Producing Clean Hydrogen Using a Modular Two-Stage Intensified Membrane-Enhanced Catalytic Gasifier

Topic Area: AOI 1 – Clean Hydrogen Cost Reductions via Process Intensification & Modularization for Hydrogen Shot

Funding Opportunity Announcement (FOA) Number: DE-FOA-0002400:

Clean Hydrogen Production, Storage, transport, and Utilization to Enable a Net Zero Carbon Economy

Award#:DE-FE0032191

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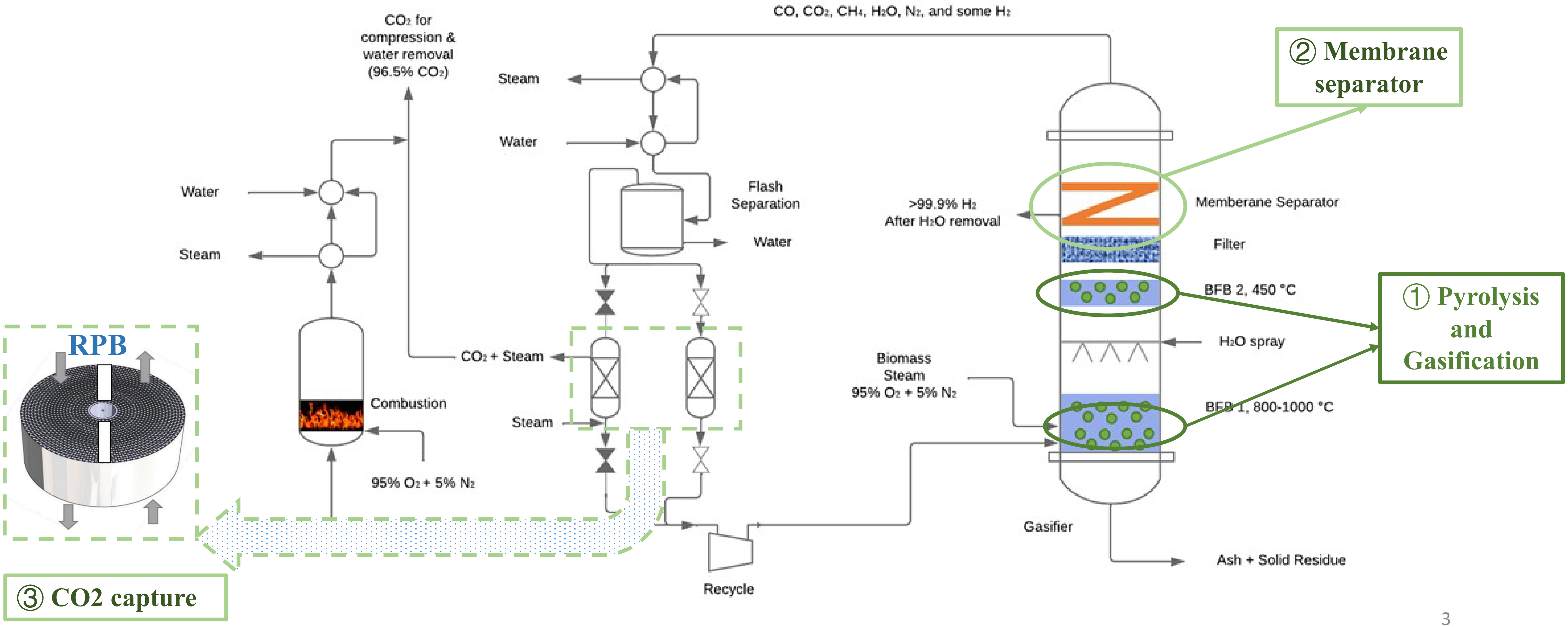
WestVirginiaUniversity.

Backgrounds

- Gasification of biomass is the key enabling technology in this project.
- Most previous works were focused on gasification at large scale and mainly for power production, not for production of ultra-pure H_2 production, using coal in general, along with other feedstocks. There were very few commercial deployments.
- Modular gasification (5-50 MWe) offers advantages due to considerably lower capital investment, distributed deployment, flexible operation using multiple feedstocks, niche applications, etc.
- However, conventional modular gasification suffers due to economy of scale thus making them costlier per unit of product(s) compared to the large-scale deployment.
- Novel intensification and modularization technology can be helpful in improving productivity of these smaller scale gasifiers thus offsetting some of the cost disadvantages. Furthermore, while some of the intensification strategies may not be feasible in large-scale, modular scale deployment can take advantage of that.

Producing Clean Hydrogen Using a Modular Two-Stage Intensified Membrane-Enhanced Catalytic Gasifier



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Producing Clean Hydrogen Using a Modular Two-Stage Intensified Membrane-Enhanced Catalytic Gasifier

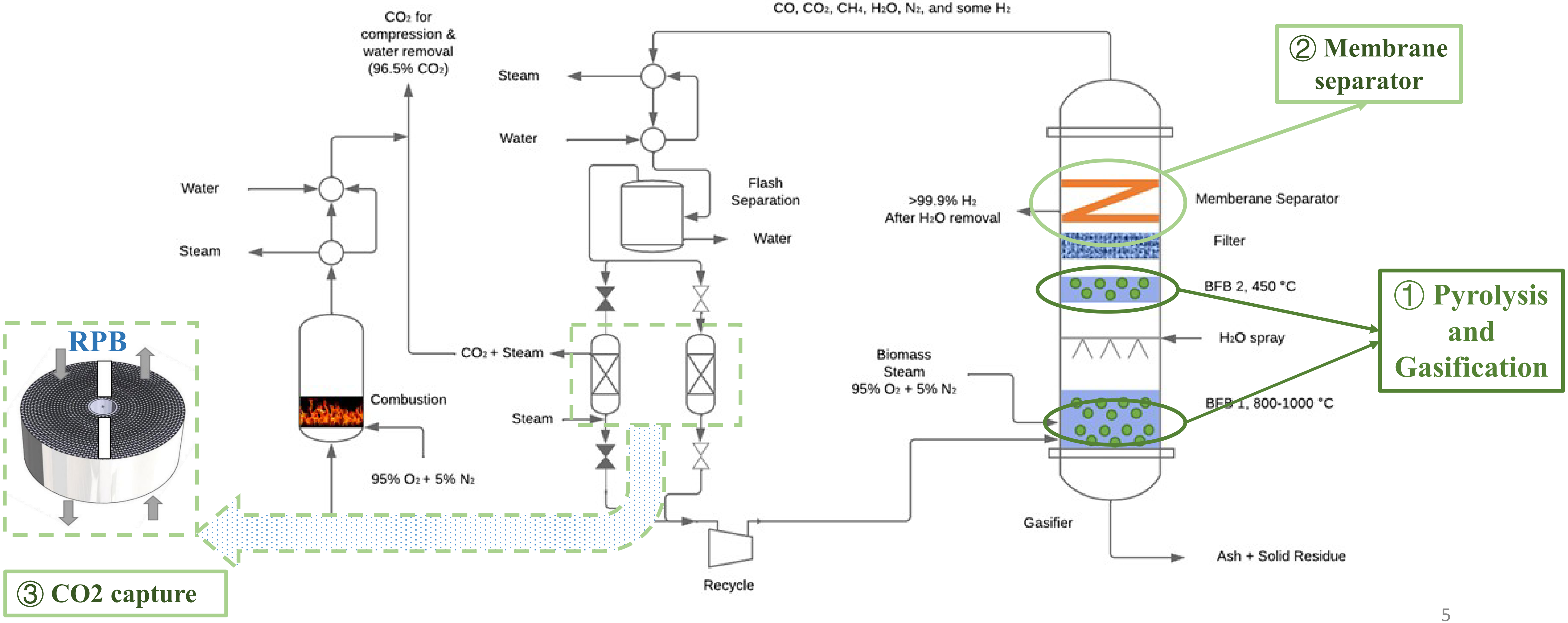
Task 1 Project Management and Planning

Task 2 BFB Biomass Gasifier Design, Integration and Performance Testing

Task 3 Design of High-Temperature Resistant Hydrogen Membrane for Ultra-High Purity H_2 Production

Task 4 Process Modeling, Optimization, and Techno-Economic Analysis

Task 2 BFB Biomass Gasifier Design, Integration and Performance Testing



② Membrane separator

① Pyrolysis and Gasification

③ CO2 capture

Task 2 BFB Biomass Gasifier Design, Integration and Performance Testing

Cold Flow Results

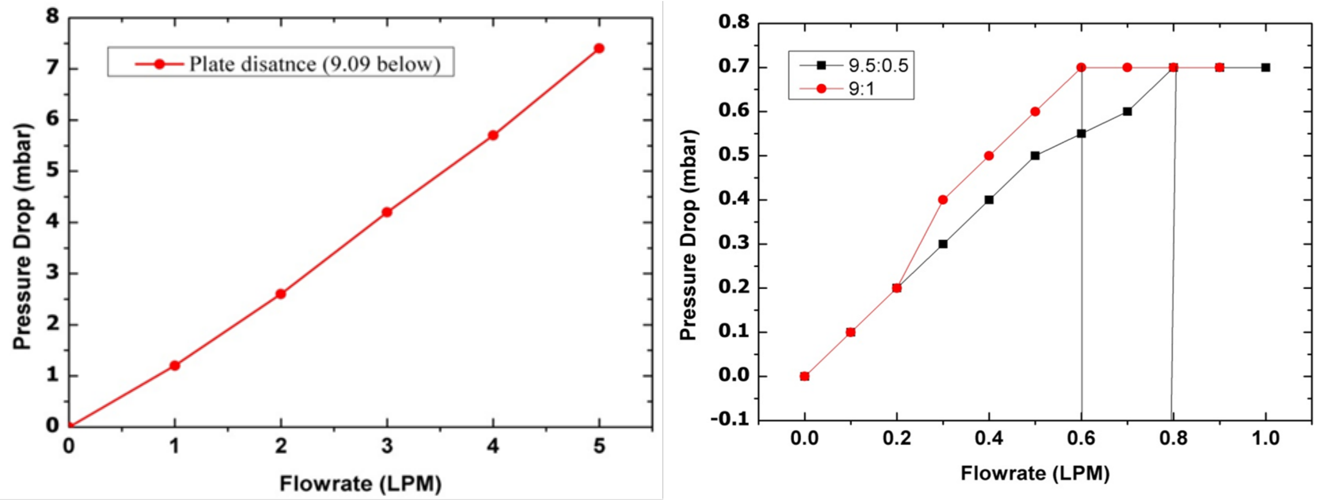


Fig: Pressure Drop Curves in 0.5 inch OD tube
(a) Due to Perforated Plate (b) Biomass to Catalyst Ratio

Key Parameters for cold Flow

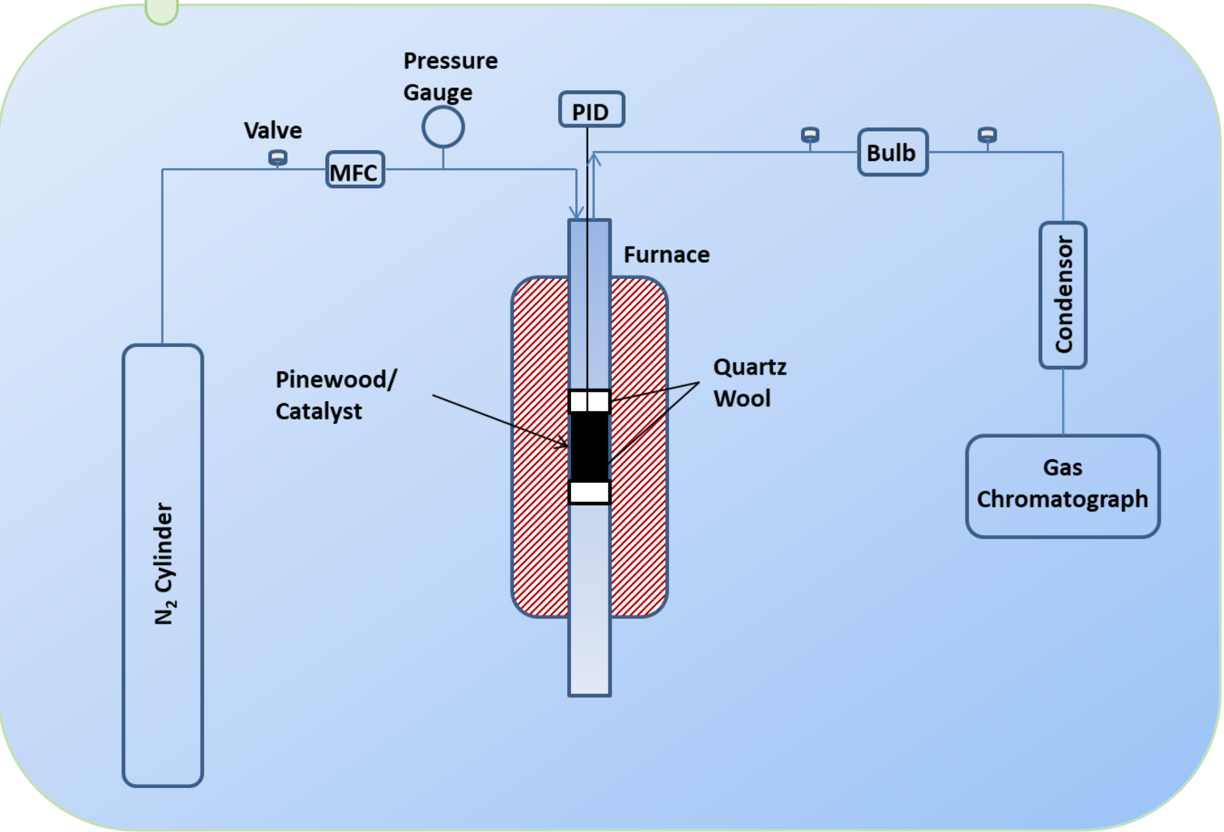
- Size:150-250 micrometers (selection based on Geldart chart)
- Gas to solids ratio,
- Bed length to diameter (L/D) ratio,
- Minimum fluidization velocity
- Impact of gas distributor
- Perforated Plate holes size: 40 µm
- Biomass to Catalyst Ratio

Cold flow data for various tube size

0.5 inch tube [ID=8.5 mm]	**0.75 inch tube [ID=16 mm]**
Pinewood taken: 0.5 gm	Pinewood taken: 1 gm
Initial height: 2.7 cm	Initial height: 2.6 cm
Final height (after fluidization): 6.7 cm	Final height (after fluidization): 7.8 cm
umf=0.65 LPM	umf=1.1 LPM
dP=0.55 mbar	dP=1.2 mbar

Task 2 BFB Biomass Gasifier Design, Integration and Performance Testing

Fixed Bed Reactor Design



Schematic of Fixed Bed Biomass Gasifier

Combustion	$C + O_2 \rightarrow CO_2$ $C + 0.5O_2 \rightarrow CO$
Char Gasification	$C + 2H_2 \rightarrow CH_4$ $C + CO_2 \rightarrow 2CO$ $C + H_2O \rightarrow CO + H_2$
Homogeneous Volatile Reactions	$CH_4 + H_2O \rightarrow CO + 3H_2$ $CO + H_2O \rightarrow CO_2 + H_2$ $CO + 0.5O_2 \rightarrow CO_2$ $CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O$ $H_2 + 0.5O_2 \rightarrow H_2O$
Tar Reactions	$C_xH_y + xH_2O \rightarrow xCO + (0.5y+x)H_2$ $C_xH_y + 0.5xO_2 \rightarrow xCO + 0.5yH_2$ $C_xH_y + CO_2 \rightarrow 2xCO + 0.5yH_2$ $C_xH_y + H_2 \rightarrow xCH_4$ $C_xH_y \rightarrow 0.25yCH_4 + (x-0.25y)C$

Gasification Reactions

Task 2 BFB Biomass Gasifier Design, Integration and Performance Testing

Syngas Production from Lignocellulose Pinewood Data at Low and High Pressure in Fixed Bed

	Pressure (bar)	Temperature (°C)	H_2 (Mole %)	CH_4 (Mole %)	CO (Mole %)	CO_2 (Mole %)	H_2/CO
PW	28	750	27.23	14.47	22.27	33.87	1.22
	32	800	30.55	19.89	15.31	32.88	2.00
	38	850	31.24	20.44	15.93	31.29	1.96
PW/Fe	30	750	31.04	11.22	22.07	34.80	1.41
	34	800	43.07	26.13	7.67	21.54	5.61
	38	850	36.50	21.04	15.74	24.71	2.32
PW/Ni	30	750	20.57	33.25	14.58	30.74	1.41
	34	800	21.59	29.85	18.39	28.49	1.17
	38	850	28.43	14.40	25.83	29.66	1.10
PW	1.01	750	34.83	22.87	18.26	21.49	1.91
	1.01	800	36.23	12.49	24.81	24.51	1.46
	1.01	850	39.24	8.25	28.72	22.20	1.37
PW/Fe	1.01	750	39.67	12.05	24.98	21.67	1.59
	1.01	800	42.32	7.70	39.67	9.76	1.07
	1.01	850	47.19	7.32	34.21	10.28	1.38
PW/Ni	1.01	750	44.88	7.52	29.41	17.07	1.53
	1.01	800	48.10	8.49	29.10	13.28	1.65
	1.01	850	54.99	4.95	30.30	9.10	1.81

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Task 2 BFB Biomass Gasifier Design, Integration and Performance Testing

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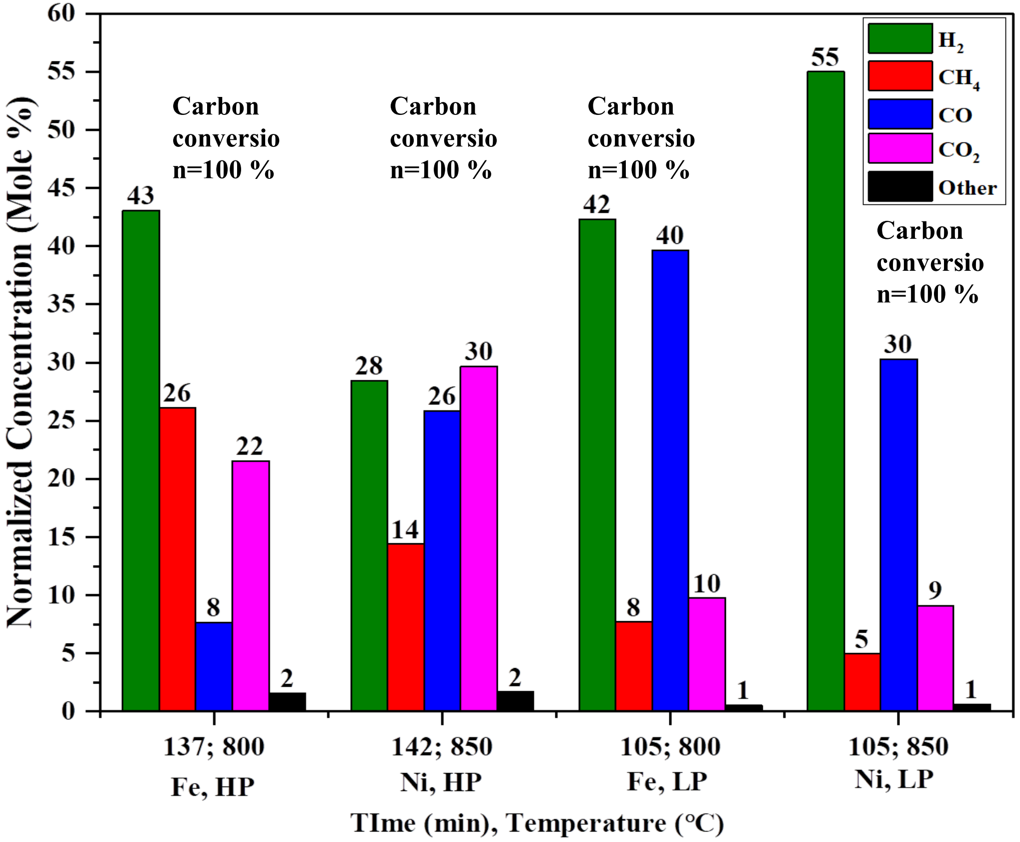
Task 2 BFB Biomass Gasifier Design, Integration and Performance Testing

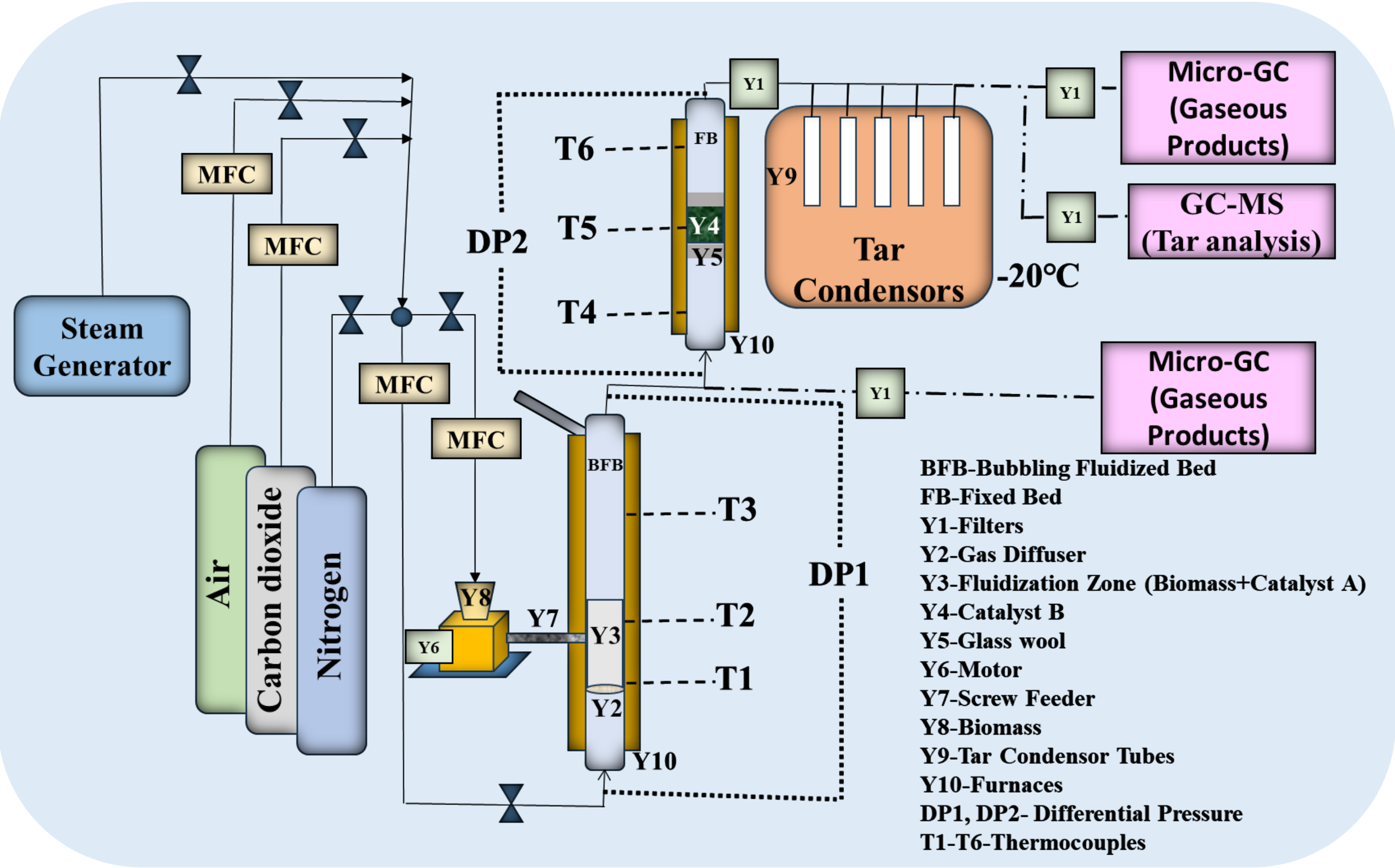
Syngas Yield in Catalytic Gasification of Pinewood in Fixed Bed



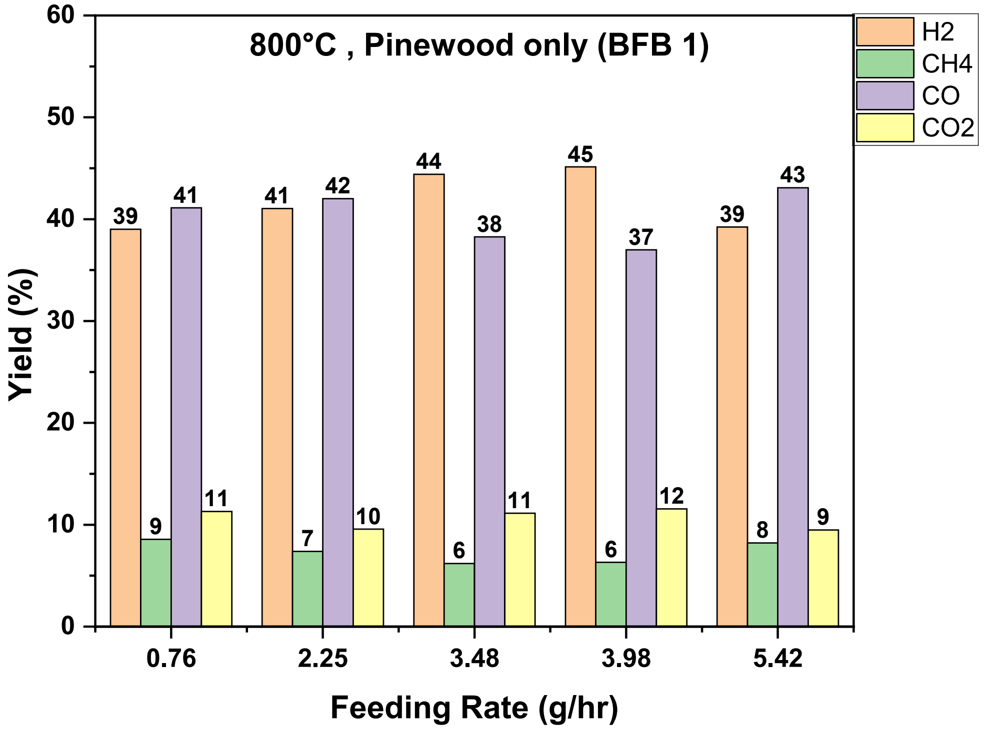
- The major difference in reaction under high and low pressures:
  - (a) High pressure reaction producing syngas with high amount of methane, i.e., high pressure driven the methanation reaction.
  - (b) Low pressure reactions producing syngas with low amount of methane and carbon dioxide.
- In pressurized reaction, non-catalytic gasification gives 99.244 % carbon conversion and catalytic gasification gives nearly 100 % carbon conversion .
- In low-pressure reaction, non-catalytic gasification gives 86.54 % carbon conversion and catalytic gasification gives nearly 100 % carbon conversion

Task 2 BFB Biomass Gasifier Design, Integration and Performance Testing

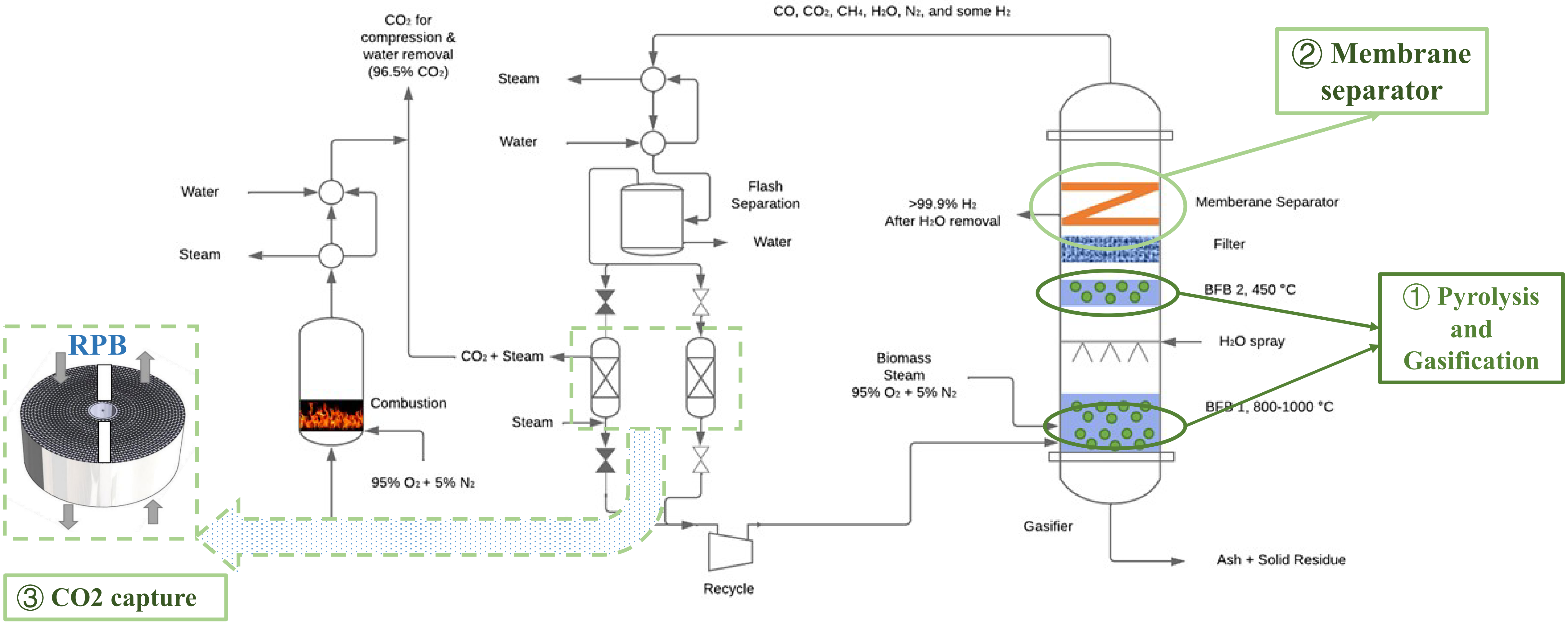
Schematic of Continuous Biomass Gasifier Syngas Yield in BFB1



BFB-Bubbling Fluidized Bed
FB-Fixed Bed
Y1-Filters
Y2-Gas Diffuser
Y3-Fluidization Zone (Biomass+Catalyst A)
Y4-Catalyst B
Y5-Glass wool
Y6-Motor
Y7-Screw Feeder
Y8-Biomass
Y9-Tar Condensor Tubes
Y10-Furnaces
DP1, DP2- Differential Pressure
T1-T6-Thermocouples



Task 3 Design of High-Temperature Resistant Hydrogen Permeation Membrane for Ultra-high Purity H_2 Production



② Membrane separator

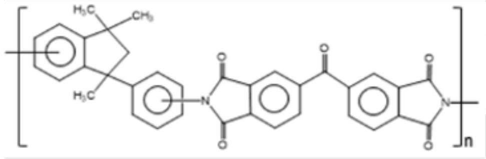
① Pyrolysis and Gasification

③ CO2 capture

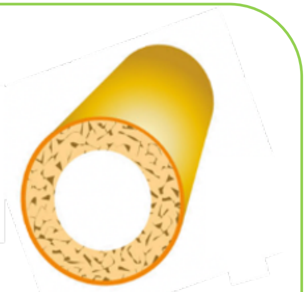
Task 3 Design of High-Temperature Resistant Hydrogen Permeation Membrane for Ultra-high Purity H_2 Production

Membrane Preparation and Characterization

Aim : To develop a membrane to produce high-purity H_2 (99.98%) in conjunction with bubbling fed fluidized reactor



Matrimid®



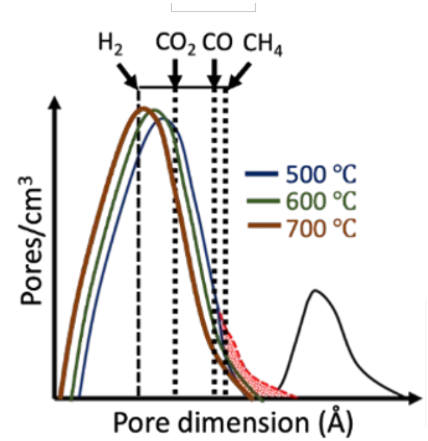
Matrimid®- based hollow fiber membrane

- Matrimid® - derived hollow fiber membranes are spun using a conventional (dry-jet/wet-quench) method of fiber spinning.
- The polymer hollow fibers are pyrolyzed in an inert environment in a three-zone furnace to form carbon molecular sieve (CMS) hollow fiber membranes.
- In the molecular level, the CMS microstructure has microporous Langmuir voids which allow for high sorption of gases as well as ultramicroporous sites which provide selectivity between similar-sized (e.g., H_2 – 0.289 nm; CO – 0.376 nm and CO_2 – 0.38 nm) penetrants.

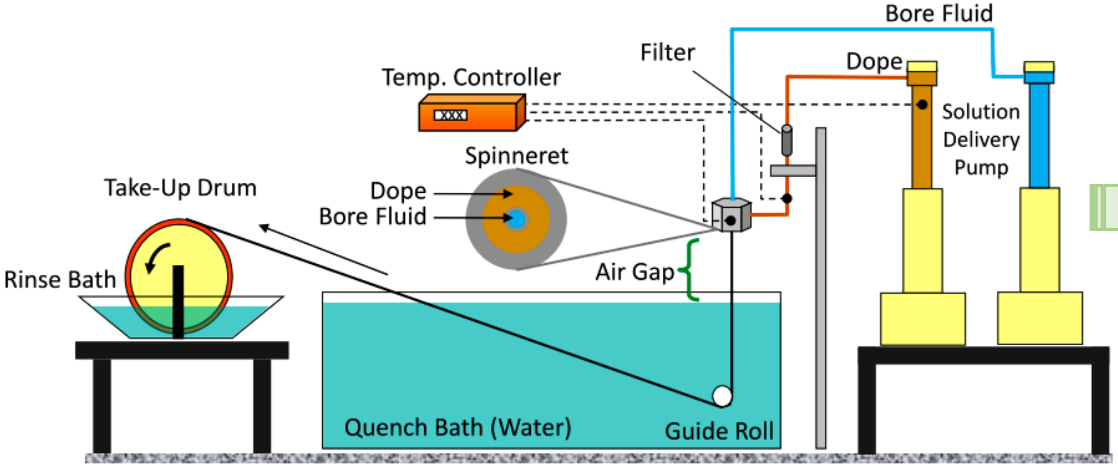
Task 3 Design of High-Temperature Resistant Hydrogen Permeation Membrane for Ultra-high Purity H_2 Production

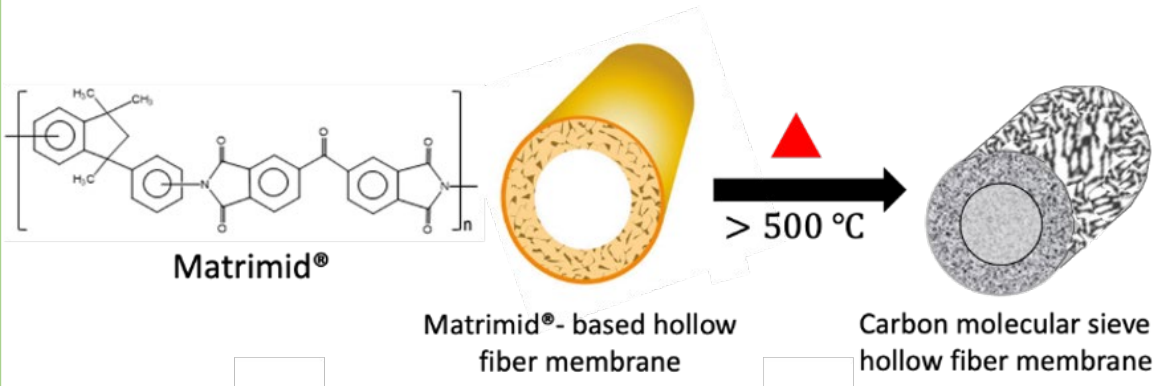
Membrane Preparation and Characterization---Methodology

CMS hollow fibers and ¼” SS module



Bimodal pore-size distribution in CMS

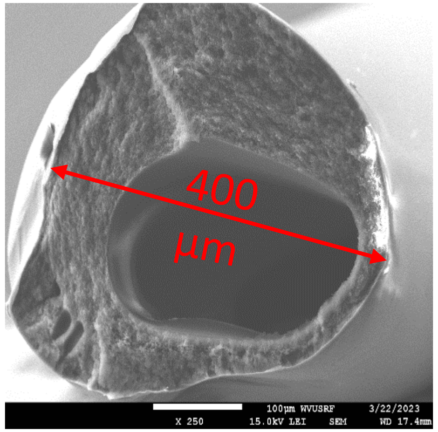




Sanyal *et al*. Journal of Membrane Science, 551, 113-122, 2018

Task 3 Design of High-Temperature Resistant Hydrogen Permeation Membrane for Ultra-high Purity H_2 Production

Matrimid® precursor fiber



Results from the best conditions for each of the 2 precursors

Spin 1
- CO_2 P/L = 126-167 GPU
- CO_2/CH_4 selectivity (α) = 16-18.5

Spin 2
- CO_2 P/L = 211GPU
- CO_2/CH_4 selectivity (α) = 7.3

Final temperature = 550 °C
Inert (Argon) atmosphere (O_2 < 1 ppm)

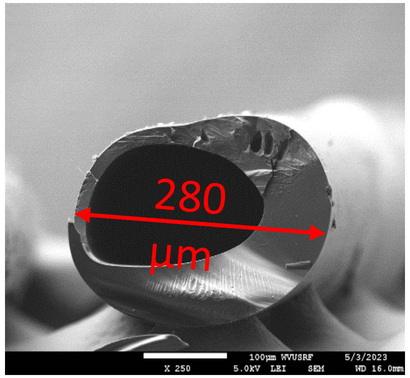
$T_{initial}$ (°C)	T_{final} (°C)	Ramp Rate (°C/min)
50	250	13
250	T_{final} -15	4
T_{final} -15	T_{final}	0.25
T_{final}	T_{final}	2 hr soak

Standard 550 °C pyrolysis protocol

The precursor were primarily tested with CO_2 and CH_4 as probes to ensure the precursors had some molecular sieving nature

Literature value of Matrimid® dense film for CO_2/CH_4 separation ~ 27 (under similar testing conditions)

Matrimid®-derived CMS fiber



Results from resulting CMS hollow fiber membranes pyrolyzed at 550°C (inert)

H_2 P/L = 26 GPU
H_2/CO_2 selectivity (α) = 4

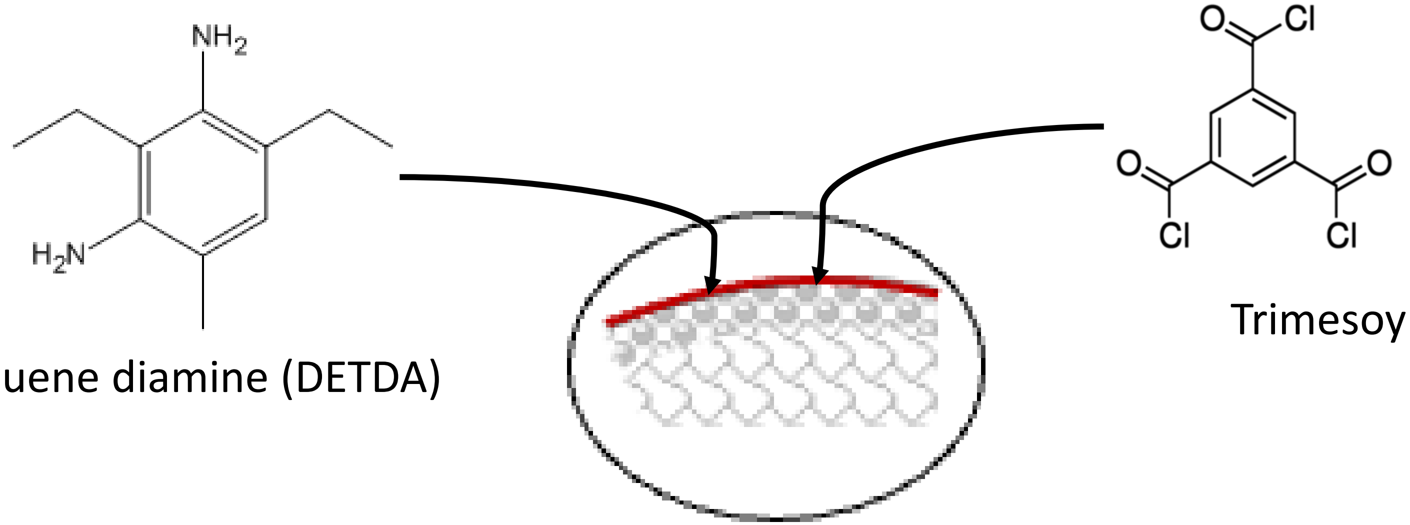
Without any additional treatment or unless processed under higher temperatures, these CMS membranes have very low H_2/CO_2 selectivities

Task 3 Design of High-Temperature Resistant Hydrogen Permeation Membrane for Ultra-high Purity H_2 Production

CMS Selective Layer Engineering Using Amine/acyl Chloride Treatment

- Typically, this strategy is used for precursor "defect curing". If any minor defects exist, they can be "cured" by utilizing DETDA/TMC post-treatment

- In this project, we are using this strategy to engineer the selective layer precisely to provide high H_2 P/L as well as high H_2/CO_2 selectivities.



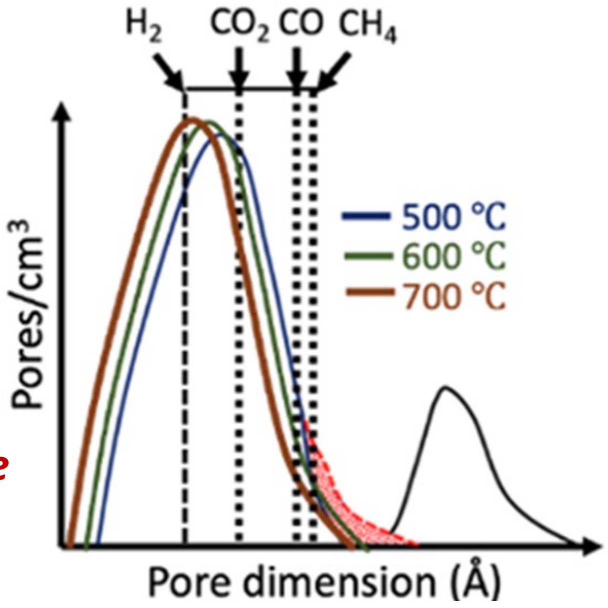
Diethyl toluene diamine (DETDA)

Trimesoyl chloride (TMC)

Task 3 Design of High-Temperature Resistant Hydrogen Permeation Membrane for Ultra-high Purity H_2 Production

Hypothesis to validate – combining high temperature (800-900 ℃) pyrolysis and DETDA/TMC treatment would lead to highly selective membranes

Recall high temperature pyrolysis is expected to lead to tightened ultramicropores



Our plan is to reduce the DETDA concentration (~0.05 wt.%) and exposure time (5-10 mins) to avoid excessive tightening

Both cases led to low H_2 P/L and low H_2/CO_2 and H_2/CH_4 selectivities

Pyrolysis Temp **800℃** fast (fast refers to high **15 ℃/*min***)

	Stage 3 CMS(V150,D,T) Spin 2				
	GPU CO2	**GPU H2**	**GPU CH4**	**H2/CO2**	**H2/CH4**
Module 2	8.8	28.8	11.8	3.272727	2.787489
Module 3	7.73	30.3	10.87	3.919793	2.787489
Module 4	0.84	4.6	1.2	5.47619	3.833333

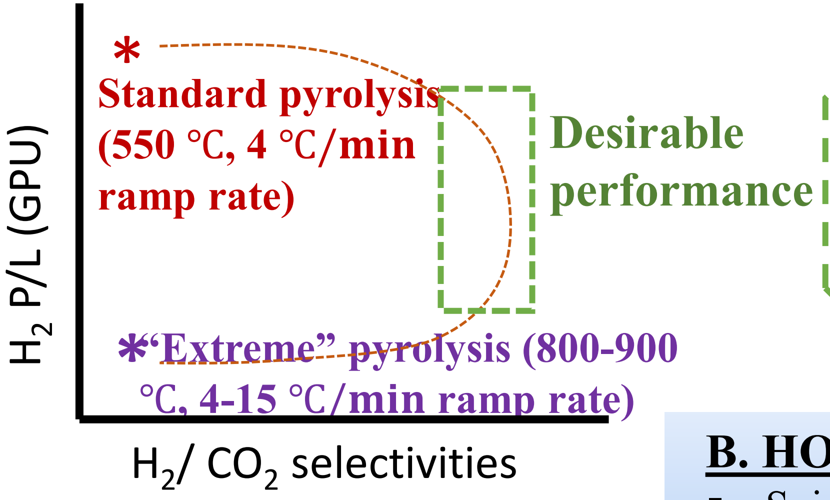
Pyrolysis Temp **900℃** standard (**4 ℃/*min*** ramp rate)

	Stage 3 CMS(V[ND],D,T) Spin 2				
	GPU CO2	**GPU H2**	**GPU CH4**	**H2/CO2**	**H2/CH4**
Module 1	1.4	3.8	0.4	2.714286	9.5
Module 2	0.8	0.7	3.4	0.875	0.205882
Module 3	2.8	0.9	10.5	0.321429	0.085714
Module 4	6.8	4	13.5	0.588235	0.296296

Task 3 Design of High-Temperature Resistant Hydrogen Permeation Membrane for Ultra-high Purity H_2 Production

Hypothesis to validate – combining high temperature (800-900 ℃) pyrolysis and DETDA/TMC treatment would lead to highly selective membranes, cont.

A. PYROLYSIS CONDITIONS AND DEFECT-CURING STRATEGY TUNING

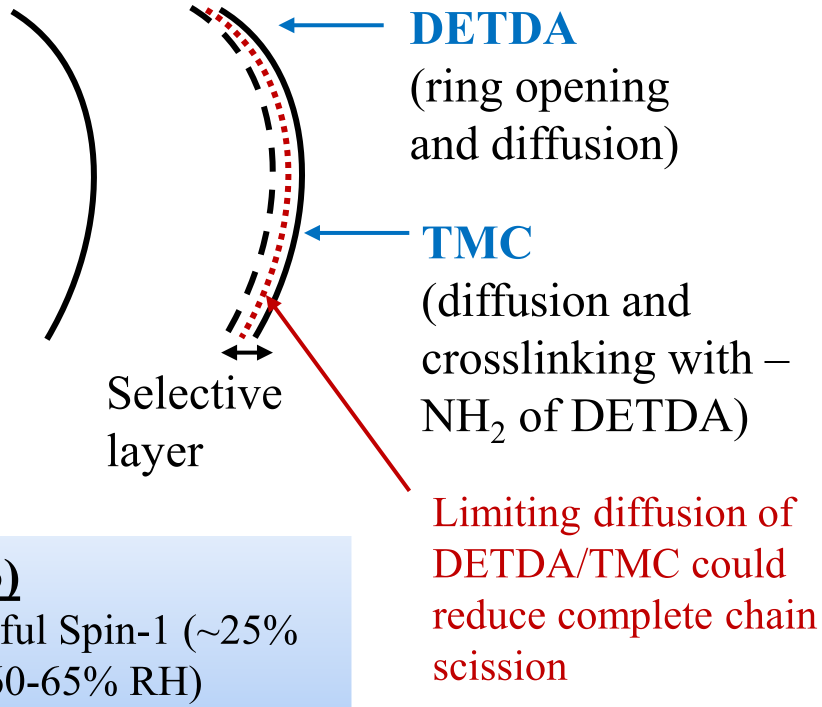


*** Standard pyrolysis (550 ℃, 4 ℃/min ramp rate)**

Desirable performance

***“Extreme” pyrolysis (800-900 ℃, 4-15 ℃/min ramp rate)**

550℃ pyrolysis with lower (<0.1 wt.%) DETDA/TMC concentrations and lower (<30 mins) exposure time



DETDA (ring opening and diffusion)

TMC (diffusion and crosslinking with –NH_2 of DETDA)

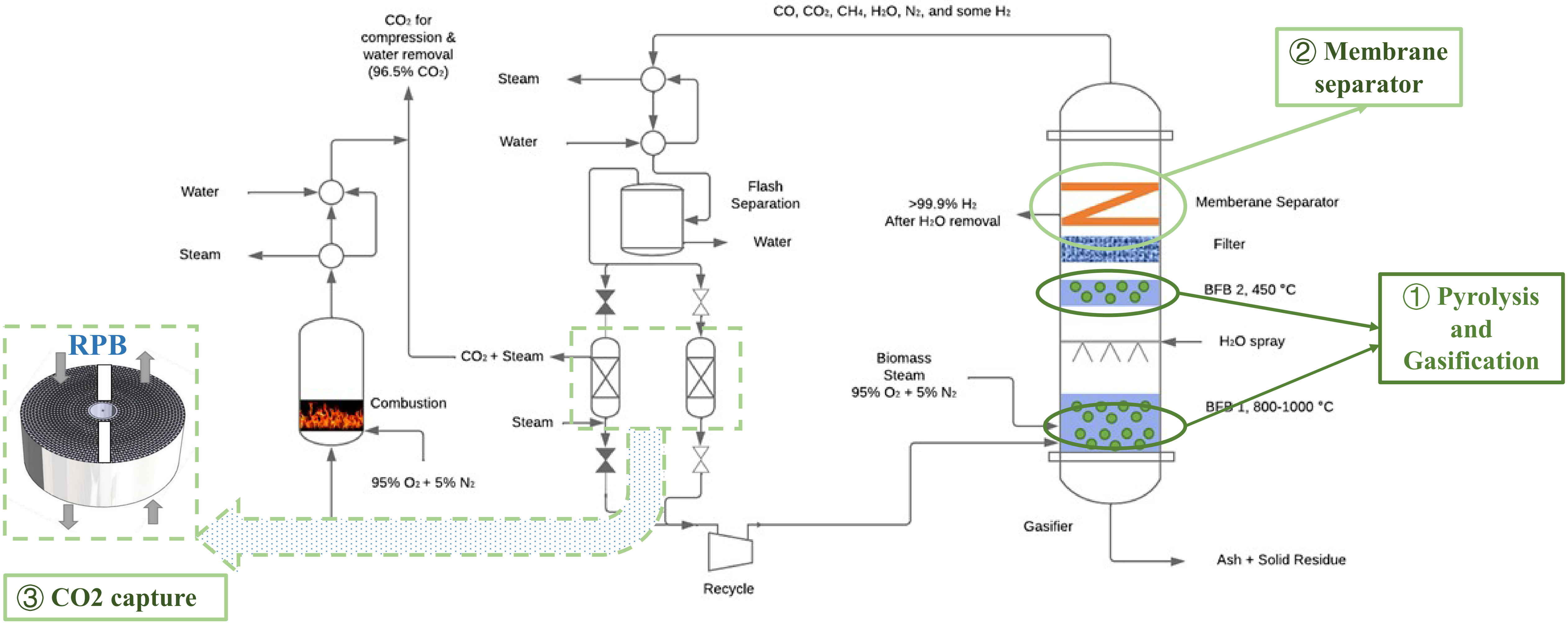
Limiting diffusion of DETDA/TMC could reduce complete chain scission

We speculate that we may have overtightened the ultramicropores

B. HOLLOW FIBER SPINNING (SPIN – 3)

- Spin -3 was done at ~35% RH (closer to successful Spin-1 (~25% RH) and much lower RH compared to Spin -2 (60-65% RH)
- Spin-3 precursor testing (evaluation of “*defect-free*” nature) and SEM imaging being pursued now.

Task 4 Process Modeling, Optimization, and Techno-Economic Analysis



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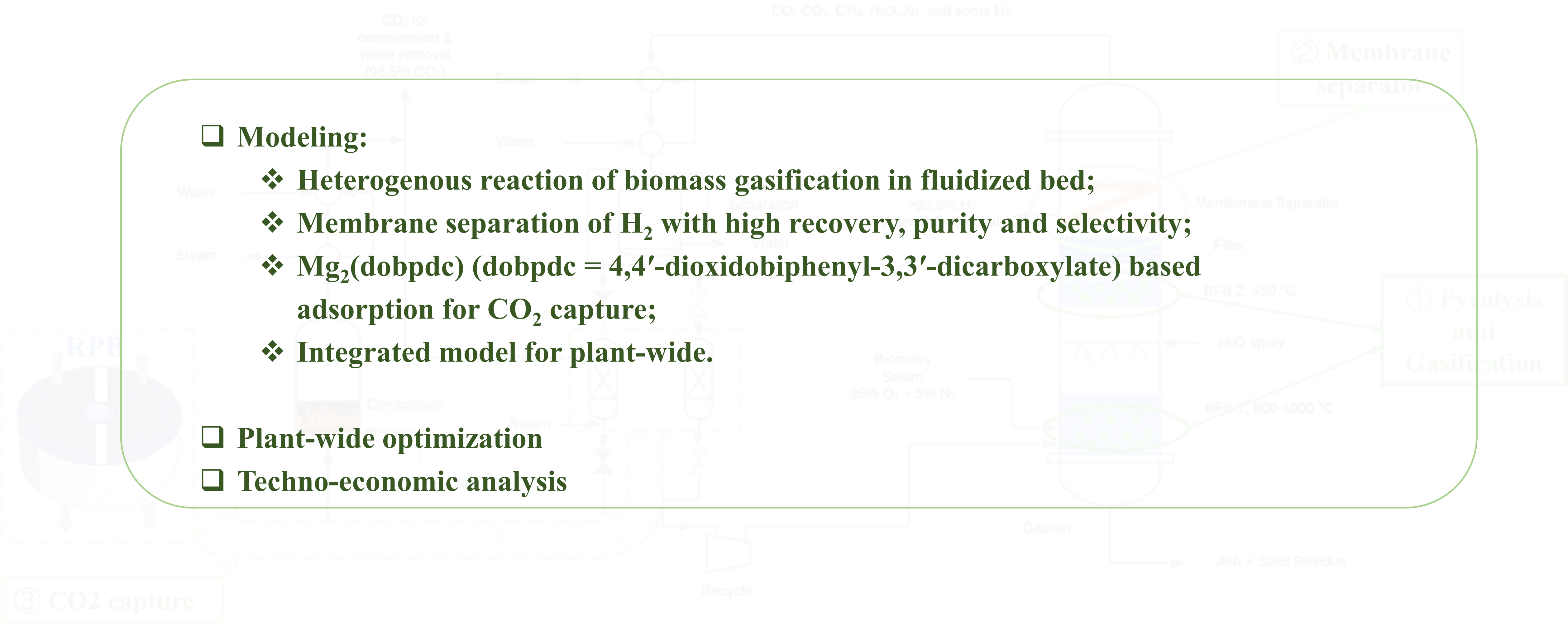
Task 4 Process Modeling, Optimization, and Techno-Economic Analysis



- ❑ **Modeling:**
  - ❖ **Heterogenous reaction of biomass gasification in fluidized bed;**
  - ❖ **Membrane separation of H_2 with high recovery, purity and selectivity;**
  - ❖ **Mg_2(dobpdc) (dobpdc = 4,4′-dioxidobiphenyl-3,3′-dicarboxylate) based adsorption for CO_2 capture;**
  - ❖ **Integrated model for plant-wide.**

- ❑ **Plant-wide optimization**
- ❑ **Techno-economic analysis**

Task 4 Process Modeling, Optimization, and Techno-Economic Analysis

Shrinking particle model assumption has been validated

Statistical Summary of coal and biomass feed composition (% w/w)

Feed Source	Mean	Standard Deviation	Variance	SEM
Coal	9.12	2.96	8.76	1.48
Biomass	1.12	1.12	1.26	0.28

db: Dry basis. Source: As reported by (Alauddin et al., 2010)
VM: volatile matter; FC: fixed carbon. Source: As reported by (Kasule et al., 2014)

Ash content in biomass is significantly lower than that in coal.

$$k_{overall} = \frac{1}{\frac{1}{k_{dg}} + \frac{1}{Y^2 k_s}}$$

$Y = r_c/R$; r_c is the radius of the particle along reactor length and R the radius of the particle at the inlet.

k_{dg}, k_s are the gas film diffusion coefficient, and the surface reaction constants, respectively.

P_{eff} is the effective partial pressure of the i^{th}-component in the gas participating in the gasification reactions.

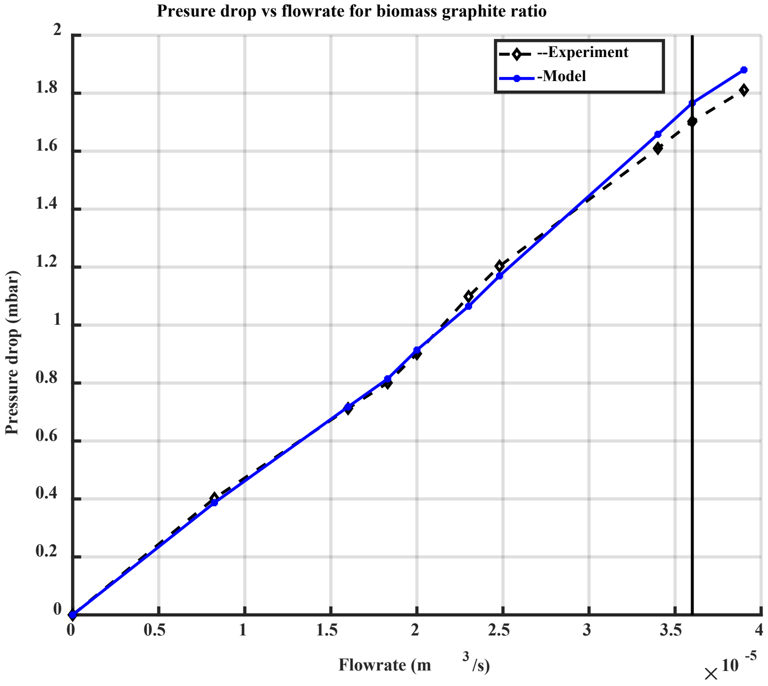
A shrinking particle model is assumed for heterogenous gasification modeling.

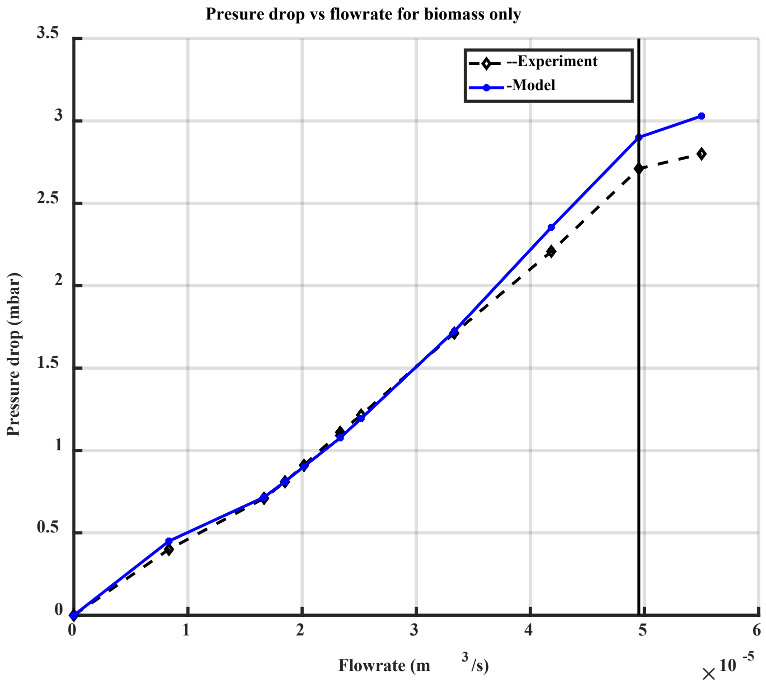
Task 4 Process Modeling, Optimization, and Techno-Economic Analysis

Cold flow model validation with pressure drop

Comparison of Experimental and Model results

	Biomass only	Biomass/ Catalyst (9:1)
Experiment (ΔP)(mbar)	**2.71**	**1.70**
Calculated (ΔP)(mbar)	**2.95**	**1.76**
U_{mf} (150-200 µm) (m/s)	**0.37**	**0.32**

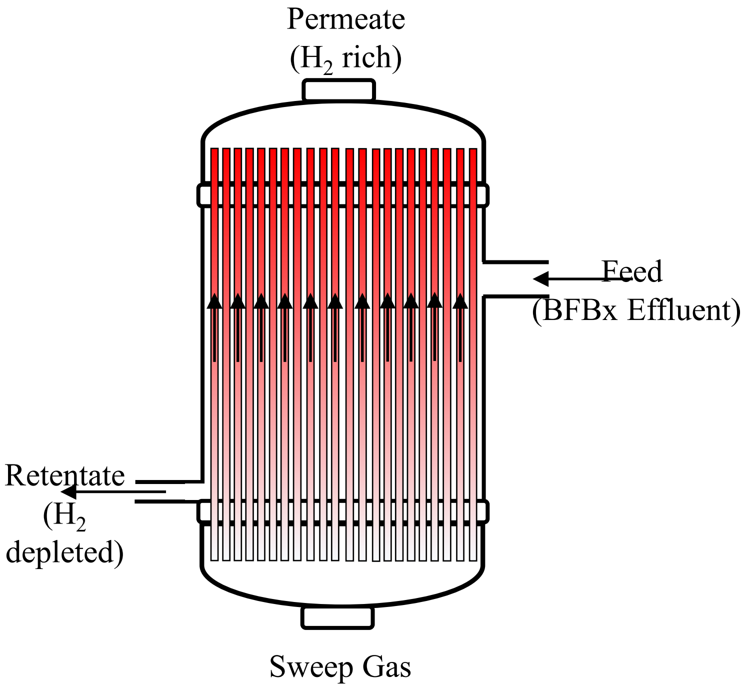

Presure drop vs flowrate for biomass graphite ratio


Presure drop vs flowrate for biomass only

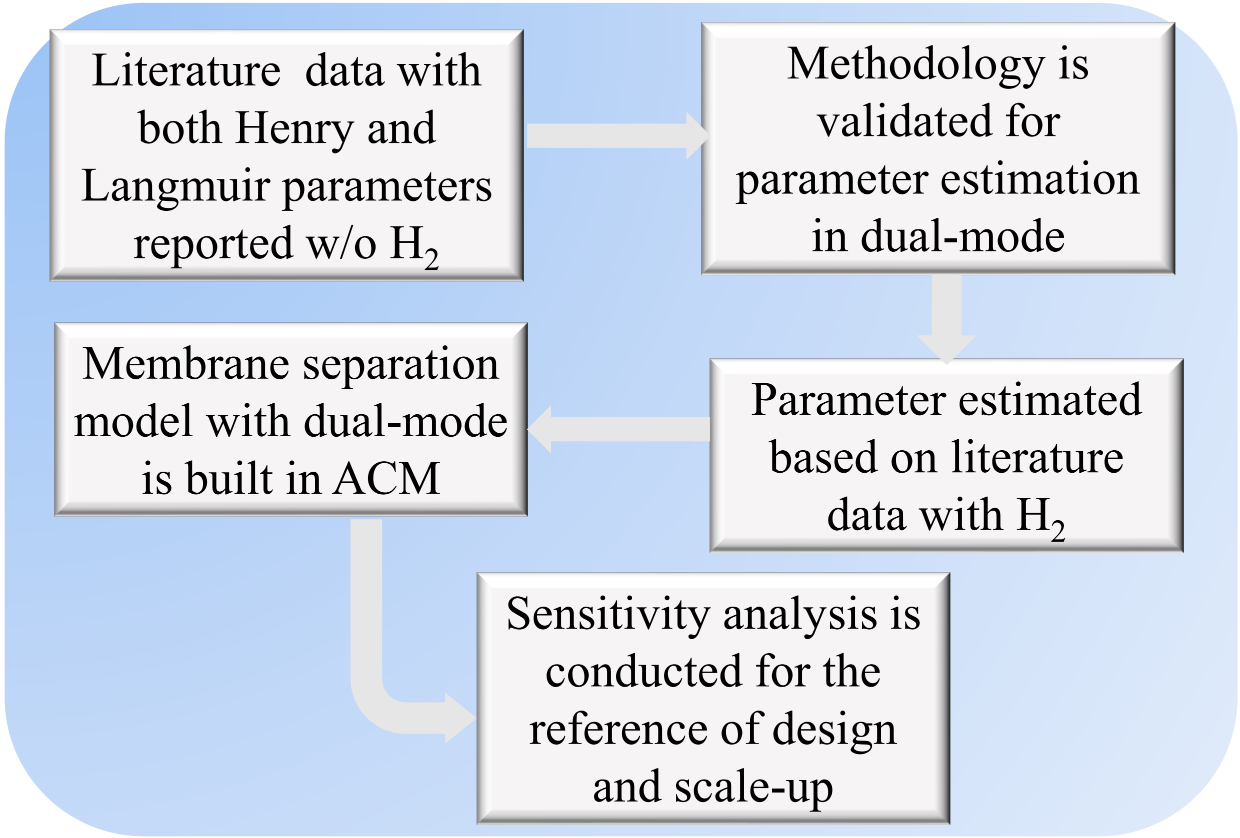
Considering the presence of the perforated plate at the gas entrance, the pressure drop is calculated, which is found to agree well with experimental data.

Task 4 Process Modeling, Optimization, and Techno-Economic Analysis

Membrane Separation Modeling and Parameter Estimation



Membrane Separator schematic representation



Literature data with both Henry and Langmuir parameters reported w/o H_2

Methodology is validated for parameter estimation in dual-mode

Parameter estimated based on literature data with H_2

Membrane separation model with dual-mode is built in ACM

Sensitivity analysis is conducted for the reference of design and scale-up

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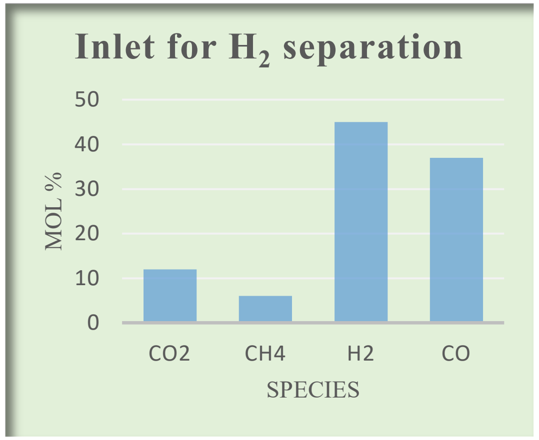
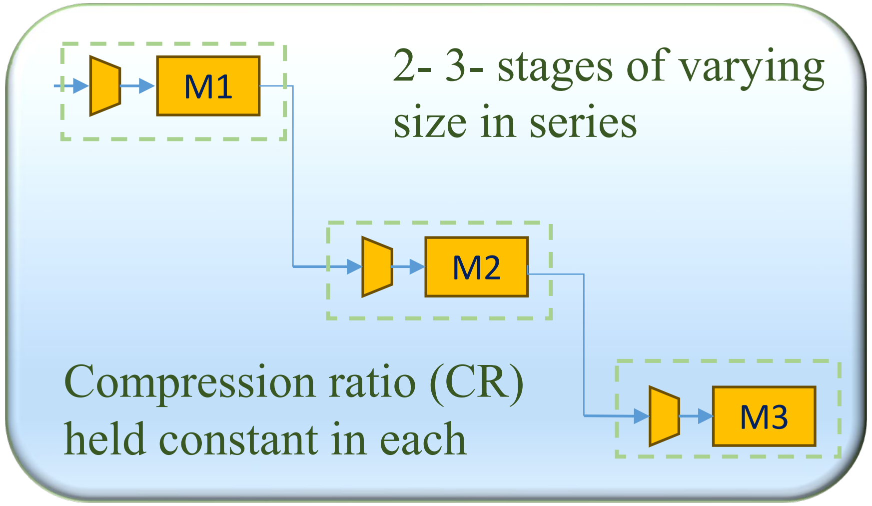
Multi-stage membrane units for H_2 Separation

Assumptions:



$18.72/GJ electricity, $20/m^2 membrane area, annual rate 8%, membrane/compressor lifetime = 5 years, 8000 working hours per year.



2- 3- stages of varying size in series

Compression ratio (CR) held constant in each

3-stage setting for membrane separation.

Separation requirement:

H_2 Purity: >95%;
H_2 Recovery: >75%.

Specification for CR sensitivity

Stage	Specifications in each stage
M1	H_2 recovery > 90%, variable size
M2	H_2 recovery >70%, variable size (later assumes specification of **M3**)
M3	H_2 purity > 95%, variable size

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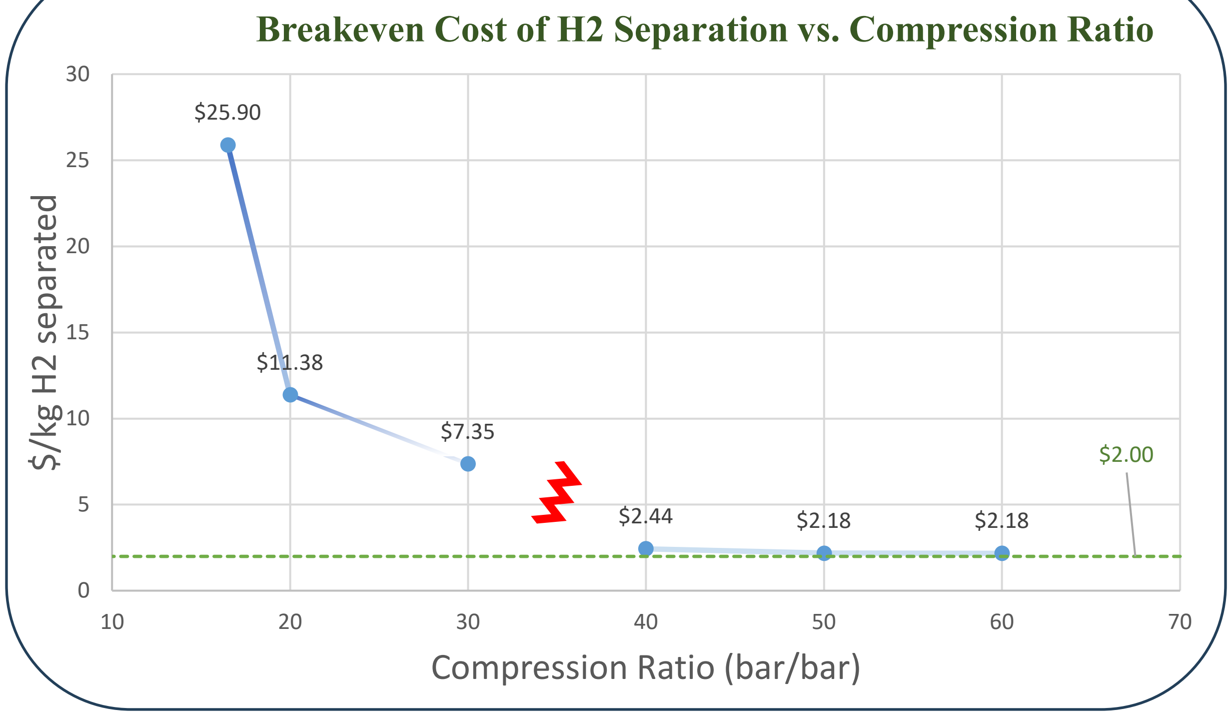
Results and Sensitivity for Breakeven Cost *vs.* Compression Ratio

- Minimum pressure ratio for 3-stage system to achieve purity target is **16.5** bar
- CR > 30 bar made third stage redundant and decreased cost considerably



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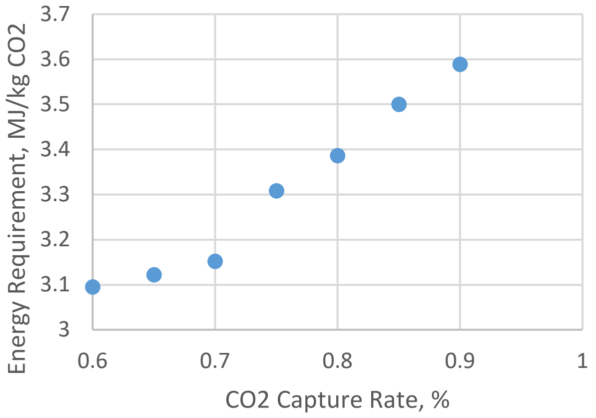
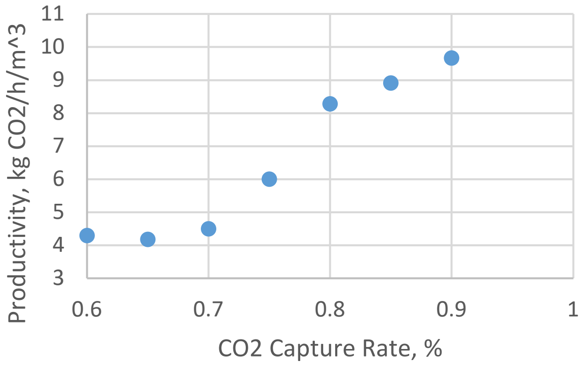
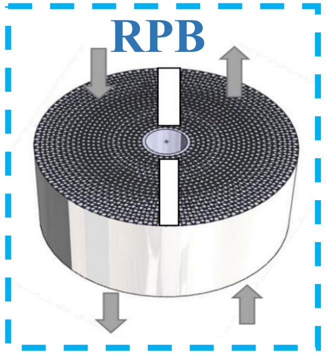
CO_2 Separator Modeling and Model Validation

Flue gas source	CO_2 conc. (%)	P (atm)	CO_2 partial pressure (atm)
Gas turbine	3-4	1	0.03-0.04
Fired boiler of oil refinery and petrochemical plant	~8	1	0.08
Natural gas fired boilers	7-10	1	0.07-0.10
Oil-fired boilers	11-13	1	0.11-0.13
Coal-fired boilers	12-14	1	0.12-0.14
IGCC[a] after combustion	12-14	1	0.12-0.14
Hydrogen production	**15-20**	**22-27**	**3-5**
Streel production (blast furnace)	20-27	1-3	0.2-.06
Aluminum production	1-2	1	0.01-0.02
Cement process	14-33	1	0.14-0.33

[a] IGCC, integrated gasfication combined cycle. Figueroa et al., 2008; Chu, 2009).







Optimal results are obtained with changing bed length, rotation speed, fraction of bed, direct and indirect steam flowrate, etc.

Inlet mole fraction of CO_2 is assumed to be 15%

Future Work

- Test various operational parameters in the two-stage gasifier by using different gasifying agents and catalysts
- Incorporate gasifier with the membrane reactor
- Optimize pyrolysis and other conditions for membrane manufacturing and and optimize the defect curing step for maximizing selectivity and recovery
- Complete and validated non-isothermal gasifier model, ands membrane model, CO_2 capture system models.
- Complete optimization and techno-economic analysis of the plant-wide system

Acknowledgement

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- Support from Drs. Evelyn Lopez and Sarah Michalik from NETL

Thank you for your attention

Questions?

Process Modeling------CO$_2$ Separator Modeling and Model Validation

CO$_2$ in Different Processes and Capture Result by RPB for 15%

	Doc	Value	Value	Value	Value	Value	Value	Value
ads.L	Bed Length [m]	23.9412409	24.55167824	24.85108349	32.15661792	40	40	35.9496199
ads.D	Bed diameter [m]	10	10	10	10	10	10	10
ads.w_rpm	bed rotational speed [revolutions/min]	0.005478633	0.005143607	0.004068796	0.003185256	0.00204922	0.001996311	0.00207667
ads.theta	Fraction of bed [-]	0.453319628	0.464745531	0.519057412	0.607292384	0.762785759	0.763577131	0.753768243
des.theta	Fraction of bed [-]	0.546680372	0.535254469	0.480942588	0.392707616	0.237214241	0.236422869	0.246231757
ads.P_in	Inlet flue gas pressure [bar]	1.760509026	1.760509026	1.693486368	1.724899349	1.686161928	1.687734435	1.642636601
ads.P_out	Outlet adsorber pressure [bar]	1.01325	1.01325	1.01325	1.01325	1.01325	1.01325	1.01325
ads.F_in	Inlet adsorber gas flow [mol/s]	566.5837506	566.5837506	566.5837506	566.5837506	566.5837506	566.5837506	566.5837506
ads.Tg_in	Inlet flue gas temperature [K]	363	363	363	363	363	363	363
ads.Tx	heat exchange fluid temperature, constant [K]	365.7212138	368	368	368	368	368	368
des.P_in	Inlet flue gas pressure [bar]	1.075940703	1.06975813	1.07760409	1.104928447	1.192434454	1.174187106	1.138756112
des.P_out	Outlet adsorber pressure [bar]	1.01325	1.01325	1.01325	1.01325	1.01325	1.01325	1.01325
des.F_in	Inlet adsorber gas flow [mol/s]	25.78230611	17.65192243	20.75773675	17.20535863	16.9812937	14.33384576	12.52131998
des.Tg_in	Inlet flue gas temperature [K]	393	393	393	393	393	393	393
des.Tx	heat exchange fluid temperature, constant [K]	429.4869519	429.8433447	433	429.9436207	432.3018625	430.2352644	429.4414099
ads.CO2_capture	CO2 capture fraction	0.9	0.85	0.8	0.75	0.7	0.65	0.6
energy_requirement	Energy requirement [MJ/kg CO2]	3.588480552	3.499373905	3.38575141	3.307960847	3.151155623	3.121919606	3.094440133
productivity	Productivity [kg CO2/h/m^3]	9.66733661	8.903253309	8.27857612	5.997936807	4.500378451	4.178922848	4.292081257
ads.y_in[N2]	inlet mole fraction	0.8	0.8	0.8	0.8	0.8	0.8	0.8
ads.y_in[CO2]	inlet mole fraction	0.15	0.15	0.15	0.15	0.15	0.15	0.15
ads.y_in[H2O]	inlet mole fraction	0.05	0.05	0.05	0.05	0.05	0.05	0.05
ads.y_out[N2]	outlet mole fraction	0.924855491	0.916905444	0.909090909	0.901408451	0.893854749	0.886426593	0.879120879
ads.y_out[CO2]	outlet mole fraction	0.01734104	0.025787966	0.034090909	0.042253521	0.05027933	0.058171745	0.065934066
ads.y_out[H2O]	outlet mole fraction	0.057803468	0.05730659	0.056818182	0.056338028	0.055865922	0.055401662	0.054945055

Producing Clean Hydrogen Using a Modular Two-Stage Intensified Membrane-Enhanced Catalytic Gasifier