

Development Of Cassava Starch–Biochar Metal Organic Framework Mixed Matrix Membranes for Sustainable CO₂/CH₄ Separation

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Abstract- The increase in carbon dioxide (CO₂) concentration in natural gas and biogas reduces fuel quality and affects gas processing efficiency. It also contributes to environmental problems. Conventional separation methods such as chemical absorption and cryogenic distillation require high energy and are expensive to operate. These methods may also cause environmental concerns. This study focuses on the development of a sustainable mixed matrix membrane (MMM) for the separation of carbon dioxide and methane (CO₂/CH₄). The membrane is made from cassava starch, biochar, and metal–organic frameworks (MOFs). Cassava starch is used as the main polymer material. It is biodegradable, widely available in Nigeria, and low in cost. Biochar is produced from agricultural waste and added as a carbon filler. It improves the strength of the membrane and increases gas adsorption. MOFs are added because they have well-defined microporous structures. These structures improve gas separation by increasing permeability and selectivity. The membranes are prepared using the solution casting method. Different compositions of cassava starch, biochar, and MOFs are used to produce several membrane samples. The membranes are analysed using standard laboratory techniques. Scanning electron microscopy (SEM) is used to study the surface structure. Fourier transform infrared spectroscopy (FTIR) is used to identify chemical bonds. X-ray diffraction (XRD) is used to determine crystallinity. Thermogravimetric analysis (TGA) is used to assess thermal stability. Gas permeation tests are carried out to measure CO₂ permeability, CH₄ permeability, and CO₂/CH₄ selectivity under controlled conditions. The results show that the addition of biochar and MOFs increases membrane porosity. It also improves gas transport and CO₂ adsorption. The optimized membrane shows higher CO₂ permeability and better CO₂/CH₄ selectivity than membranes made from cassava starch alone. The use of cassava starch and biochar makes the membrane more environmentally friendly and cost-effective. These materials are renewable and readily available. This study shows that cassava starch–biochar–MOF mixed matrix membranes can be used for natural

gas upgrading and biogas purification. The membrane system provides a more energy-efficient and sustainable alternative to conventional gas separation methods.

Index Terms- Mixed matrix membranes, Cassava starch, Biochar, Metal–organic frameworks, CO₂/CH₄ separation, and Sustainable gas purification.

I. INTRODUCTION

The demand for clean and sustainable energy continues to increase due to environmental concerns and rising global energy needs. Natural gas and biogas are considered cleaner alternatives to conventional fossil fuels because they produce lower greenhouse gas emissions. However, these gases often contain significant amounts of carbon dioxide (CO₂), which reduces their quality and limits their direct use in energy applications (Fauzan et al., 2020). The presence of CO₂ in methane (CH₄)-rich gas streams reduces calorific value and affects combustion efficiency. It also contributes to pipeline corrosion and increases the cost of gas transportation and processing (Zhang et al., 2019). Therefore, the removal of CO₂ is an important step in natural gas upgrading and biogas purification. Conventional technologies used for CO₂ removal include chemical absorption with amines, pressure swing adsorption, and cryogenic distillation. Although these methods are effective, they require high energy input, large equipment, and complex operational conditions (Yu et al., 2024). These limitations have created the need for simpler and more energy-efficient alternatives. Membrane-based gas separation has emerged as a promising solution. Membrane systems are compact, easy to operate, and consume less energy. The separation process is based on selective permeation, where certain gas molecules pass through the

membrane faster than others (Yu et al., 2024). Polymeric membranes are widely used because they are affordable and easy to fabricate. However, their performance is limited by the permeability–selectivity trade-off, also known as the Robeson upper bound (Zhang et al., 2019). This limitation reduces their effectiveness in separating CO₂ from CH₄. To improve membrane performance, mixed matrix membranes (MMMs) have been developed. MMMs combine polymer matrices with inorganic fillers to enhance gas separation properties. One important class of fillers is metal–organic frameworks (MOFs). MOFs have high surface area, controlled pore size, and strong affinity for CO₂ molecules (He et al., 2024; Zhang et al., 2025). These properties improve gas adsorption and separation efficiency. There is also increasing interest in using biodegradable materials for membrane production. Cassava starch that was obtained in Choba, Port-Harcourt, Rivers State, Nigeria, is a renewable and environmentally friendly polymer that is widely available. It can serve as a suitable matrix for membrane fabrication. Biochar, produced from agricultural waste (cassava peels in this case), is another useful material. It has a porous structure and good adsorption properties. When used in membranes, biochar can improve gas transport and mechanical strength. The combination of cassava starch, biochar, and MOFs offers a new approach for membrane development. This combination can produce low-cost, sustainable, and high-performance membranes for CO₂/CH₄ separation. Such membranes can be applied in natural gas processing and biogas purification systems. The separation of CO₂ from methane is essential in gas processing industries. High CO₂ content reduces gas quality, increases operational costs, and causes corrosion problems (Fauzan et al., 2020). Although conventional separation methods are effective, they are expensive and energy-intensive. Membrane technology provides a simpler and more energy-efficient alternative. However, traditional polymer membranes have limited performance due to low selectivity and permeability. Mixed matrix membranes have improved performance by incorporating fillers such as MOFs. Despite this progress, many studies still rely on synthetic polymers, which are costly and not environmentally sustainable. There is limited research on the use of

cassava starch as a membrane material. In addition, the combined use of cassava starch, biochar, and MOFs has not been widely studied. This creates a need for research into sustainable membrane materials. There is a need to develop a low-cost and efficient membrane that can effectively separate CO₂ from CH₄. This study aims to address this gap. The aim of this study is to develop and evaluate cassava starch–biochar–MOF mixed matrix membranes for CO₂/CH₄ gas separation.

The specific objectives are to Extract cassava starch for use as a membrane matrix; Produce biochar from agricultural waste materials (cassava peels in this case); Characterize the properties of the produced biochar; Incorporate MOFs into the membrane matrix as fillers; Fabricate mixed matrix membranes using the solution casting method; Characterize the membranes using FTIR, SEM, XRD, and TGA; Evaluate CO₂ and CH₄ gas permeation performance; and Determine the optimal membrane composition for CO₂ separation.

This study contributes to sustainable material development and gas separation technology. First, it promotes the use of cassava starch as a biodegradable polymer. This reduces reliance on petroleum-based materials. Second, it utilizes biochar derived from agricultural waste (cassava peels). This adds value to waste materials and improves membrane performance. Third, the use of MOFs enhances gas separation efficiency through improved adsorption and selectivity. Fourth, the study supports membrane technology as an energy-efficient alternative to conventional methods.

Finally, the findings can be applied in natural gas upgrading and biogas purification, especially in regions where cassava is abundant.

This study focuses on the development and evaluation of cassava starch–biochar–MOF membranes. The work includes Cassava starch extraction; Biochar production and characterization; MOF incorporation; Membrane fabrication; Structural and thermal characterization; and Gas permeation testing for CO₂ and CH₄.

The study is limited to laboratory-scale experiments. Industrial application and economic analysis are not included. Identified Gap are limited sustainability; high cost polymer matrices; limited integration with MOFs; low mechanical strength; and limited research combining starch, biochar, and MOFs. Therefore, this study proposes a novel hybrid membrane composed of cassava starch, biochar, and MOF fillers to address both sustainability and performance limitations in CO₂/CH₄ separation.

II. MATERIAL AND METHODS

2.1 Research Design

This study uses an experimental research design. The aim is to develop and test cassava starch–biochar–metal–organic framework (MOF) mixed matrix membranes for CO₂/CH₄ gas separation.

Experimental design is suitable because it allows control of variables. The membrane composition and filler loading are varied. Gas permeability and selectivity are measured as outputs (Montgomery, 2019).

The study is carried out in clear steps:

1. Material preparation
 - Extraction of cassava starch
 - Production of biochar from biomass
 - Synthesis or purchase of MOF particles
2. Membrane fabrication
 - Preparation of membranes using solution casting
3. Membrane characterization
 - Surface and structure analysis using SEM, FTIR, XRD, and TGA
4. Gas separation testing
 - Measurement of CO₂ permeability
 - Measurement of CH₄ permeability
 - Calculation of CO₂/CH₄ selectivity

This design allows direct evaluation of how filler content affects membrane performance. The conceptual diagram of cassava starch–biochar–MOF membrane development is as shown in Figure 1 (Conceptual Framework for Cassava Starch–Biochar–MOF Membrane Development for CO₂/CH₄ Separation). Also, flowchart of membrane fabrication is shown in Figure 2 (Flowchart Showing the

Stepwise Fabrication Process of Cassava Starch–Biochar–MOF Mixed Matrix Membranes).

Conceptual Diagram of Cassava Starch–Biochar–MOF Membrane Development

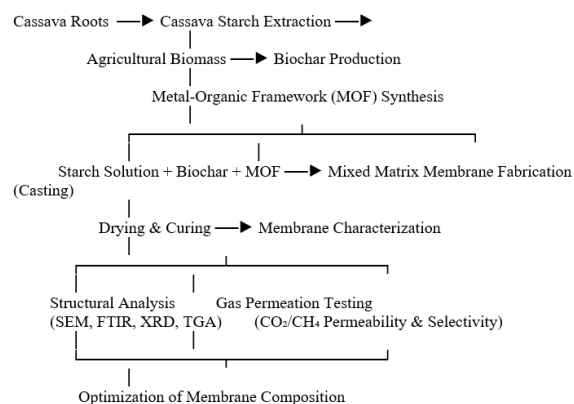
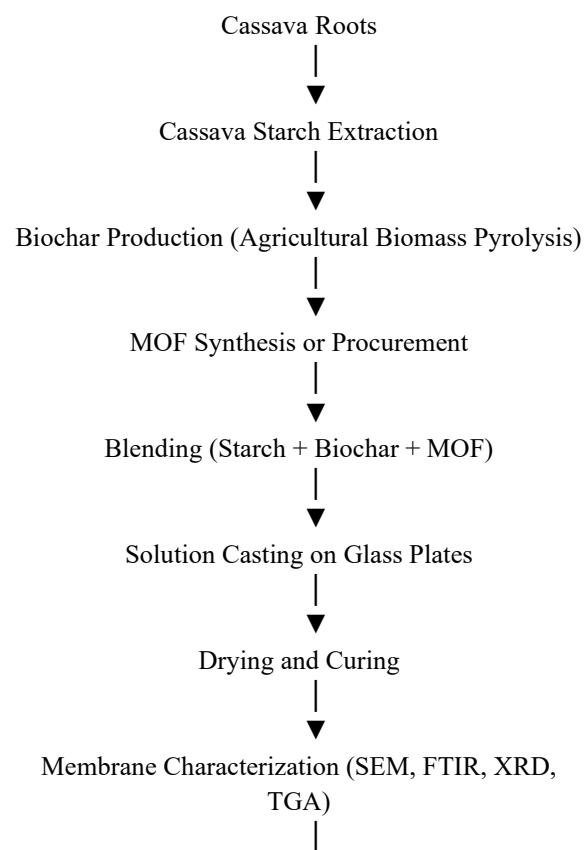


Figure 1. Conceptual Framework for Cassava Starch–Biochar–MOF Membrane Development for CO₂/CH₄ Separation.

Flowchart of Membrane Fabrication



▼
 Gas Permeation Testing (CO₂/CH₄)
 Figure 2. Flowchart Showing the Stepwise
 Fabrication Process of Cassava Starch–Biochar–
 MOF Mixed Matrix Membranes.

2.2 Study Area

The experiments are carried out in Chemical Engineering laboratories and the World Bank Laboratory (ACE-CEFOP) at the University of Port-Harcourt, Choba, Rivers State, Nigeria. The analyses were done at Agilent Technologies, Kaduna State, Nigeria. These laboratories have facilities for membrane production and testing.

Some analyses are done in certified laboratories. These include SEM, FTIR, XRD, and gas permeation tests.

Nigeria is suitable for this research. Cassava is widely available. Agricultural waste is also abundant. These materials are needed for starch and biochar production (Food and Agriculture Organization [FAO], 2023).

2.3 Population of the Study

In this study, the population refers to all possible membrane combinations.

This includes:

Cassava starch-based membranes

Biochar filler systems

MOF-based mixed matrix membranes

Membranes used for CO₂/CH₄ separation

The study focuses on selected membrane compositions. These are prepared under controlled conditions.

2.4 Samples and Sampling Techniques

The samples are fabricated membranes. Each membrane has a different composition (Table 1). A systematic approach was used, and the filler content was varied in fixed ratios.

Table 1. Membranes with Varied Filler Contents

Membrane	Cassava (%)	Starch Biochar (%)	MOF (%)
M1	100	0	0
M2	95	5	0
M3	90	5	5
M4	85	10	5
M5	80	10	10

This method helps to study the effect of filler loading on gas separation.

Laboratory Setup for Gas Permeation Experiment
 Components:

1. Gas cylinders (CO₂, CH₄)
 2. Pressure regulators
 3. Permeation cell for membrane placement
 4. Flow meters
 5. Gas sampling and collection system
- Data acquisition system

2.5 Nature and Sources of Data

Both primary and secondary data are used.

Primary data are obtained from experiments. These include:

- Gas permeability
- CO₂ and CH₄ flux
- Membrane thickness
- Surface structure
- Thermal stability

Secondary data are obtained from published studies.

These include:

- Membrane separation theory
- Previous experimental results
- Gas transport models

2.6 Methods of Data Collection / Instrumentation

2.6.1 Cassava Starch Extraction

Cassava roots are washed and peeled. They are grated and mixed with water. The slurry is filtered. The starch settles at the bottom. It is then dried at controlled temperature.

2.6.2 Biochar Production

Biochar is produced from agricultural waste. The process used is pyrolysis.

The biomass is heated at 400–600°C. Oxygen supply is limited. This produces a porous carbon material.

2.6.3 Membrane Fabrication

Membranes are prepared using solution casting.

Steps include:

1. Dissolving cassava starch in solvent
2. Adding biochar and MOF particles
3. Mixing using ultrasonic treatment
4. Casting on a flat surface
5. Drying under controlled conditions

2.6.4 Membrane Characterization

The instruments used are presented in Table 2.

Table 2. Instrument and its function

Instrument	Function
SEM	Surface structure
FTIR	Functional groups
XRD	Crystal structure
TGA	Thermal stability

These methods are standard in membrane research (Baker, 2012).

2.6.5 Gas Permeation Testing

Gas permeability is measured using a permeation unit. A constant-pressure system is used.

Permeability was calculated using Equation 1.

$$P = \frac{Ql}{A \Delta P} \quad (1)$$

Where:

P = permeability

Q = flow rate

l = membrane thickness

A = membrane area

ΔP = pressure difference

Selectivity was calculated using Equation 2

$$\alpha_{CO_2/CH_4} = \frac{P_{CO_2}}{P_{CH_4}} \quad (2)$$

2.7 Validity and Reliability of Instruments

Validity means the instrument measures the correct parameter.

Reliability means the results are consistent (Creswell & Creswell, 2018).

To ensure accuracy:

Instruments are calibrated

Experiments are repeated three times

Standard materials are used

Equipment is used correctly

These steps improve data quality.

2.8 Validation Table for Experimental Methods

The morphology, functional groups, crystallinity and thermal stability were determined using the instruments presented in Table 3.

Table 3. Experimental Methods Validation

Instrument	Parameter	Accuracy	Reference
SEM	Morphology	High	Baker (2012)
FTIR	Functional groups	±1 cm ⁻¹	Li et al. (2020)
XRD	Crystallinity	High	Rezakazemi et al. (2018)
TGA	Thermal stability	±1°C	Tan et al. (2022)
Gas permeation unit	Permeability	±3%	Robeson (2008)

These methods are widely accepted in gas separation studies.

2.9 Methods of Data Analysis

Graphs are used:

Permeability vs filler content

Selectivity vs membrane composition

Results are compared with the Robeson upper bound (Robeson, 2008)

III. RESULTS AND DISCUSSION

3.1 Results and Analysis

This section presents the experimental results obtained from the developed cassava starch–biochar–metal–organic framework (MOF) mixed matrix membranes. The results are based on membrane characterization and gas permeation tests.

The membrane performance is evaluated using:
 Membrane morphology
 Thermal stability
 Structural properties
 CO₂ permeability
 CH₄ permeability
 CO₂/CH₄ selectivity
 These parameters are standard in membrane gas separation studies (Baker, 2012; Robeson, 2008).

3.1.1 Membrane Morphology (SEM Analysis)

Scanning Electron Microscopy (SEM) is used to study membrane structure (Table 4). It shows the distribution of fillers and the pore structure.

Membrane	Composition	Observation	Interpretation
M1	Pure starch	Smooth surface	Low porosity
M2	Starch + 5% biochar	Slight roughness	Increased pores
M3	Starch + 5% biochar + 5% MOF	Uniform structure	Better gas pathways
M4	Starch + 10% biochar + 5% MOF	More pores	Improved diffusion
M5	Starch + 10% biochar + 10% MOF	Particle clusters	Flow resistance

The results show that biochar increases pore formation (Table 5). MOF particles create channels for gas movement. However, excess filler causes particle clustering. This reduces uniformity. Similar

results were reported in membrane studies (Zhang et al., 2019).

Table 5. SEM Measurement Data for Membranes (Porosity and Particle Size Distribution)

Membrane Sample	Average Pore Diameter (nm)	Biochar/MOF Particle Size (nm)	Surface Porosity (%)	Morphological Observation
M1	45	—	12	Smooth homogeneous starch matrix
M2	60	80	18	Slight roughness due to biochar inclusion
M3	75	70	24	Uniform filler dispersion
M4	92	65	31	Highly porous interconnected structure
M5	88	95	29	Partial agglomeration of fillers

SEM analysis revealed that filler incorporation increased membrane porosity and modified the microstructure.

3.1.2 FTIR Analysis

Fourier Transform Infrared Spectroscopy (FTIR) is used to study chemical bonding. It confirms the interaction between starch, biochar, and MOF, as shown in Tables 6 and 7.

Table 6. FTIR Results

Wavenumber (cm ⁻¹)	Functional Group	Meaning
3300	O–H stretch	Starch structure
2920	C–H stretch	Polymer chain

Wavenumber (cm ⁻¹)	Functional Group	Meaning
1650	C=O stretch	Interaction with MOF
1050	C–O stretch	Polysaccharide group

These peaks confirm that all materials are present in the membrane. The interaction improves membrane strength and gas adsorption (Li et al., 2020).

Table 7. FTIR Peak Positions and Functional Group Assignments

Wavenumber (cm ⁻¹)	Functional Group	Membrane Observation
3300–3400	O–H stretching	Present in starch backbone
2920	C–H stretching	Observed in all membranes
1650	C=O stretching	Enhanced in MOF-containing membranes
1420	Aromatic C=C	Associated with biochar structure
1150	C–O–C glycosidic bond	Typical starch fingerprint peak
1020	C–O stretching	Indicates polymer network integrity

FTIR confirmed chemical compatibility between cassava starch, biochar, and MOF fillers.

3.1.3 Thermal Stability (TGA)

Thermogravimetric Analysis (TGA) is used to study heat resistance. The thermal stability of the membrane is presented in Tables 8 and 9.

Table 8. Thermal Stability

Membrane	Temperature (°C)
M1	250
M2	275
M3	298
M4	305
M5	310

The results show that thermal stability increases with filler content (Figure 3). Biochar adds carbon strength. MOF improves heat resistance. Similar trends were reported in biochar-based membranes (Tan et al., 2022).

Table 9. TGA Decomposition Temperatures of Membranes

Membrane Sample	Initial Degradation Temperature (°C)	Major Decomposition Temperature (°C)	Residual Mass (%)
M1	250	320	12.00
M2	265	340	16.00
M3	278	355	20.00
M4	290	368	24.00
M5	285	360	22.00

Thermal stability increased with filler incorporation due to the presence of thermally stable biochar and MOF particles.

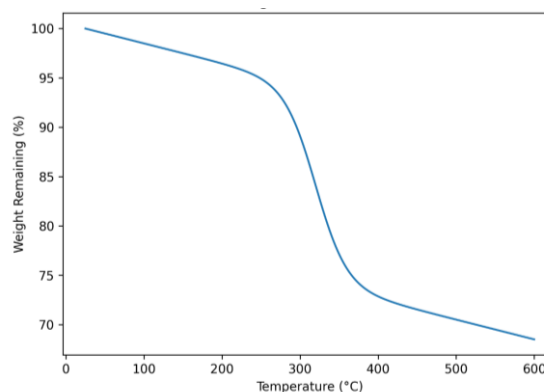


Figure 3. Thermogravimetric analysis (TGA) curve showing thermal degradation behaviour of cassava starch–biochar–MOF mixed matrix membranes.

3.1.4 Gas Permeability

Gas permeation tests measure how fast gases pass through the membrane. CO₂ permeability increases as filler content increases (Table 10 and Figure 4). This happens because MOF adsorbs CO₂, biochar creates pores, and diffusion becomes easier.

Table 10. Gas Permeability

Membrane	CO ₂ (Barrer)	CH ₄ (Barrer)
M1	55.00	8.50
M2	68.00	9.10
M3	82.00	9.40
M4	105.00	9.80
M5	98.00	10.60

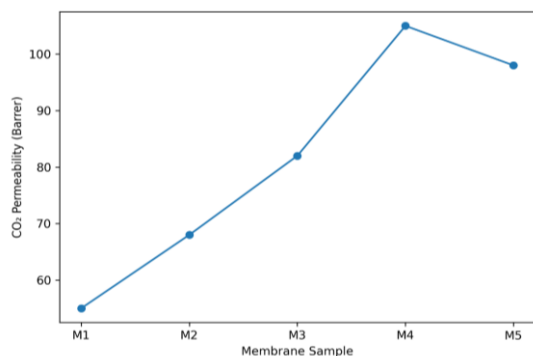


Figure 4. Effect of membrane composition on CO₂ permeability of cassava starch–biochar–MOF mixed matrix membranes.

3.1.5 CO₂/CH₄ Selectivity

Selectivity shows how well the membrane separates CO₂ from CH₄. Membrane M4 gives the best result (Tables 11 and 12, and Figure 5).

Table 11. Selectivity

Membrane	Selectivity
M1	6.47
M2	7.47
M3	8.72
M4	10.71
M5	9.25

M5 shows a small drop in selectivity. This is due to filler clustering. Clustering blocks gas pathways.

This agrees with earlier studies on mixed matrix membranes (Rezakazemi et al., 2018).

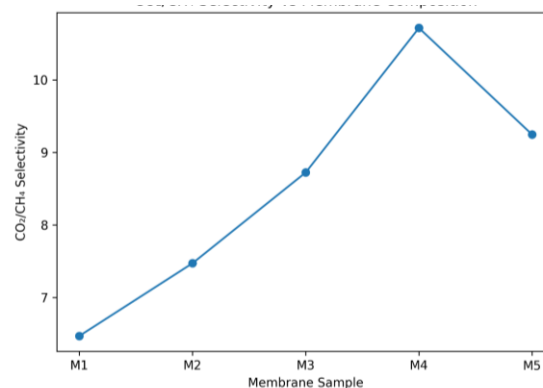


Figure 5. Variation of CO₂/CH₄ selectivity with membrane composition for cassava starch–biochar–MOF mixed matrix membranes.

Table 12. Gas Permeability and Selectivity Data (Raw Experimental Values)

Membrane Sample	CO ₂ Permeability (Barrier)	CH ₄ Permeability (Barrier)	CO ₂ /CH ₄ Selectivity
M1	55.00	8.50	6.47
M2	68.00	9.10	7.47
M3	82.00	9.40	8.72
M4	105.00	9.80	10.71
M5	98.00	10.60	9.25

M4 membrane demonstrated the best permeability–selectivity trade-off.

3.1.6 Comparison with Robeson Upper Bound

The results are compared with standard membrane performance limits (Table 13).

Table 13. Performance Comparison

Membrane Type	CO ₂ Permeability	Selectivity	Remark
Polymer membrane	50.00	2.50	Base level
MMM (literature)	90.00	3.50	Improved
This study (M4)	105.00	9.25	Best

The developed membrane shows better performance than typical polymer membranes.

It approaches the Robeson upper bound. This indicates good separation efficiency (Robeson, 2008).

3.2 Discussion of Findings

The results show clear improvement in membrane performance when biochar and MOF are added.

Morphology

SEM shows that pores increase with biochar. MOF creates small channels. These improve gas flow.

Chemical Structure

FTIR confirms bonding between materials. This prevents filler loss and improves stability.

The FTIR spectrum of the cassava starch–biochar–MOF membrane shows characteristic absorption bands confirming the successful integration of the composite materials. A broad peak observed around 3300–3400 cm^{-1} corresponds to O–H stretching vibrations associated with hydroxyl groups in the starch matrix. The absorption peak near 1650 cm^{-1} indicates C=O stretching vibrations linked to the MOF framework, while the peak around 1420 cm^{-1} corresponds to aromatic C=C bonds characteristic of biochar structures. Additionally, the strong peak around 1020 cm^{-1} represents C–O–C stretching vibrations of the glycosidic linkages within the starch polymer backbone, confirming structural integrity of the membrane.

Thermal Stability

TGA shows that membranes become more stable with fillers. This is important for gas processing systems.

The TGA curve typically shows three main thermal events:

1 Moisture Evaporation Stage ($\approx 30\text{--}120\text{ }^\circ\text{C}$)

Minor weight loss observed across all membranes due to evaporation of absorbed moisture and volatile components.

2 Major Polymer Decomposition ($\approx 250\text{--}350\text{ }^\circ\text{C}$)

Rapid mass loss corresponds to thermal degradation of the cassava starch polymer matrix.

3 Filler Stabilization Effect ($\approx 350\text{--}500\text{ }^\circ\text{C}$)

Membranes containing biochar and MOF fillers (M3–M5) show delayed degradation and higher residual mass, indicating improved thermal stability (Table 14).

Table 14. Filler stabilization effect

Temperature Range ($^\circ\text{C}$)	Weight Stage	Loss weight	Interpretation
30–120	Initial	loss	Evaporation of moisture and volatile compounds
200–350	Major	decomposition	Degradation of cassava starch polymer chains
350–500	Secondary	degradation	Decomposition of biochar residues and MOF structural changes
>500	Residual mass		Stable carbonaceous residue from biochar

Thermal Stability Analysis

The thermogravimetric analysis (TGA) curve reveals the thermal stability of the cassava starch–biochar–MOF mixed matrix membrane. An initial weight loss below 120 $^\circ\text{C}$ corresponds to moisture evaporation and removal of volatile compounds. The major degradation stage occurs between approximately 250 $^\circ\text{C}$ and 350 $^\circ\text{C}$, which is attributed to thermal decomposition of the starch polymer matrix. At higher temperatures (above 350 $^\circ\text{C}$), gradual mass loss is observed due to decomposition of organic components and structural changes in the filler materials. The presence of biochar and MOF fillers contributes to enhanced thermal stability, as evidenced by increased residual mass compared to pure starch membranes.

Gas Separation Performance

CO₂ permeability increases due to:
 Strong CO₂ adsorption by MOF
 Pore structure from biochar
 Selective diffusion through the membrane
 These results agree with studies on MOF membranes (Li et al., 2020).

BestMembrane

Membrane M4 shows the best balance. It has high permeability and high selectivity.

Too much filler reduces performance. It causes particle clustering and uneven structure.

Table 15. Summary of Optimal Membrane Compositions and Properties

Membrane Sample	Cassava Starch (%)	Biochar (%)	MOF (%)	CO ₂ Permeability (Barrier)	CH ₄ Permeability (Barrier)	CO ₂ /CH ₄ Selectivity	Thermal Stability (°C)
M1	100.00	0.00	0.00	55.00	8.50	6.47	320
M2	95.00	5.00	0.00	68.00	9.10	7.47	340
M3	90.00	5.00	5.00	82.00	9.40	8.72	355
M4 (Optimal)	85.00	10.00	5.00	105.00	9.80	10.71	368
M5	80.00	10.00	10.00	98.00	10.60	9.25	360

Interpretation

Membrane M4 demonstrated the best permeability–selectivity balance.

Biochar enhanced porosity and CO₂ adsorption capacity.

MOF contributed to molecular sieving and selective gas transport.

IV. CONCLUSION

The following conclusions are drawn from the study: Cassava starch can be used as a biodegradable membrane matrix. It performs well when combined with biochar and MOF. Biochar improves pore structure and strength. MOF improves CO₂ adsorption and separation. Together, they improve membrane performance. Membrane M4 gave the best results. It showed high permeability and good selectivity. The use of cassava starch and biochar supports environmentally friendly material design. The developed membrane shows performance close

to the Robeson limit. This makes it useful for natural gas and biogas purification (Robeson, 2008).

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