energy consumption, simple operation, and modularity. In this study, polysulfone (PSf) - based membranes were successfully fabricated using the dry phase inversion method, with polymer concentrations varied at 6%, 8%, 10%, 12%, 14%, and 16% to evaluate their effect on membrane morphology and gas separation performance. Structural characterizations using SEM, XRD, and FTIR confirmed that the PSf membranes exhibited symmetric dense morphologies, with the 10% PSf membrane producing an optimal dense layer thickness of 30.66 µm. Gas permeation tests using single gases (H₂ and CO₂) at 1 bar showed H2/CO2 selectivity values of 2.83; 3.24; 3.95; 3.47; 3.38; and 3.39 for sequential variations of polymer concentration. Among all tested concentrations, the 10% PSf membrane exhibited the highest selectivity, with an H₂ permeability reaching 3.45 GPU. This performance is attributed to balanced dense layer structure that is thick enough to maintain selectivity while allowing adequate permeability for small gas molecules like H2. These results confirm the potential of 10% PSf membrane for effective H2/CO2 separation in industrial applications.

**Keywords:** Gas Separation, H2/CO2 Selectivity, Polymeric Membrane, PSf Membrane, Solution-Diffusion Mechanism

## **INTRODUCTION**

The global push toward renewable energy is closely tied to the target of Net Zero Emissions (NZE) and the Paris Agreement, which aims to keep the rise in global temperature below 2 °C [1]. Hydrogen (H₂) has been widely discussed as one of the key energy carriers in this transition. It is efficient, sustainable, and its combustion leaves only water as the byproduct [2–4]. On a large scale, hydrogen is generally produced from coal gasification and natural gas reforming, followed by the water–gas shift reaction. The syngas stream produced from these processes contains about 30% CO₂ and 45% H₂, so a separation step is unavoidable [5–7].

Conventional methods such as absorption or cryogenic distillation are proven but come with drawbacks: they consume a lot of energy and often involve solvents that are not environmentally friendly [8–10].. Membrane separation, on the other hand, is much simpler. It is compact, energy-efficient, and relatively easy to scale up [11–13]. Glassy polymers are among the most used materials for gas separation membranes because their rigid chains create free volume that can discriminate gases by size [14–16]. Polyimides, for instance, give high selectivity but tend to plasticize under CO₂, while cellulose acetate is low-cost but easily degrades in humid conditions [17–20].

Polysulfone (PSf) is a polymer that combines good thermal and chemical stability with ease of processing [21,22]. Its aromatic and sulfone groups provide rigidity, while fabrication by phase inversion gives control over the dense selective layer. Most studies on PSf membranes have looked at mixed matrix or composite systems [15–17]. In contrast, systematic work on neat PSf membranes is still limited, even though such studies are useful. First, they establish a baseline to understand how polymer concentration affects both morphology and gas transport. Second, neat PSf membranes are easier and cheaper to produce, making them suitable as reference materials and as potential candidates for scale-up [23]..

In this work, we prepared PSf membranes by dry phase inversion with polymer concentrations ranging from 6 to 16 wt%. The membranes were characterized by SEM, XRD, and FTIR, and their H₂/CO₂ separation performance was tested in single-gas experiments at 1 bar and 298 K. The aim was to determine the optimum PSf concentration that provides the best balance between permeability and selectivity, while also offering insight for future development of PSf-based mixed matrix membranes.

## **EXPERIMENTAL**

## **Materials**

Polysulfone (Udel, Solvay) was used as the membrane polymer, while chloroform (CHCl₃, analytical grade, Merck) served as the solvent. High-purity gases, H₂ and CO₂ (UHP, 99.9%), were employed in the permeation experiments.

## **Membrane Preparation**

The casting solution (dope) was prepared by dissolving polysulfone (PSf) granules in chloroform, with polymer concentrations of 6, 8, 10, 11, 12, 14, and 16 wt% relative to the total solution weight. Prior to use, the PSf granules were dried overnight to remove residual moisture. The dried PSf was gradually added to chloroform and continuously stirred on a hot plate for 24 hours to ensure complete dissolution and homogeneity. The detailed composition of the prepared dope solutions is presented in Table 1. In this study, flat sheet membranes were fabricated using the dry phase inversion technique. The dope solution was cast onto a sterilized glass plate and spread evenly with a glass rod in a single pass. The cast film was left to stand in open air inside a fume hood for 5–7 minutes to allow solvent evaporation. Afterward, the membrane was carefully peeled from the glass plate and stored for further characterization.

**TABLE 1**. Dope Solution Compositions

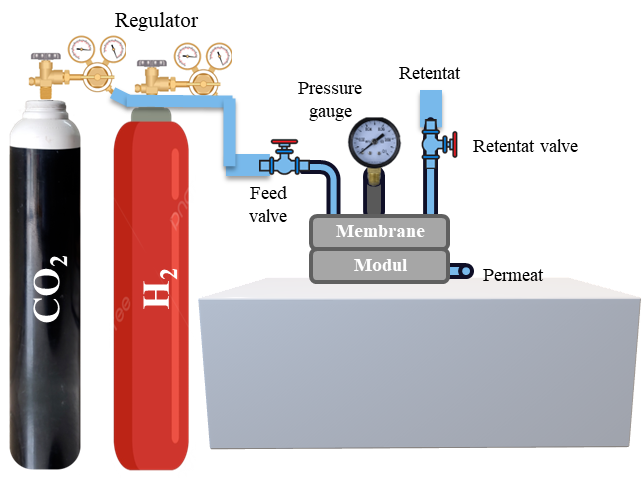
|  |  |  |
| --- | --- | --- |
| **Membrane Code** | **PSf Concentration (wt%)** | **Chloroform (wt%)** |
| PSf-6 | 6 | 94 |
| PSf-8 | 8 | 92 |
| PSf-10 | 10 | 90 |
| PSf-12 | 12 | 88 |
| PSf-14 | 14 | 86 |
| PSf-16 | 16 | 84 |

## **Characterization**

The fabricated PSf membranes were characterized by several techniques. X-ray diffraction (XRD) was used to examine the degree of crystallinity, while Fourier transform infrared spectroscopy (FTIR) was applied to confirm the presence of characteristic functional groups. Surface and cross-sectional morphologies were observed with scanning electron microscopy (SEM). Membrane thickness was estimated from cross-sectional SEM images, and the values were determined using ImageJ software. The images confirmed that the membranes had a dense, symmetric structure, with thickness varying depending on polymer concentration.

## **Gas Permeation Performance Test**

The membrane performance was evaluated through single gas permeation tests using H₂ and CO₂ gases. The schematic diagram of the permeation apparatus is shown in Figure 1. During the preparation stage, the membranes were cut into circular shapes to match the diameter of the permeation module. The specifications and parameters used in the gas permeation tests are listed in Table 2. The gas permeation rate was determined by measuring the time required for the feed gas pressure to decrease (change in pressure over time, ) and by monitoring the pressure difference between the pressure gauge installed on the permeation module and the permeate reactor throughout the test [24,25]. The membrane was considered to successfully allow gas permeation if, when the permeate reactor was closed, a pressure reading was observed on the pressure gauge. Each sample was tested five times to ensure reproducibility and reliability of the results.



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**FIGURE 1.** Single Gas Separation Performance Testing Apparatus

**TABLE 2.** Specifications and parameters used in the single gas permeation test

|  |  |
| --- | --- |
| **Specification** | **Value** |
| Reactor volume (cm3) | 25 |
| Feed gas pressure (bar) | 1.025 |
| Temperature (K) | 298 |
| Effective membrane area (cm2) | 17.94 (r = 2.39 cm) |
| Feed gas | H2 (UHP 99.99%)  CO2 (UHP 99.99%) |

The data processing of pressure change over time was used to determine the gas permeability value. Gas permeability measurements were conducted to evaluate the rate at which the gas permeates through the membrane. The permeability of the membrane was calculated using Equation 1 as follows.

(1)

where Pi is the permeability of gas i, expressed in GPU. The term represents the change in pressure (bar) with respect to time (s). V is the volume of the reactor (cm³), A is the effective membrane area (cm²), P2 is the feed gas pressure, and P1 is the atmospheric pressure during the test (both in cmHg). Subsequently, the permeability data can be processed to determine the gas selectivity value using Equation 2 as follows:

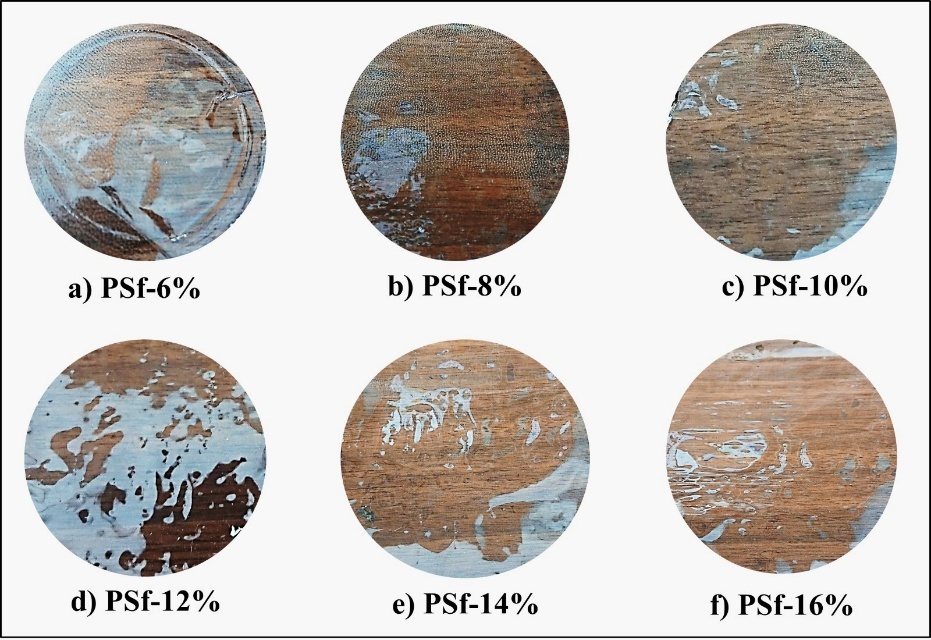
(2)

where α is the membrane selectivity for gas i over gas j, Pi is the permeability of gas i, and Pj is the permeability of gas j.

## **RESULTS**

## **Membrane morphology**

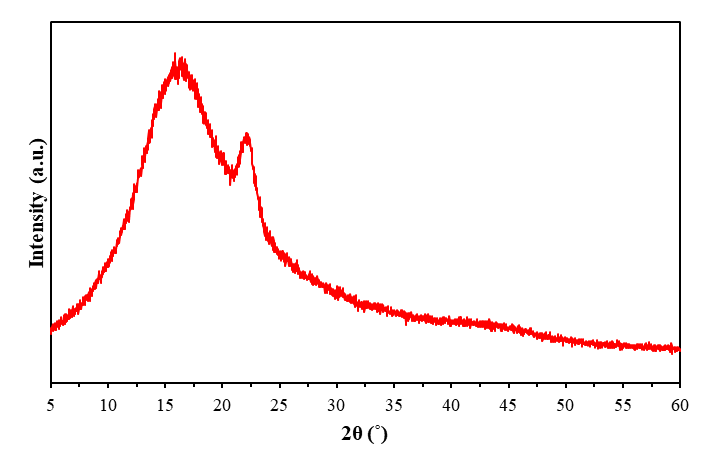
Figure 1 presents the appearance of PSf membranes at different concentrations. At 6% and 8%, the films look thin and fragile, with clear streaks and uneven surfaces, showing that the low polymer content could not form a stable layer. The 10% membrane appears more uniform, with a smoother surface compared to the lower concentrations. At higher concentrations (12–16%), the surfaces become thicker and more opaque, but irregular patches are visible, indicating non-uniform spreading during casting. These visual differences highlight the influence of polymer concentration on membrane formation, with 10% giving the most consistent surface.



**FIGURE 2** Macroscopic photograph of prepared membrane

## **XRD Analysis**

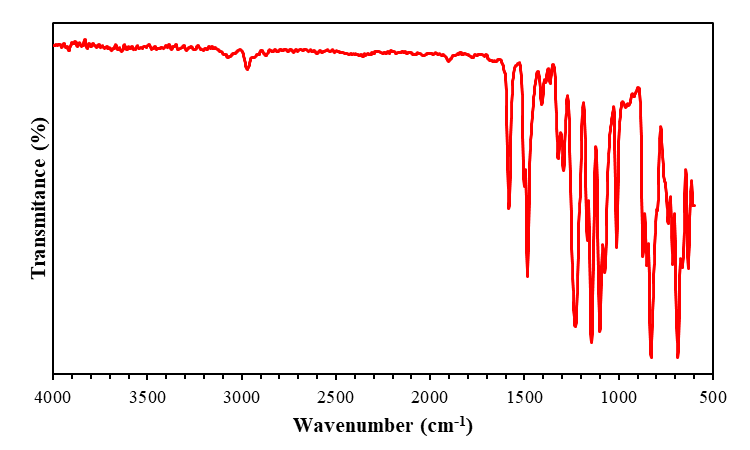
X-ray diffraction (XRD) analysis was performed to evaluate the structural features of the fabricated membranes. According to Figure 3, the PSf-based membranes demonstrated semicrystalline behavior, characterized by a broad amorphous peak at approximately 2θ = 17.6°. Moreover, both the neat PSf and mixed matrix membranes (MMMs) displayed a notable peak around 2θ = 22°, indicating the existence of crystalline phases in their structures [26]. The observed crystallinity is likely influenced by the systematic alignment of polymer chains, which takes place during the slow phase inversion process. In this study, membrane fabrication employed the dry phase inversion method, where the dope solution was left to evaporate in dry conditions, permitting a gradual solvent evaporation and a slower solidification process [27]. This controlled evaporation allows polymer chains sufficient time to rearrange into an ordered structure, contributing to the formation of crystalline domains.



**FIGURE 3.** Diffractogram Pattern of 10% PSf-Based Membrane

## **FTIR Analysis**

The FTIR spectra of the fabricated membranes are presented in Figure 4. It can be observed that the samples exhibit characteristic absorption bands corresponding to the functional groups typically found in PSf-based polymer membranes. At approximately 880–850 cm⁻¹, an absorption band attributed to C–H rocking vibrations is observed. In the range of 1170–1010 cm⁻¹, a C–C stretching vibration is detected, while bands at around 1140 and 1320 cm⁻¹ correspond to symmetric stretching of Ar–SO₂–Ar groups. Additionally, an absorption band at approximately 1230 cm⁻¹ is associated with Ar–O–Ar stretching, where Ar refers to the aromatic ring structure. A symmetric S=O stretching vibration appears at around 1290 cm⁻¹, and an absorption band at approximately 1580 cm⁻¹ is attributed to the C=C stretching of aromatic rings [28].



**FIGURE 4.** FTIR spectra of 10% PSf-based polymer membranes

**SEM Analysis**

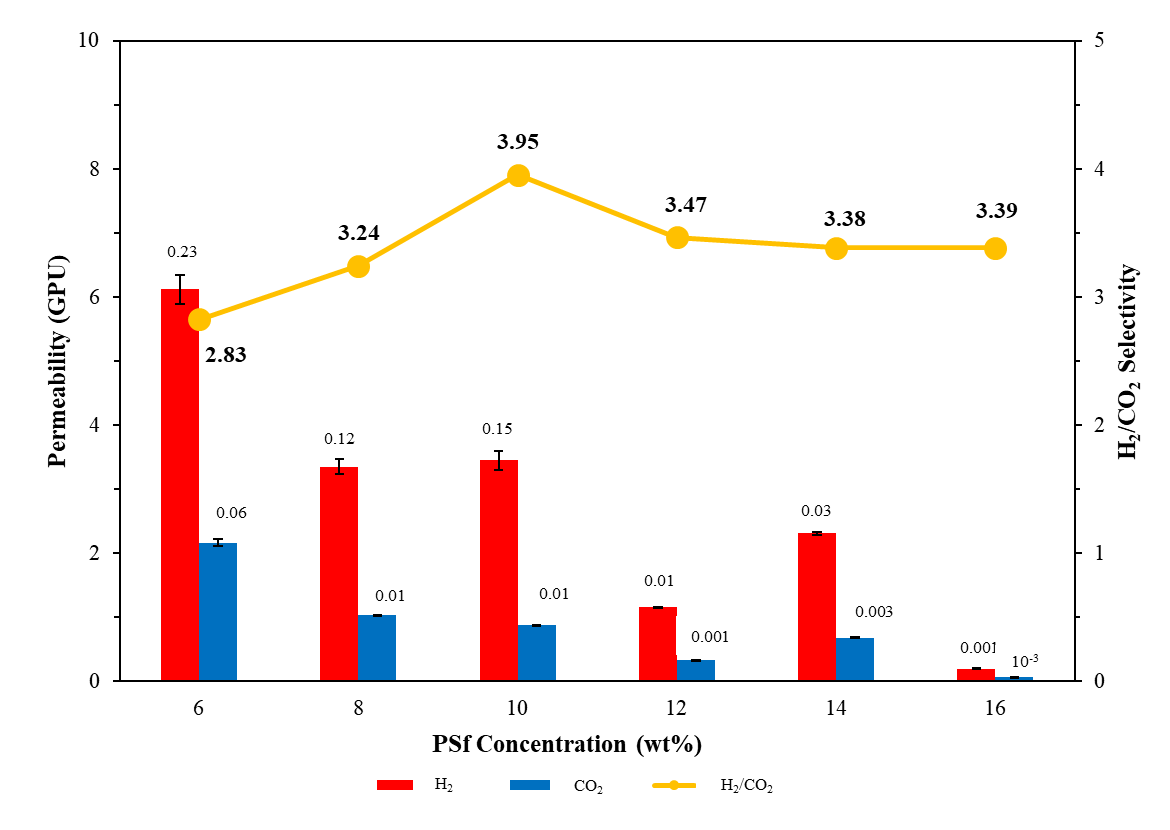
Figure 5 shows the SEM images of the PSf membrane surface and cross-section. The surface (Fig. 5a) looks generally smooth, with only a few small irregularities. The cross-sections (Fig. 5b–c) clearly show a dense and symmetric structure, with the selective layer measured at about 30.66 µm. This dense character agrees with the transparent appearance of the membranes seen in the photographs, confirming that the films produced here are essentially non-porous. Such a compact layer is important for gas separation because transport follows the solution–diffusion mechanism. Gas molecules dissolve into the polymer matrix, diffuse across the dense layer, and finally desorb on the permeate side. If the layer is too thin, defects can reduce selectivity, while if it is too thick, permeability drops sharply. The 10% PSf membrane sits in between, giving a thickness that is enough to minimize defects but still thin enough to let hydrogen, with its smaller kinetic diameter, pass more readily than CO₂. This balance is consistent with the higher H₂/CO₂ selectivity obtained for this membrane.



**FIGURE 5.** SEM images of PSf membranes fabricated with 10% polymer concentration (a) surface area, (b) cross section area, (c) cross section area with 3 times magnification compared to b area

## **Gas Permeance Performance Test**

The permeability results for H₂ and CO₂ at different PSf concentrations are shown in Figure 6. As expected, H₂ always passed through the membranes faster than CO₂ because of its smaller kinetic diameter (2.89 Å vs. 3.30 Å). At 6 wt%, the H₂ permeability was still high (6.12 GPU), but the selectivity was only 2.83. The membrane at this concentration looked thin and fragile, which likely introduced defects and allowed both gases to pass more easily. At 8 wt%, the permeability of H₂ dropped to 3.35 GPU while selectivity rose slightly to 3.24, showing that the denser layer started to form more effectively. The best result came at 10 wt%, where H₂ permeability was 3.45 GPU and the H₂/CO₂ selectivity reached 3.95. This matches the SEM observation of a uniform dense layer around 30.66 µm, thick enough to hold back CO₂ but not so thick that it blocked H₂. When the polymer concentration was increased further, the membranes became too dense. At 12 wt%, H₂ permeability fell to 1.15 GPU, and at 14 wt% it was 2.31 GPU. The thickest membrane, 16 wt%, had almost no permeability left (0.20 GPU), even though the selectivity stayed around 3.3. This shows the trade-off clearly: lower concentrations gave high flux but poor selectivity, while higher concentrations gave better selectivity but sacrificed permeability. The 10 wt% membrane sat in the middle, offering the most balanced performance.



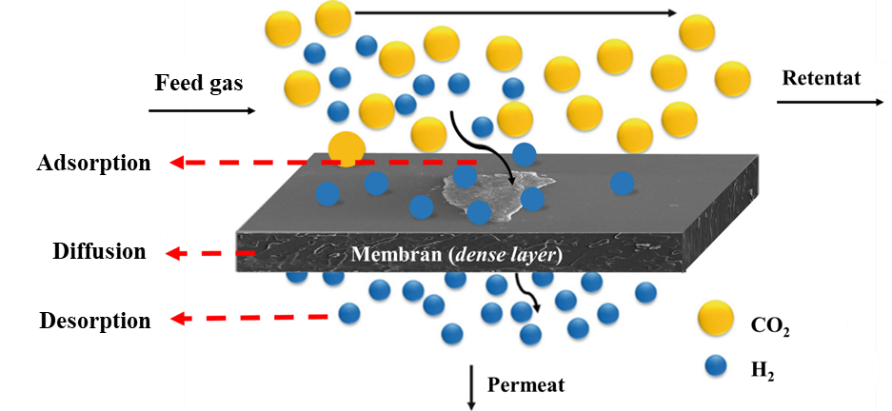
**FIGURE 6.** Single Gas Permeation Performance of PSf-Based Polymer Membranes

Complete data from single-gas tests for H₂ and CO₂ are presented in Table 3. The results indicate that the permeability of both H₂ and CO₂ decreases with increasing polymer concentration, suggesting that higher concentrations yield denser membranes and likely greater thickness, although this requires verification through cross-sectional SEM analysis for each concentration. In the fabrication of PSf-based membranes via the dry phase inversion method, the polymer concentration in the dope solution plays a decisive role in determining the thickness and morphological characteristics of the dense layer, as it directly affects the solidification rate and final membrane structure. At concentrations below 10% (e.g., 6% and 8%), the dope solution is overly dilute, resulting in an excessively thin and fragile dense layer. While this condition yields high gas permeability, it increases the likelihood of non-uniform porosity, defects, or voids, which compromise gas selectivity, and also raises the risk of pore interpenetration into the membrane interior, thereby weakening the solution–diffusion mechanism. Conversely, at concentrations above 10% (e.g., 12%, 14%, and 16%), the solution becomes more viscous, producing an overly thick dense layer that may preserve or enhance selectivity but significantly reduces permeability due to restricted gas diffusion. Quantitatively, the lowest reduction in H₂ permeability, 43.66%, occurs at a PSf concentration of 10%, while the highest reduction, 96.76%, is observed at 16%, both relative to the membrane with the lowest concentration. A similar trend is observed for selectivity, where the highest increase in H₂/CO₂ selectivity, 28.51%, is obtained at a PSf concentration of 10%. Although this improvement is relatively modest, it highlights the potential for further enhancement through the incorporation of additional materials, either in the form of composites or mixed matrix membranes (MMMs).

**TABLE 3.** Single gas separation performance at room temperature and 1 bar pressure

|  |  |  |  |
| --- | --- | --- | --- |
| **Membrane type** | **H2 Permeability (GPU)** | **CO2 Permeability (GPU)** | **H2/CO2 selectivity** |
| PSf – 6% | 6.12 ± 0.23 | 2.17 ± 0.06 | 2.83 |
| PSf – 8% | 3.35 ± 0.12 | 1.03 ± 0.01 | 3.24 |
| PSf – 10% | 3.45 ± 0.15 | 0.87 ± 0.01 | 3.95 |
| PSf – 12% | 1.15 ± 0.01 | 0.33 ± 0.001 | 3.47 |
| PSf – 14% | 2.31 ± 0.03 | 0.68 ± 0.003 | 3.38 |
| PSf – 16% | 0.20 ± 0.001 | 0.06 ± 0.0001 | 3.39 |

The membrane fabricated at 10% PSf concentration achieved an optimal balance between these two extremes, producing a dense layer with a thickness of approximately 30.66 µm. This specific thickness is critically important, as the gas separation process in these membranes relies on the solution-diffusion mechanism, which is driven by the pressure gradient between the feed and permeate sides. As illustrated in Figure 6, this mechanism consists of three sequential stages: adsorption, diffusion, and desorption [29]. In the adsorption stage, gas molecules dissolve at the membrane surface due to physical and chemical interactions with the membrane matrix, influenced by the gas’s solubility coefficient and the membrane’s affinity. More soluble gases are more readily absorbed. During the diffusion stage, gas molecules migrate through the membrane material from regions of higher to lower concentration, with factors such as molecular size and gas-membrane interactions affecting the diffusion rate. Smaller molecules like H₂ (kinetic diameter: 2.89 Å) diffuse faster than larger molecules such as CO₂ (3.3 Å) [30]. The 30.66 µm dense layer in the 10% membrane was sufficiently thick to restrict the passage of larger gas molecules while remaining thin enough to allow smaller gases like H₂ to diffuse effectively, resulting in the highest H₂/CO₂ selectivity observed in this study.



**FIGURE 7.** Gas Separation Mechanism via Solution-Diffusion

## **CONCLUSION**

PSf membranes were successfully fabricated using the dry phase inversion method, and their structure and gas separation performance were systematically investigated. The results demonstrated that polymer concentration in the dope solution significantly influenced membrane morphology and function, as higher concentrations produced thicker dense layers that reduced permeability while maintaining or enhancing selectivity. The optimum performance was achieved at 10% PSf, where the dense layer thickness reached 30.66 µm and resulted in an H₂/CO₂ selectivity of 3.95, representing a 73.9% improvement compared to the lowest polymer concentration. These findings highlight the critical role of polymer concentration in tuning membrane performance, provide a practical baseline for future mixed matrix membrane development, and indicate the potential feasibility of 10% PSf membranes for industrial gas separation applications.

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