**Effects of Post-treatment Low Temperature Heating under Vacuum on the P84 co-Polyimide Membrane Properties for Gas Separation**

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**Abstract:**

This study aims to evaluate the effect of post-treatment annealing on the P84 co-polyimide membrane for gas separation. Neat P84 polyimide films were cast on a glass plate and followed by heating at 35, 50, and 65 °C to examine how drying temperature below the glass transition influences structure and gas transport. X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM) were used to correlate microstructural changes with transport behavior. The 50 °C film showed a larger intersegmental spacing (d ≈ 5.57 Å) compared with the 35 °C film (d ≈ 3.97 Å), together with weaker O–H absorption and a smoother surface. Despite this, gas permeation tests revealed the opposite trend: CO₂ permeability dropped from about 95 Barrer at 35 °C to 3–4 Barrer at 50 °C, while H₂ decreased from ~260 to ~8 Barrer. Ideal selectivities remained broadly similar across all films. These results indicate that mild heating promotes surface densification and thermally induced relaxation, which lowers permeability even as average spacing increases. The study shows that sub-Tg drying provides a simple way to adjust microstructure and improve handling strength in P84 membranes without high-temperature treatment**.**

Keywords: P84 co-polyimide, gas separation, annealing temperature, thermally arranged, H2/CO2

# Introduction

Polyimide membranes are widely studied for gas separation because of their strong thermal stability, mechanical robustness, and resistance to solvents. P84, a co-polyimide derived from BTDA–TDI/MDI, is one of the most used commercial grades. It has a glass transition temperature of about 315 °C and is typically processed into dense films or hollow fibers [1–4]. Even though the polymer backbone is rigid, its final microstructure is strongly influenced by fabrication conditions, particularly the drying step [5]. Solvent evaporation, chain packing, and surface consolidation during drying can leave a measurable fingerprint on gas transport properties.

Much of the existing work on polyimides has focused on high-temperature treatments such as annealing close to Tg or thermal rearrangement (TR) above 350°C [6]. These processes are known to significantly increase free volume and permeability. For instance, Aguilar-Lugo et al. reported that TR polybenzoxazole membranes derived from ortho-functional polyimides achieved CO₂ permeability above 1000 Barrer with CO₂/CH₄ selectivity of 28, surpassing the Robeson 2008 upper bound [7]. Similar results were obtained in recent studies where thermal rearrangement improved chain rigidity, reduced aging, and enhanced tolerance to plasticization [8–10]. While effective, such treatments require high energy input and may complicate scaling up fabrication. In contrast, solvent removal and chain relaxation at sub-Tg conditions could also induce microstructural changes—such as surface densification, removal of bound species, and redistribution of free volume—that affect permeability [11–13]. Yet, these low-temperature effects remain poorly documented in the literature, leaving open the question of how routine drying steps might influence membrane performance.

The present work focuses on connecting these low-temperature drying effects with structural and transport data in P84 membranes. By combining XRD, FTIR, and SEM, we analyze how drying at 35–65°C tunes intersegmental spacing, functional group signatures, and surface morphology, and how these features correlate with single-gas permeation. The study emphasizes that even a routine processing parameter, drying temperature, can act as a simple knob to adjust membrane microstructure and transport, providing an alternative to more energy-demanding high-temperature modification routes.

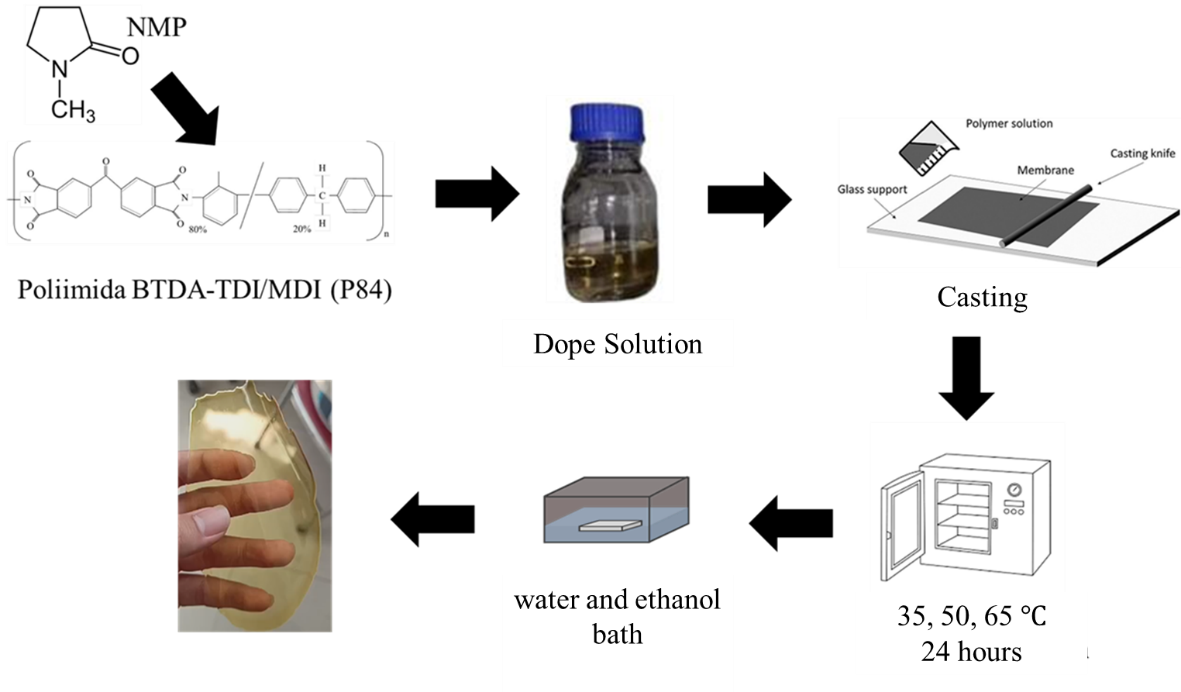
# Experimental

## Materials and Chemical

The polymer used in this study was P84 co-polyimide (BTDA–TDI/MDI, HP Polymer, Austria). N-methyl-2-pyrrolidone (NMP, Merck) served as the solvent. High-purity gases (N₂, CO₂, and H₂; 99.99%) were obtained from PT. Samator, Indonesia.

## Fabrication of P84 Membrane

Polyimide solutions were prepared by dissolving 15wt% of P84 co-polyimide in NMP following standard casting procedures [14–16]. The solutions were spread onto clean glass plates and dried in controlled ovens at 35, 50, and 65°C. These drying temperatures were selected to represent sub-Tg conditions where solvent removal and chain relaxation occur without thermal rearrangement, allowing the influence of low-temperature drying on morphology to be examined. Films were detached from the substrate and conditioned to constant weight prior to testing.



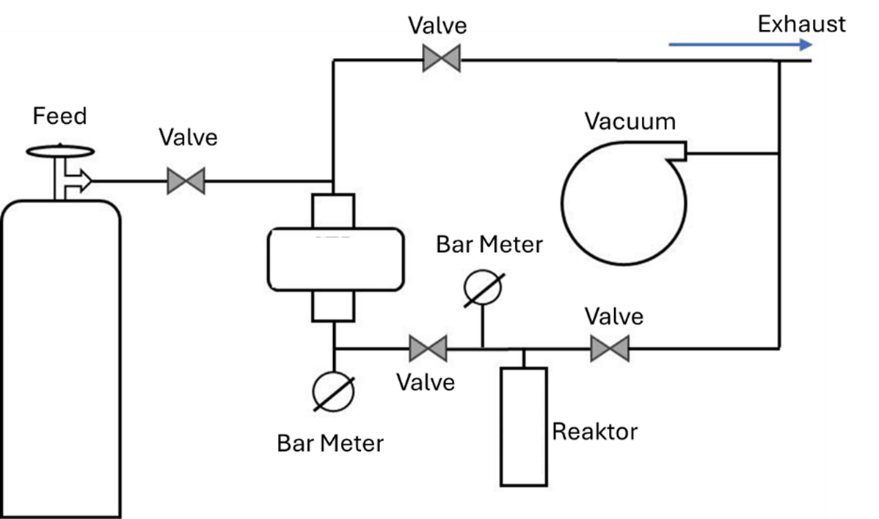
**FIGURE 1.** Schematic diagram of membrane preparation

## Sample Characterization

Neat P84 films were characterized using X‑ray diffraction **using,** Bruker D8 Advance diffractometer with Cu Kα radiation (λ = 1.5418 Å). Infrared spectra were collected by **FTIR** Thermo Scientific Nicolet iS10 at 2–4 cm⁻¹ resolution. Surface morphology was examined by **scanning electron microscopy using** Hitachi FE-SEM**,** Regulus 8220**.**

# Gas Permeation Testing

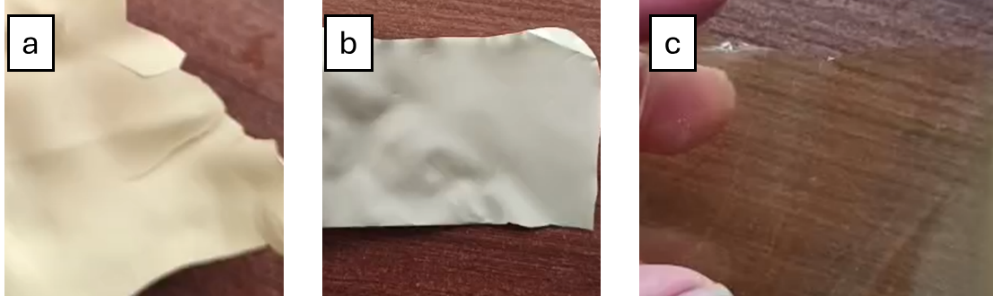
Single-gas permeation experiments were performed using CO₂, N₂, CH₄, and H₂ at 1 bar and 25 °C. Permeability was calculated using the standard constant-volume/variable-pressure method, and ideal selectivity (αA/B) was determined as the ratio of permeabilities (PA/PB) [17,18].



**FIGURE 2.** Schematic permation test

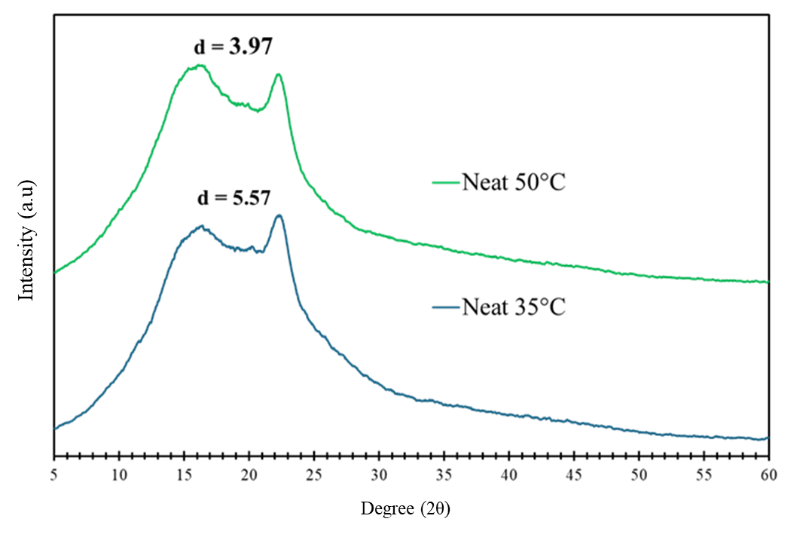
# Result and Discussion

Figure 3 shows the physical appearance of P84 films after vacuum drying at different conditions. The film obtained directly from the NIPS process without thermal treatment (Figure 3a) was very brittle and curled easily. It broke when bent and could not be used for gas permeation testing. The absence of a drying step left residual solvent in the matrix, resulting in weak chain packing and poor mechanical stability. After drying at 35°C (Figure 3b), the membrane became noticeably stronger. Unlike the untreated film, it could be bent and handled without breaking, indicating that mild heating already provided sufficient consolidation. The surface appeared smoother and more compact, and the film retained enough integrity for gas permeation testing. Importantly, this sample later showed the best separation performance with high permeability while maintaining reasonable selectivity. This combination suggests that drying at 35°C preserves free volume and sorption pathways, which support high gas uptake and transport. The membrane dried at 50°C (Figure 3c) showed further improvement in visual appearance and handling. The film was transparent and uniform, with a smoother surface compared to the 35°C sample. Handling strength was also good, allowing the membrane to be tested. However, the transport data told a different story. Permeability decreased sharply at 50°C, with CO₂ dropping to only 3–4 Barrer and H₂ to about 8 Barrer. This indicates that while the film gained mechanical stability, surface densification and stronger chain relaxation reduced the number of effective pathways for gas sorption and diffusion. At 65°C, the film again became brittle and cracked during handling, making it unsuitable for testing. The excessive sub-Tg heating likely caused stronger relaxation and rigidification of the chains, which stiffened the film but sacrificed toughness. Taken together, these results show that drying temperature strongly influences both the physical integrity and the gas transport properties of P84 membranes. The 35 °C film represented the best balance, with sufficient strength for handling and the highest permeability among all samples. In contrast, higher drying temperatures improved surface consolidation but suppressed permeability. This highlights the dual role of drying: it enhances handling properties, but if pushed too far, it can also restrict transport by over-consolidating the structure.



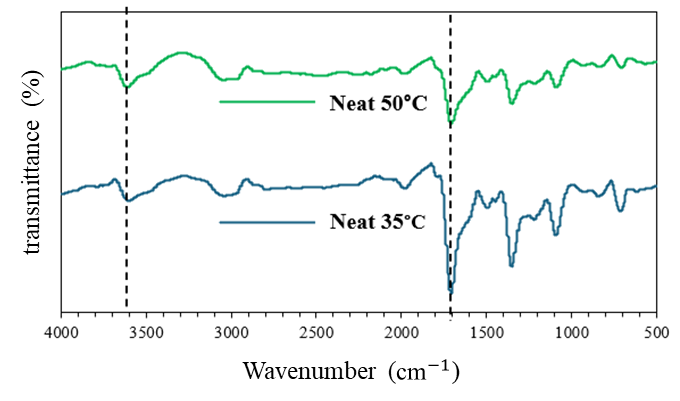
**FIGURE 3** Physical appearance of the prepared membrane

Figure 4 presents the XRD patterns of neat P84 films dried at 35 °C and 50 °C. Both samples show a broad amorphous halo typical of glassy polyimides, with only a faint shoulder at higher angles. After background subtraction and peak fitting, the main halo maximum is located at 2θ ≈ 22.5° for the 35 °C film and shifts to 2θ ≈ 15.9° for the 50°C film. Using Cu Kα radiation (λ = 1.5418 Å), these correspond to average intersegmental spacings of about 3.97 Å and 5.57 Å, respectively. The shift to lower angle for the 50°C film suggests that chain segments are more relaxed and spaced further apart, probably due to solvent being removed more effectively during drying. At 35°C, the smaller spacing may indicate that residual solvent still occupies part of the free volume and prevents large-scale rearrangements. However, these structural differences do not directly translate to transport behavior. Despite having the larger d-spacing, the 50 °C film showed much lower permeability, while the 35°C film with its smaller spacing displayed significantly higher gas flux. This mismatch implies that the average spacing measured by XRD does not fully capture the pathways relevant for permeation. A reasonable explanation is that the 50 °C film develops a denser outer region as chains relax and solvent escapes at the surface, which hinders gas sorption and slows diffusion. The interior may remain relatively open, but transport is controlled by the surface consolidation. This interpretation fits well with the FTIR and SEM results. FTIR showed weaker O–H absorption at 50°C, pointing to reduced bound species, while SEM images revealed a smoother and more compact surface. Together, the data suggest that sub-Tg drying changes the balance between bulk free volume and surface densification. The diffraction halo shift shows larger average spacing, but gas permeation is mainly determined by the denser surface layer formed at higher drying temperature [19].



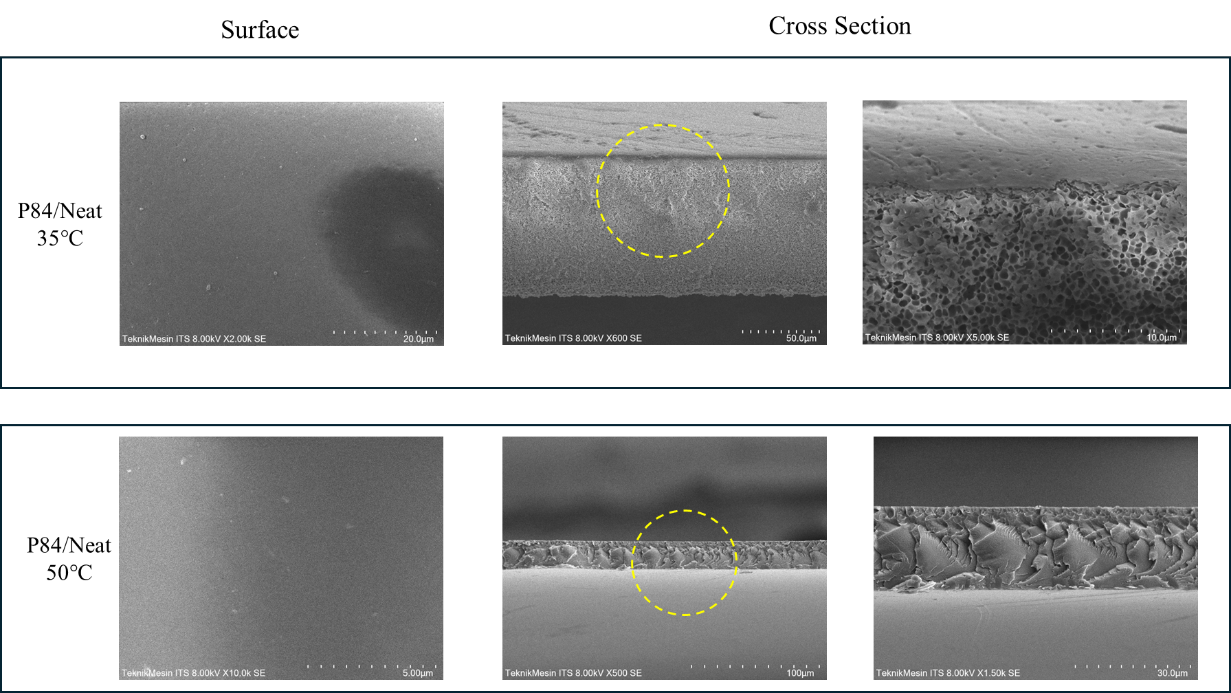
**FIGURE 4.** XRD diffractogram of P84 Neat membrane

Figure 5 compares the infrared spectra of neat P84 films dried at 35 °C and 50 °C. Both spectra show the characteristic absorption bands of fully imidized polyimide: the asymmetric and symmetric imide carbonyl peaks near 1780 and 1720 cm⁻¹, and the imide C–N stretching around 1375–1395 cm⁻¹ [20–22]. The presence of these bands confirms that the imidization process was complete and that the polymer backbone remained chemically intact under the drying conditions. The main distinction between the two films lies in the broad O–H stretching envelope in the range of 3200–3600 cm⁻¹ [23–25]. The 35°C film displays a stronger and wider O–H band, which indicates the presence of greater amounts of bound water and residual solvent molecules trapped in the polymer matrix. In contrast, the 50 °C film shows a noticeably weaker and narrower O–H feature, consistent with more effective removal of sorbed species during drying. These observations suggest that mild heating not only aids solvent evaporation but also reduces local heterogeneity in the polymer environment. This change in O–H absorption provides a molecular explanation for the differences observed in gas transport. The higher moisture and solvent retention at 35°C maintains a looser microstructure with more available sorption sites, resulting in higher permeability values. By comparison, the 50°C drying step consolidates the film surface and reduces sorbed species, which corresponds to the sharp decrease in CO₂ and H₂ permeability. Similar correlations between O–H content, free volume redistribution, and gas uptake have been reported in earlier studies of polyimides and thermally rearranged membranes [26]. Overall, the FTIR results support the interpretation that sub-Tg drying temperature alters the microstructure of P84 not by changing the backbone chemistry, but through differences in solvent removal and chain relaxation. At 35°C, residual sorbed species contribute to higher gas permeability, while at 50 °C their removal produces a denser structure that favors stability but suppresses transport.



**FIGURE 5.** FTIR of P84 Neat membrane

Electron micrographs reinforce this interpretation. The 35°C film presents a slightly textured surface with occasional shallow features, while the 50 °C film appears smoother and more compact at comparable magnification and imaging conditions. No macroscopic defects such as pinholes or tears were observed. The smoother surface at 50 °C is consistent with consolidation during drying and helps explain the lower permeabilities measured on that film. Viewed in the same field of view and magnification, the surfaces look distinct. The 35°C film shows a subtle, sponge‑like texture with shallow pits and ridges, suggesting a more heterogeneous outer region; in contrast, the 50°C film looks plain and compact, with few discernible features. This side‑by‑side appearance explains the transport behavior: the textured 35°C surface offers easier access to pathways and higher gas uptake, giving much higher permeability, whereas the smoother, consolidated 50°C surface throttles uptake and diffusion, so permeability falls even though the fitted halo indicates a larger mean spacing through the thickness. In this case, the excessive temperature that affects the polymer causes the molecules in it to have additional energy to relax, so that they have a tight and organized morphology, where this process is called thermally induced relaxation [27,28].



**FIGURE 6.** Scanning Electron Microscope of P84 Neat membrane

Table 1 summarizes the single-gas permeation results at 1 bar and 25 °C. The film dried at 35 °C gave the highest permeability for all gases tested. For instance, CO₂ permeability was about 95 Barrer at 35 °C but dropped sharply to around 3–4 Barrer at 50 °C. A similar trend was observed for H₂, which decreased from roughly 260 Barrer to less than 10 Barrer over the same temperature change. CH₄ and N₂ followed the same pattern, with values falling by two orders of magnitude between 35 and 50 °C. Selectivity values, on the other hand, did not change as drastically. For the CO₂/CH₄ pair, the ratio increased slightly from 0.66 at 35 °C to 1.19 at 50 °C, while H₂/CO₂ selectivity stayed in the range of 2–3 for all samples. This shows that the main effect of drying temperature is on permeability rather than separation factors. When combined with the XRD results, these data highlight an unusual behavior: the smaller intersegmental spacing at 35 °C is associated with much higher permeability, while the larger spacing at 50 °C corresponds to reduced permeability. This can be explained by the formation of a denser surface layer at 50 °C, which restricts sorption and diffusion even though the bulk spacing increases. At 35 °C, the less consolidated surface allows easier gas uptake and faster transport, giving superior performance.

**Table 1.** Gas Separation Performance of prepared membrane

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Temperature (oC)** | **Feed Pressure (Bar)** | **Gas** | | | | **Selectivity** | |
| **CH4** | **N2** | **CO2** | **H2** | **CO2/CH4** | **H2/CO2** |
| **Average Barrer** | | | |
| 35 | 1 | 143,48 | 101,09 | 94,87 | 259,52 | 0,66 | 2,74 |
| 50 | 3,03 | 2,58 | 3,62 | 7,63 | 1,19 | 2,11 |
| 65 | 2,18 | 1,49 | 1,25 | 3,45 | 0,58 | 2,75 |

# Conclusion

Drying temperature between 35 and 65 °C has a clear impact on the structure and properties of neat P84 membranes. At 35 °C, the films maintained sufficient mechanical strength for testing and showed the highest gas permeability, especially for CO₂ and H₂. Drying at 50 °C produced membranes with smoother and denser surfaces, larger average d-spacing, and improved handling, but with much lower permeability. At 65 °C, the films became brittle and unsuitable for testing. These results indicate that even modest sub-Tg drying steps can strongly affect both morphology and gas transport, mainly through surface consolidation and removal of bound species rather than changes in the polymer backbone. From a practical perspective, controlled drying also improves handling by reducing residual solvent and water, lowering the risk of cracks or defects in P84 films. Compared to the more common high-temperature approaches such as annealing and thermal rearrangement, the present work shows that simple low-temperature drying can already be used to tune membrane performance. For future work, it will be important to examine the stability of these membranes under higher feed pressures and mixed-gas conditions, where competitive sorption and plasticization effects may become relevant. Long-term aging studies are also needed to see whether the differences introduced by drying persist over time or converge as the films relax. These investigations will help clarify how sub-Tg drying can be used as a practical tool to balance permeability, selectivity, and mechanical robustness in P84 membranes.

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