

# Transdigital<sup>®</sup>

journal



Volume 7, Issue 13: January-June 2026

ISSN: 2683-328X

Sociedad de Investigación sobre Estudios Digitales S. C.



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FUNCTIONAL SEMILADDER POLYMERS  
FOR GAS SEPARATION:  
ASSESSING FEASIBILITY WITHIN AN  
INTEGRATED CATALYTIC-MEMBRANE SYSTEM

POLÍMEROS DE SEMIESCALERA  
FUNCIONALES PARA LA SEPARACIÓN DE GASES:  
EVALUACIÓN DE LA VIABILIDAD DENTRO DE UN  
SISTEMA INTEGRADO DE MEMBRANA CATALÍTICA

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Section: Research article

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Received: 07/10/2025

Accepted: 27/02/2026

## **POLÍMEROS DE SEMIESCALERA FUNCIONALES PARA LA SEPARACIÓN DE GASES: EVALUACIÓN DE LA VIABILIDAD DENTRO DE UN SISTEMA INTEGRADO DE MEMBRANA CATALÍTICA**

### **FUNCTIONAL SEMILADDER POLYMERS FOR GAS SEPARATION: ASSESSING FEASIBILITY WITHIN AN INTEGRATED CATALYTIC-MEMBRANE SYSTEM**

#### **RESUMEN**

El calentamiento global supone una amenaza creciente para todos los seres vivos y sus ecosistemas, lo que impulsa la necesidad de procesos industriales más eficientes y sostenibles. La separación de gases es un proceso indispensable para la purificación, el almacenamiento y la utilización de los gases producidos en la industria. Los métodos tradicionales, como la destilación criogénica y la absorción, son eficaces, pero consumen mucha energía y son complejos, mientras que las separaciones basadas en membranas ofrecen una alternativa más eficiente, más sencilla y modular, desde el punto de vista energético. La eficacia de la membrana viene determinada principalmente por el tipo de material utilizado en su fabricación y por sus propiedades (térmicas, químicas, etc.). En este trabajo, se centra la atención en los polímeros multifuncionales aromáticos de semiescalera que incorporan unidades de xanteno en su cadena principal, sintetizados mediante una policondensación robusta, en un solo paso y sin metales, de derivados de isatina con bisfenoles. Los materiales resultantes presentan una alta estabilidad química y térmica, lo que los hace muy adecuados para aplicaciones de separación difíciles. Los estudios de transporte de gases demuestran que las membranas basadas en bisfenol AF presentan una permeabilidad superior en comparación con las derivadas del 4,4'-bifenol. En particular, los polímeros de semiescalera fluorados que incorporan N-propil isatina muestran un mejor equilibrio entre permeabilidad y selectividad en múltiples pares de gases. Estos resultados subrayan el gran potencial de las membranas de polímeros de semiescalera como materiales energéticamente eficientes para tecnologías avanzadas de separación de gases. Aunque las membranas de polímeros de semiescalera se han probado con gases de pureza ultraalta, es fundamental evaluar su rendimiento con mezclas de gases reales para aplicaciones prácticas. Para ello, el sistema de membranas se integrará con un reactor catalítico para suministrar mezclas de gases representativas, lo que permitirá una evaluación más realista de las propiedades de transporte.

**Palabras clave:** membrana, polímero, CO<sub>2</sub>, separación, catalizador

#### **ABSTRACT**

Global warming poses a growing threat towards all living beings and their ecosystems, driving the need for more efficient and sustainable industrial processes. Gas separation is a process that is indispensable for purification, storage, and utilization of gases produced in industry. Traditional methods like cryogenic distillation and absorption are effective but energy-intensive and complex, whereas membrane-based separations offer a more energy-efficient, simpler, and modular alternative. The effectiveness of membrane is primarily determined by the type of material used in its fabrication and by its properties (thermal, chemical, etc.). In this work, attention is focused on semi-ladder aromatic multifunctional polymers incorporating xanthene units in their main chain, synthesized via a robust, one-step, metal-free polycondensation of isatin derivatives with bisphenols. The resulting materials exhibit high chemical and thermal stability, making them well-suited for challenging separation applications. Gas transport studies demonstrate that membranes based on bisphenol AF exhibit superior permeability compared to those derived from 4,4'-biphenol. In particular, fluorinated semi-ladder polymers incorporating N-propyl isatin show an enhanced permeability-selectivity balance across multiple gas pairs. These results underscore the strong potential of semi-ladder polymer membranes as energy-efficient materials for advanced gas separation technologies. Although semi-ladder polymer membranes have been tested with ultra-high-purity gases, evaluating their performance with real gas mixtures is crucial for practical applications. To address this, the membrane system will be integrated with a catalytic reactor to supply representative gas mixtures, enabling a more realistic assessment of transport properties.

**Keywords:** membrane, polymer, CO<sub>2</sub>, separation, catalyst

## 1. INTRODUCTION

The use of nonrenewable energy in several processes associated with fossil fuels is a major concern, as it is responsible for different forms of pollution such as global warming resulting from greenhouse gas emissions (GHG). Currently, extensive research endeavors are focused on the process of separation (into pure gases) (Hernández-Martínez et al., 2024). In this sense, bisphenols have become important due to their application and use in different processes and applications, especially in the production of resins and polycarbonate (Li et al., 2024).

Hydroxyalkylation of fluorinated ketones with catechol, resorcinol, or hydroquinone in superacid media enables the synthesis of bisphenols and condensation polymers containing xanthene units, producing high molecular weight polymers with rigid backbones and intrinsic microporosity (Zhili et al.). On the other hand, the incorporation of fluorine enhances the rotational mobility of these polymers, leading to improved gas transport properties, making these polymers ideal to form membranes with high selectivity for some gas pairs, although their performance remains constrained by the inherent permeability-selectivity trade-off (Robeson, 2008).

Polyhydroxyalkylation of isatin and N-alkylated derivatives (N-propyl, N-bromohexyl) react with various bisphenols to obtain functional xanthene-based polymers. Owing to their rigid backbones and tunable fractional free volume (FFV) these materials are attractive candidates for gas mixture separation processes, where membrane-based technologies offer potential economic advantages. Accordingly, structure-property relationships were evaluated in terms of chain rigidity, FFV, and gas permeability/selectivity. On the other hand, bromoalkylation enables post-polymerization functionalization via the Menshutkin reaction, introducing cationic groups to produce solid polyelectrolytes, further expanding their application in materials chemistry and anion transport membranes (Cracowski et al., 2012) and could be used in a gas (mixture) separation process.

In this context, the relevance of supplying the gas mixture through in situ chemical generation should be highlighted. The required gas stream can be produced via the reverse water-gas shift (RWGS) reaction carried out in a tubular catalytic reactor using nickel oxide (NiO) as the catalyst.

## 2. RESEARCH METHOD

### 2.1. Materials

All raw materials were obtained from Aldrich. Prior to use, the following materials underwent purification: dichloromethane, trifluoroacetic acid (TFA), and trifluoromethanesulfonic acid (TFSA) were distilled; while isatin, N-

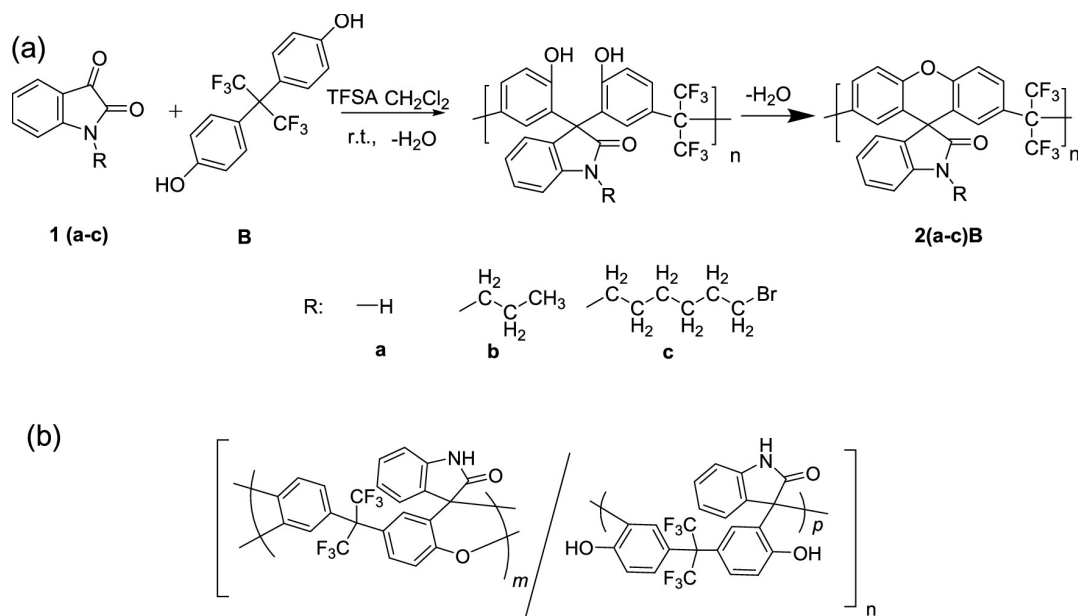
propyl isatin, and N-bromohexyl isatin were purified by recrystallization with charcoal from ethanol (isatin was specifically recrystallized from ethanol). Conversely, bisphenols were used as received, and several other reagents (including 1,6-dibromohexane (99.5%), isatin (98%), N,N-dimethylformamide (DMF, 99.5%), potassium carbonate (K<sub>2</sub>CO<sub>3</sub>, 99%), dimethyl sulfoxide (DMSO, 99%), and N-methyl-2-pyrrolidone (NMP, 99%)) were used as purchased without further purification.

## 2.2. Synthesis

Polymers synthesized via polyhydroxyalkylation reactions have demonstrated attractive properties for the separation of commercially relevant gas mixtures. Specifically, the 'B' family of polymers (Figure 1), which incorporates Bisphenol AF into the backbone, has shown great promise.

**Figure 1**

(a) Reaction scheme for the polyhydroxyalkylation of isatin and its derivatives with bisphenol AF; (b) molecular structure of the xanthene-based semiladder polymer bearing phenolic functional groups (2aB).



Polymers were synthesized via one-step polyhydroxyalkylation reactions at room temperature (Table 1), catalyzed by CF<sub>3</sub>SO<sub>3</sub>H (Trifluoromethanesulfonic Anhydride, TFSA) or methanesulfonic acid (MSA).

**Table 1**

*Ratio of quantities for polymers 2aB, 2bB and 2cB*

Polymer	Isatin-based Compound (0.0026 mol)	Bisphenol AF (0.002 mol)	TFSA (mL)	Reaction Time	Precipitation Medium (H <sub>2</sub> O/methanol)
2aB	Isatin (0.3825 g)	0.6724 g	2.5	4 h	50 % v/v
2bB	N-propyl isatin (0.4916 g)	0.6724 g	1.5	19 h	1:3 ratio
2cB	N-bromohexyl isatin (0.8064 g)	0.6724 g	1.5	24 h	50 % v/v

### 2.2.1. Modification

In a typical post-synthetic modification, a solution in NMP was formed by dissolving the polymer (1.0 g) at a concentration of 10% w/v. After that, K<sub>2</sub>CO<sub>3</sub> and specific amounts of quaternary ammonium salt bromides were added. After stirring for 48 h at 60 °C, the resulting polymer was isolated by pouring the mixture into ethyl acetate and the product was washed multiple times with warm ethyl acetate. Degree of substitution was determined by <sup>1</sup>H NMR analysis.

### 2.3. Characterization

Table 2 presents the main analytical techniques used to investigate the structural, thermal, and molecular characteristics of the B family polymers.

**Table 2**

*Summary of characterization techniques*

Technique	Instrumentation/Conditions	Purpose
FTIR	Nicolet iS10 spectrometer equipped with a DTGS detector and ATR accessory	Identification of functional groups and confirmation of chemical structure

**Table 2**  
**Summary of characterization techniques**

Technique	Instrumentation/Conditions	Purpose
NMR	Bruker Avance 400 ( <sup>1</sup> H at 400.13 MHz and <sup>13</sup> C at 100 MHz); CDCl <sub>3</sub> and DMSO-d <sub>6</sub> as solvents	Structural elucidation based on proton and carbon environments
DSC	TA Instruments DSC Q20; nitrogen atmosphere (N <sub>2</sub> ) and a heating rate of 10 °C/min up to 400 °C	Determination of thermal transitions, such as glass transition temperature (T <sub>g</sub> )
TGA	TA Instruments TGA-Q50; N <sub>2</sub> and air atmospheres and a heating rate of 10 °C/min	Assessment of thermal stability and decomposition behavior
GPC	Waters 2414 ALLIANCE system with Styragel HR 4E and HR 5E columns; THF at 35 °C	Determination of molecular weight and molecular weight distribution

### 2.3.1. Gas transport properties

Polymer membranes were formed by solvent-casting technique employing a 5 wt % polymer solution in dimethyl sulfoxide (DMSO), which resulted in 50-60 μm thick films. To estimate gas transport properties, such as permeability, a variable-pressure/constant volume permeation cell was employed, where permeability coefficients for each ultra high purity gas (UHP) H<sub>2</sub>, CH<sub>4</sub> and CO<sub>2</sub> were determined from the slope of the downstream pressure versus time plot at a steady state at 35 °C and 2 atm. Permeability coefficients for each gas were estimated from the slope of the downstream pressure versus time plot once the steady state. Density (δ) were determined at 30 °C employing a density gradient column and a previously degassed zinc chloride solution (ZnCl<sub>2</sub>) and the measured data were then used to calculate fractional free volume (FFV) employing the van der Waals volume with the group contribution method proposed by Van Krevelen (Van Krevelen & Te Nijenhuis, 2009).

Diffusion ( $D_i$ ), solubility ( $S_i$ ), selectivity ( $S_{i/j}$ ) and permeability ( $P_i$ ) coefficients were derived from the measured time lag ( $\theta_i$ ) following eqs. 1, 2 and 3, where  $i$  and  $j$  are the gas pairs, and  $L$  the polymer film thickness.

$$D_i = \frac{L^2}{6\theta_i} \quad \dots (1)$$

$$S_i = \frac{P_i}{D_i} \quad \dots (2)$$

$$S_{i/j} = \frac{P_i}{P_j} \quad \dots (3)$$

The generation of the gas mixture is achieved via the Reverse Water-Gas Shift (RWGS) reaction, produced in a stainless-steel tubular reactor charged with a nickel oxide (NiO) catalyst. Following the catalytic process, the constituent gases are separated by means of a polymeric membrane. The feed, consisting of a CO and H<sub>2</sub> mixture in a 1:1 molar ratio, is supplied to the reactor under isothermal conditions at 250 °C and a pressure of 1 atm. Qualitative and quantitative identification of the resulting chemical species is performed using gas chromatography (GC).

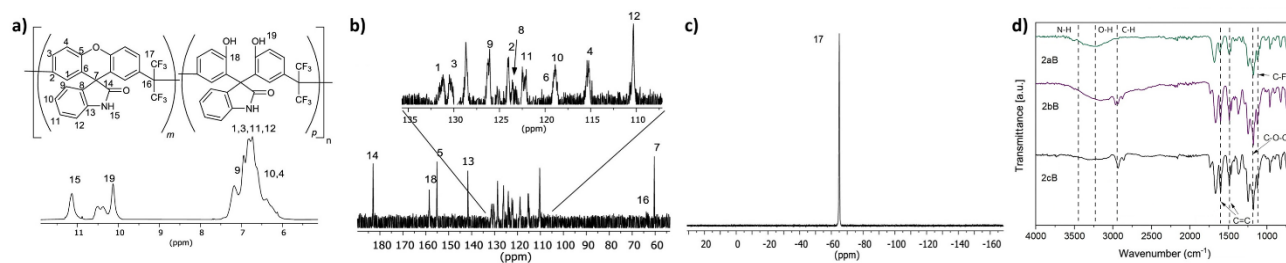
## 3. RESULTS

### 3.1. Polymer characterization

The incorporation of bisphenol AF introduces CF<sub>3</sub> groups into the polymer backbone, leading to enhanced solubility, improved processability, and increased glass transition and thermal stability (Olvera y otros, 2013) compared to analogues based on 4,4'-biphenol. Due to strong electron-withdrawing nature of the CF<sub>3</sub> groups, higher acidity (TFSA) and nonstoichiometric conditions were required, yielding polymers with moderate molecular weights. On the other hand, spectroscopic analyses (FTIR, <sup>1</sup>H/<sup>13</sup>C/<sup>19</sup>F NMR) confirmed the incorporation of CF<sub>3</sub> moieties and indicated the formation of semiladder copolymers containing xanthene units and phenolic groups (Figure 2), probably generated by partial cyclodehydration.

**Figure 2**

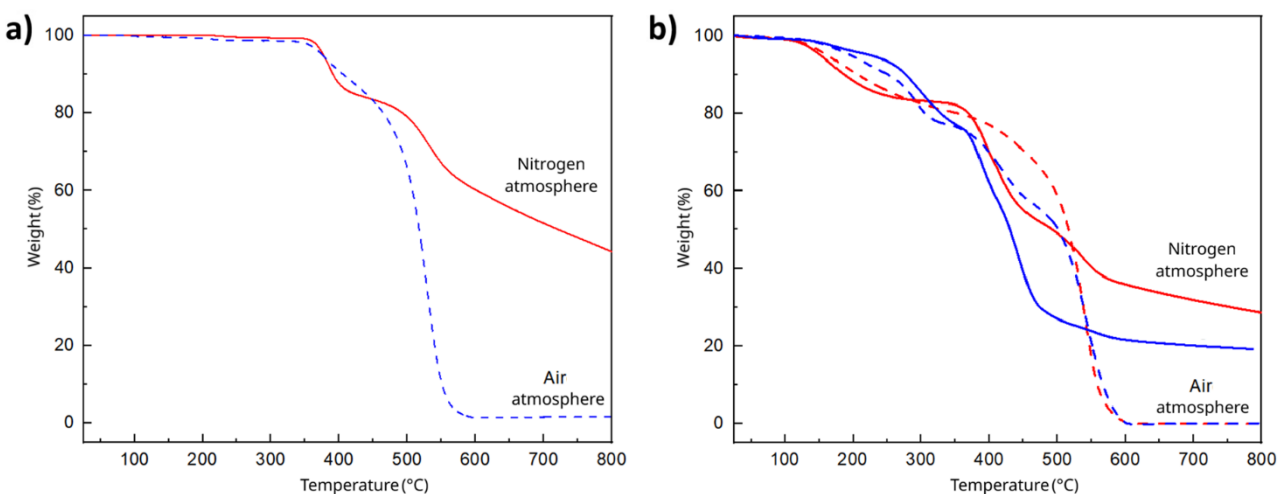
a) <sup>1</sup>H, b) <sup>13</sup>C, c) <sup>19</sup>F NMR and d) FTIR spectra of “B” polymer family



Thermogravimetric analysis showed good thermal stability, with decomposition temperatures above 360 °C for polymer 2aB (Figure 3), while polymers derived from N-propyl and N-bromohexyl isatin exhibited reduced stability, particularly in the presence of the bromohexyl group. Overall, polymers of family B combine high solubility, thermal robustness, and structural features favorable for membrane fabrication and gas separation applications. Differential scanning calorimetry analysis (DSC), however, indicates that no glass transition temperature was detected for these materials.

**Figure 3**

a) Thermal analysis of 2aB polymer, b) 2bB (red lines) and 2cB (blue lines) in N<sub>2</sub> and air atmosphere



### 3.2. Gas transport properties

2(a–c)B membrane family exhibits gas permeabilities measured at 35 °C and 2 atm following the order  $H_2 > CO_2 > CH_4$ , indicating that size-sieving is the dominant gas separation mechanism (Soto et al., 2022) (Yu and Zhang, 2021). Incorporation of the N-propyl isatin group in membrane 2bB leads to an overall increase in gas permeability and solubility, reflecting the typical permeability–selectivity trade-off described by Robeson. On the other hand, polymer 2bB showed improved selectivity for the O<sub>2</sub>/N<sub>2</sub>, CO<sub>2</sub>/N<sub>2</sub>, and CO<sub>2</sub>/CH<sub>4</sub> gas pairs compared to 2aB sample. In contrast, membrane 2cB (containing the N-bromohexyl isatin lateral group), exhibited a pronounced decrease in selectivity, with reductions of up to ~80% for H<sub>2</sub>/CH<sub>4</sub> and CO<sub>2</sub>/CH<sub>4</sub>, attributed to the permeability of larger kinetic diameter gases. While FFV decreases as the pendant group size increases, this does not translate into a direct correlation with gas permeability. Notably, membrane 2bB exhibited the lowest FFV of the entire series,

however, it showed the highest gas permeability. This behavior is attributed to the combined effects of increased chain stiffness, efficient interchain packing and the presence of fluorinated groups.

**Table 3**

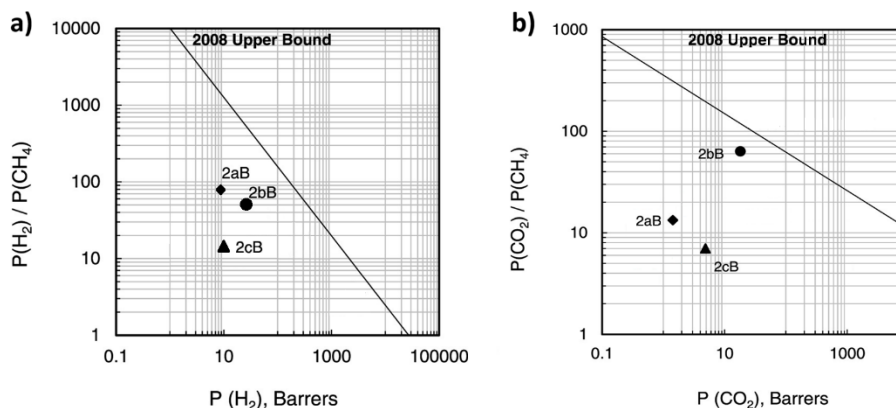
*Results of characterization and gas transport study*

Polymer	Molecular weight ( $M_w/M_n \times 10^{-4}$ g/mol)	PDI	Permeability ( $P_i$ ) [Barrer] <sup>a</sup>			Selectivity ( $S_{i/j}$ )		FFV
			$H_2$	$CH_4$	$CO_2$	$H_2/CH_4$	$CO_2/CH_4$	
2aB	9.5 / 2.8	3.3	8.4	0.11	1.43	78	13	1.43
2bB	11.6 / 5.8	1.9	26	0.52	10.3	51	20	10.3
2cB	4.1 / 2.5	1.6	10.1	0.7	4.9	14	7	4.9

Note. <sup>a</sup> 1 Barrer =  $1 \times 10^{-10}$  cm<sup>3</sup> STP m/(cm<sup>2</sup> s cm Hg)

**Figure 4**

*Results of characterization and gas transport study*

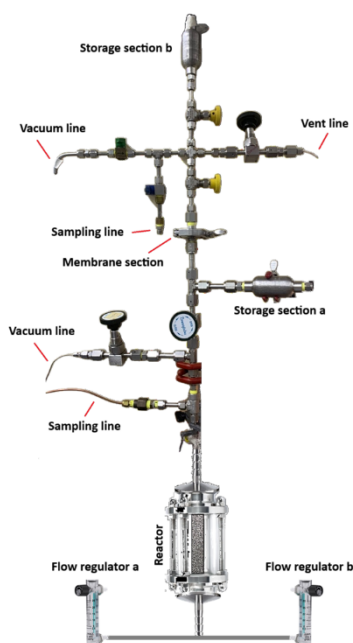


### 3.3. Reactor

A custom engineered prototype was developed for the gas generation stage to produce a mixture required as feed for the separation process using B-series polymers, which consists of a stainless-steel tubular reactor integrated with a gas separation section (Figure 5). Reactor was supplied with a 1:1 molar feed of CO<sub>2</sub> and H<sub>2</sub> and maintained at 250 °C to facilitate the RWGS reaction, where NiO catalyst was evaluated and resulted in an effluent containing CO, CO<sub>2</sub>, H<sub>2</sub>, and CH<sub>4</sub> as confirmed by gas chromatography.

**Figure 5**

*Reactor system coupled with separation stage*



## 4. CONCLUSION

In summary, a series of semiladder polymers containing xanthene and phenolic moieties were successfully synthesized, where the integration of fluorinated groups yielded membranes with exceptional thermal and chemical stability. These polymers showed favorable gas transport properties, where the introduction of CF<sub>3</sub> group enhanced both permeability and selectivity. Notably, polymer 2bB exhibited an outstanding balance between permeability and selectivity, particularly for the H<sub>2</sub>/CH<sub>4</sub> and CO<sub>2</sub>/CH<sub>4</sub> gas pairs. Owing to these promising separation factors, B-series polymers were selected for integration into the separation phase of the prototype, with subsequent evaluation planned for a second stage of the process, therefore, the system allows the evaluation of these membranes under operational conditions, specifically to determine the separation efficiency for the multi-component mixture (CO, CO<sub>2</sub>, H<sub>2</sub>, and CH<sub>4</sub>) generated by the catalytic reactor.

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