Budget Period 2 Review

LAB-SCALE DEVELOPMENT OF A HYBRID CAPTURE SYSTEM WITH ADVANCED MEMBRANE, SOLVENT SYSTEM AND PROCESS INTEGRATION

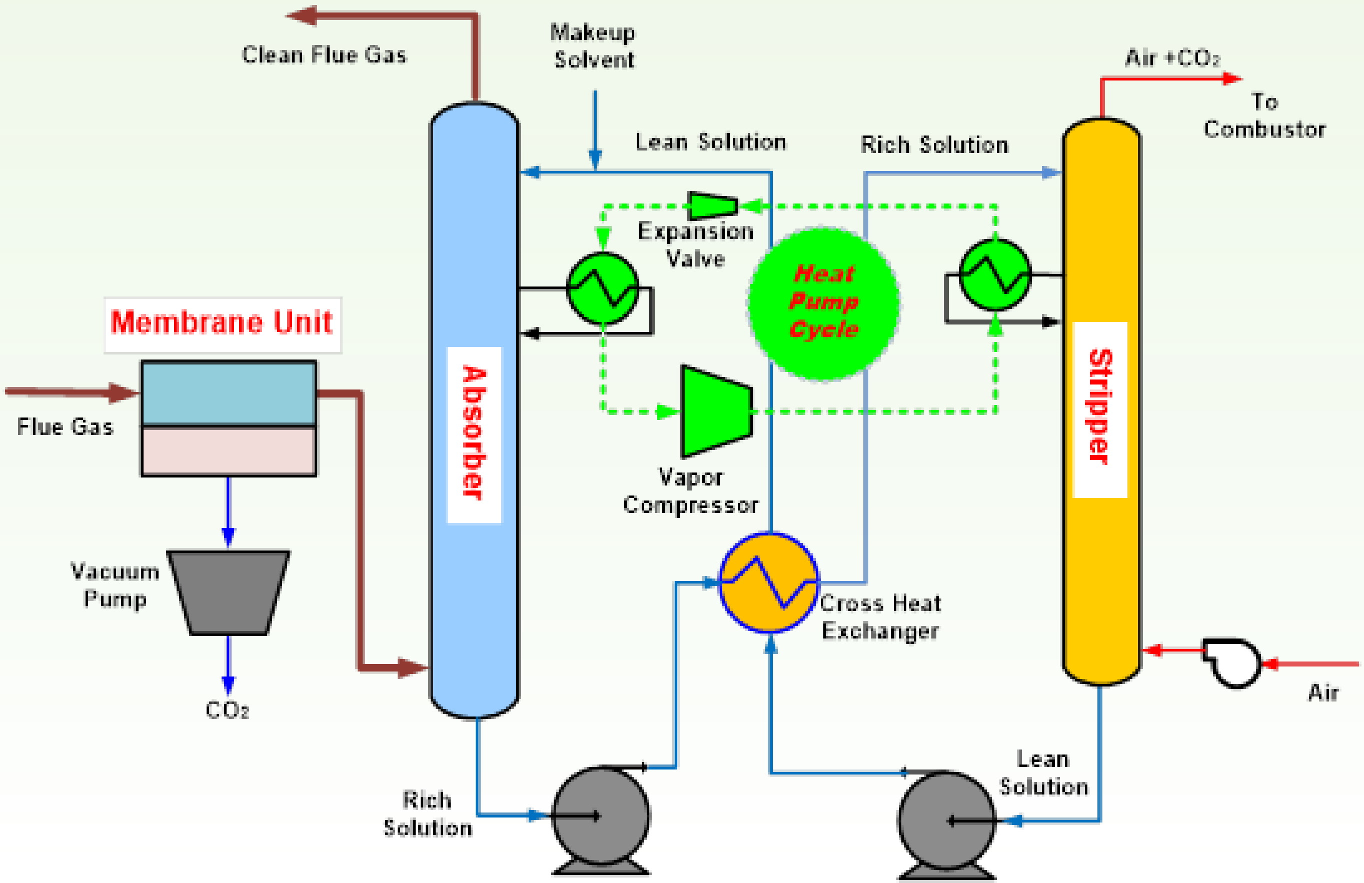
DE-FE0026464

CARBON CAPTURE SCIENTIFIC LLC

Liquid Ion Solutions

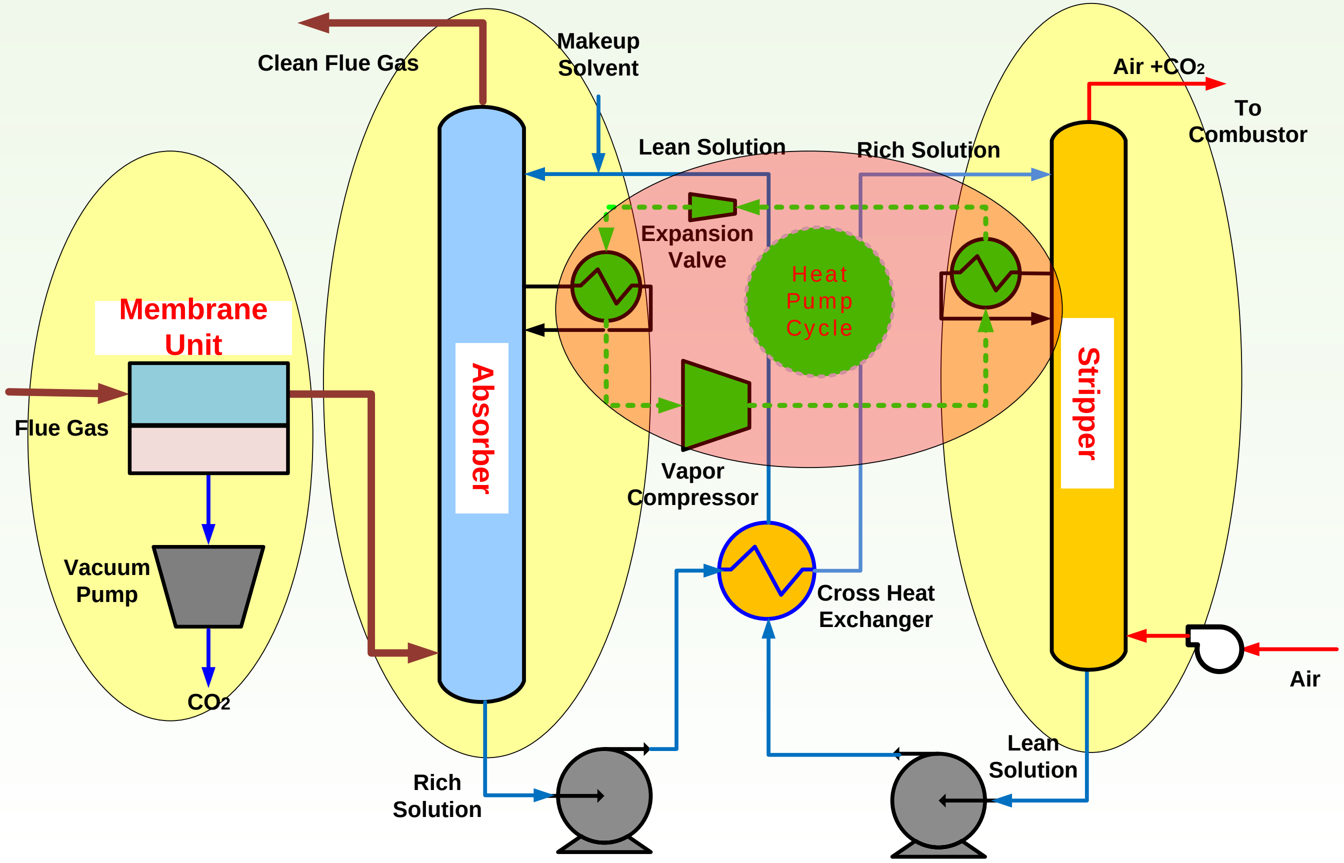
PennState

A New Kind of Membrane Integration



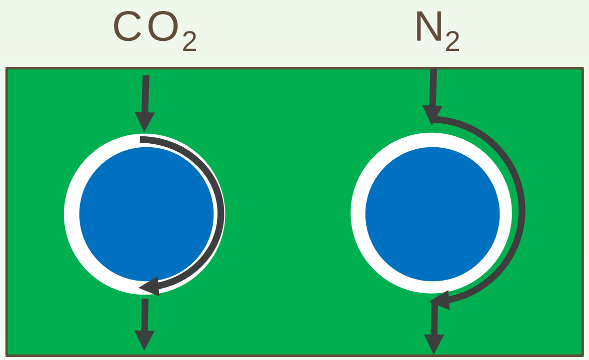
Membrane/Solvent Integrated Process

- Advantages
  - Tail-end technology which is easily used in retrofits
  - No steam extraction is required
  - Heat pump is seamlessly integrated into the cooling and heating of absorption/stripping process
  - Operating pressure of the stripper will be very flexible depending on the low quality heat
- Disadvantage
  - Capital cost could be intensive



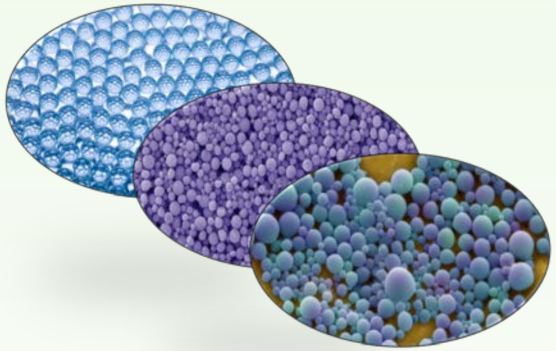
Interfacially-Controlled Envelope (ICE) Membranes

If you can't beat 'em, join 'em!

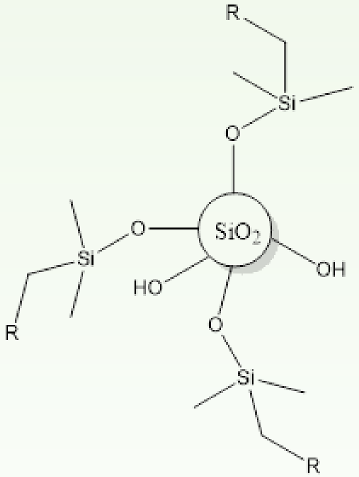


- Makes use of envelopment effects which have plagued mixed matrix membranes
- Diffusion phenomena determined by interactions with the particle and polymer surface
- Use simple nanoparticle fillers
- Advanced polymers allow an excellent starting point

Membrane Fabrication and Optimization



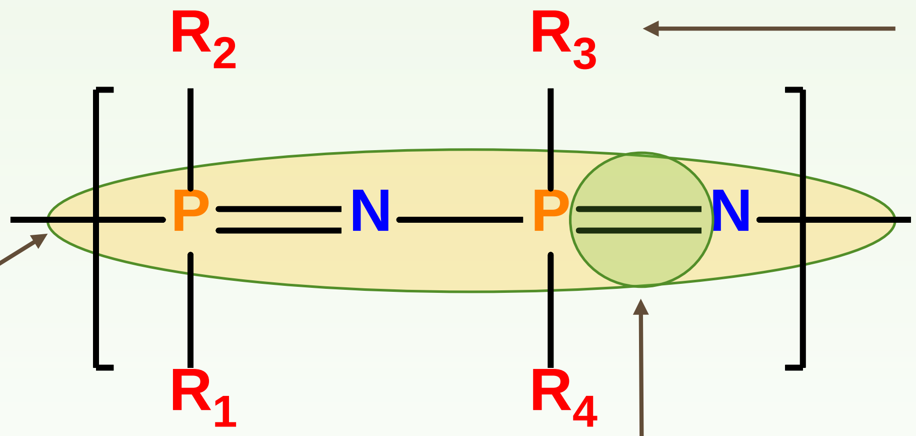
Nano-particle
Selection



Nano-particle
Modification

Membrane Film
Fabrication

What's so great about polyphosphazenes?

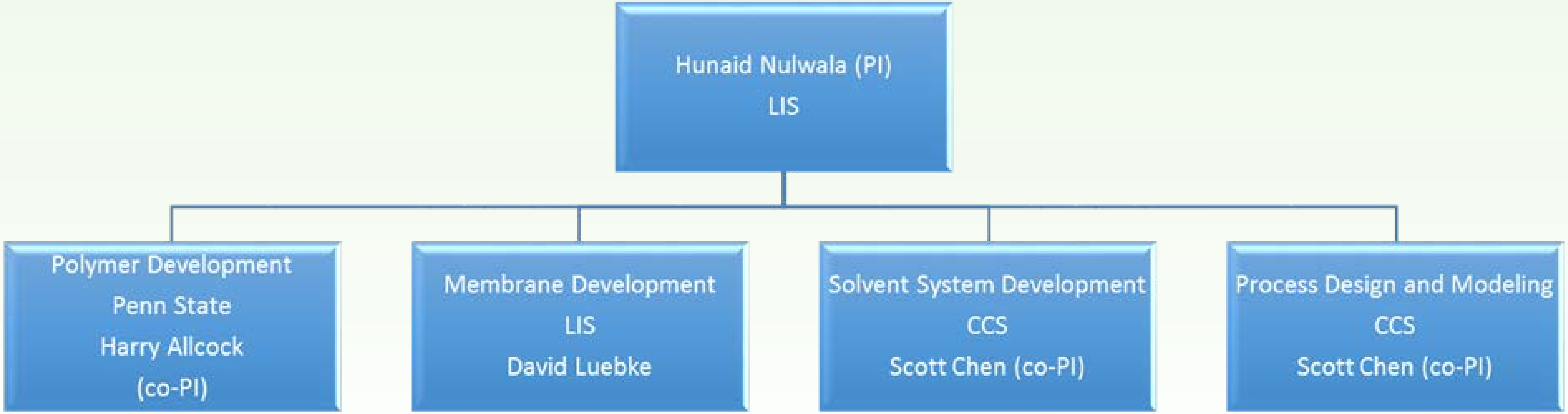


Macromolecular substitution

<u>Ultra Flexible chains</u>
High chain mobility
Improved gas solubility and diffusion

<u>Excellent chemical and thermal stability</u>
C-C =607Δ*Hf* kJ/mol vs. P-N =617 Δ*Hf* kJ/mol

Project Organization



Project Objectives

- ✓ Simulation of Membrane and Solvent Subsystems
- ✓ Synthesis and Characterization of Tailored Polyphosphazenes
- ✓ Fabrication of Interfacially-Controlled Envelope (ICE) Membranes
- ✓ Optimization of Polymer, Nano-filler, and Fabrication Process
- ✓ Design and Construction of Membrane Test Systems
- ✓ Modification of Absorber and Stripper Units of Solvent System
- ✓ Optimization of Solvent System for Operation in Hybrid System
- Demonstration of All Subsystems with Simulated Flue Gas
- Completion of Preliminary Economic Analysis

Schedule

				Budget Period 1				Budget Period 2				Budget Period 3			
				10/1/2015 - 9/30/2016				10/1/2016 - 9/30/2017				10/1/2017 - 9/30/2018			
	Start Date	End Date	Cost	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4
Task 1.0 - Project Management and Planning	10/1/2015	9/30/2018	$111,197.00												
Subtask 1.1 - Project Management and Planning	10/1/2015	9/30/2018													
Subtask 1.2 - Briefings and Reports	10/1/2015	9/30/2018													
Milestones															
- Milestone A - Update Project Management Plan				Δ											
- Milestone B - Kickoff Meeting				Δ											
Task 2.0 - Computer Simulation of Hybrid Process	10/1/2015	9/30/2016	$130,520.00												
Subtask 2.1 - Simulation of Membrane System	10/1/2015	3/31/2016													
Subtask 2.2 - Simulation of Absorption/Air Stripping System	1/1/2016	6/30/2016													
Subtask 2.3 - Simulation of Heat Pump Cycle	1/1/2016	6/30/2016													
Subtask 2.4 - Optimization of the Hybrid Process	4/1/2016	9/30/2016													
Milestones															
- Milestone C - Complete Optimized Hybrid Process Design							Δ								
Task 3.0 - Generation 0 ICE Membrane Development	10/1/2015	9/30/2016	$585,160.00												
Subtask 3.1 - Polyphosphazene Synthesis and Characterization	10/1/2015	9/30/2016													
Subtask 3.2 - Design and Contruction of Isochoric Membrane System	10/1/2015	3/31/2016													
Subtask 3.3 - Generation 0 (Neat Polymer) ICE Membrane Performance Testing	4/1/2016	9/30/2016													
Subtask 3.4 - Nano-filler Particle Selection	4/1/2016	9/30/2016													
Milestones															
- Milestone D - Down-select to Three or Fewer Polyphosphazene Candidates							Δ								
Task 4.0 - Modification, Installation, and Testing of Absorption Column	10/1/2015	9/30/2016	$200,137.00												
Subtask 4.1 - Modification of Absorption Column	10/1/2015	3/31/2016													
Subtask 4.2 - Installation of Absorption Column	1/1/2016	6/30/2016													
Subtask 4.3 - Parametric Testing of Absorption Column	4/1/2016	9/30/2016													
Task 5.0 - Generation 1 ICE Membrane Development	10/1/2016	9/30/2017	$576,420.00												
Subtask 5.1 - Polyphosphazene Optimization and Characterization	10/1/2016	9/30/2017													
Subtask 5.2 - Fabrication of Generation 1 ICE Membranes	10/1/2016	3/31/2017													
Subtask 5.3 - Generation 1 ICE Membrane Performance Testing	4/1/2017	9/30/2017													
Subtask 5.4 - Design and Construction of Mixed Gas Membrane Testing System	4/1/2017	9/30/2017													
Milestones															
- Milestone E - Complete Fabrication of Gen 1 ICE Membrane									Δ						
- Milestone F - Down-select of Optimizal Polymer/Particle Composition											Δ				
- Milestone G - Complete Construction and Shakedown of Iso-baric System											Δ				
Task 6.0 - Modification, Installation, and Testing of Air Stripper	10/1/2016	9/30/2017	$215,171.00												
Subtask 6.1 - Modification of Air Stripper	10/1/2016	3/31/2017													
Subtask 6.2 - Installation of Air Stripper	1/1/2017	6/30/2017													
Subtask 6.3 - Parametric Testing of Air Stripper	4/1/2017	9/30/2017													
Milestones															
- Milestone H - Complete Modification of Stripper									Δ						
Task 7.0 - ICE Membrane Scale-up and Simulated Flue Gas Testing	10/1/2017	9/30/2018	$498,365.00												
Subtask 7.1 - Polyphosphazene Scale-up and Characterization	10/1/2017	9/30/2018													
Subtask 7.2 - Fabrication of Optimized ICE Membranes	1/1/2018	6/30/2018													
Subtask 7.3 - Simulated Flue Gas Testing	4/1/2018	9/30/2018													
Milestones															
- Milestone I - Complete Fabrication of Optimized Membrane														Δ	
- Milestone J - Complete Simulated Flue Gas Testing															Δ
Task 8.0 - Preliminary Techno-Economic Analysis	10/1/2017	9/30/2018	$180,630.00												
Subtask 8.1 - System Performance Modeling	10/1/2017	3/31/2018													
Subtask 8.2 - Economic Analysis	1/1/2018	6/30/2018													
Subtask 8.3 - Sensitivity Analysis	4/1/2018	9/30/2018													
Milestones															
- Milestone K - Complete System Performance Model													Δ		
- Milestone L - Complete Preliminary Techno-Economic Analysis															Δ

Budget Period 2

Tasks for Budget Period 2 (2017)

- Task 5: Generation 1 ICE Membrane Development
  - 5.1: Polyphosphazene Optimization and Characterization
  - 5.2: Fabrication of Generation 1 ICE Membrane
  - 5.3: Generation 1 ICE Membrane Performance Testing **(I5) (K5 on schedule)**
  - 5.4: Design and Construction of Mixed Gas Membrane Testing System **(L5)**
- Task 6: Modification, Installation, and Testing of Air Stripper
  - 6.1: Modification of Air Stripper **(J6)**
  - 6.2: Installation of Air Stripper
  - 6.3: Parametric Testing of Air Stripper **(M6 on schedule)**

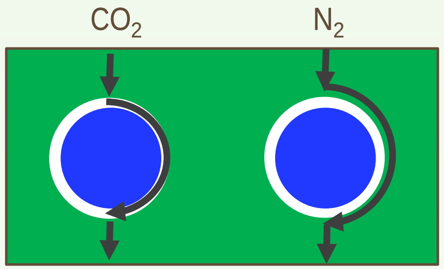
Task 5.1 and 5.2. Polymer Synthesis and Gen 1 ICE Membrane Fabrication

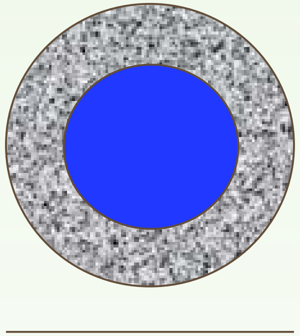
Plan

- Planned membrane fabrication work
  - Scale-up of our new casting method
  - Identify better supports for thin-film deposition
  - Characterized the fabricated films

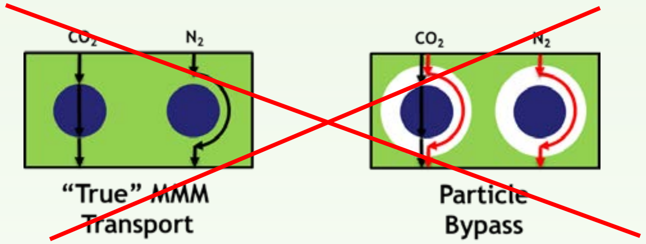
- Planned synthesis work
  - Synthesis of nano-particles and surface modification
  - Scale up of polymer synthesis (Ideal polymer)

Task 5.1 and 5.2. Polymer Synthesis and Gen 1 ICE Membrane Fabrication



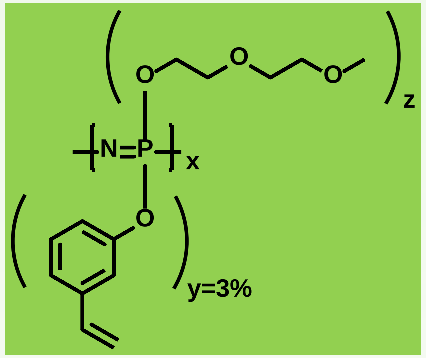


10-15 nm



- Use simple nanoparticle fillers
  - Down selected the nano-particles to Silica 10-15nm particles. (complete)
- Surface modify the particles to tune optimal interactions with CO_2 and the polymer
  - Identified ideal surface group and perfected the chemistry (complete)

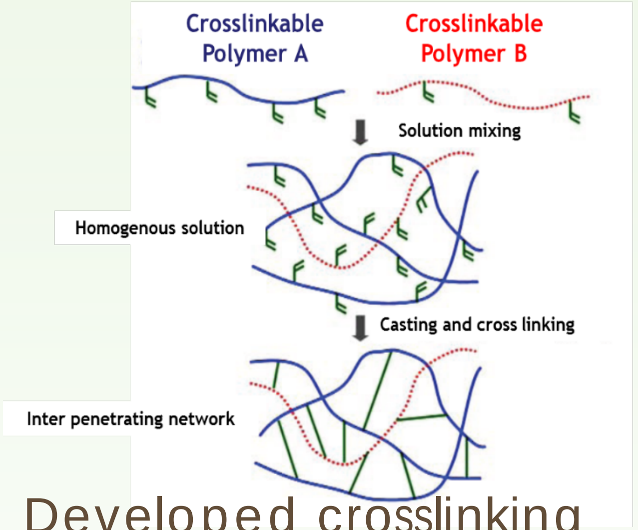
Task 5.1 and 5.2. Polymer Synthesis and Gen 1 ICE Membrane Fabrication
(Optimization of chemistry for membrane fabrication)



Identify ideal polymer for ICE membrane optimization (complete)

Optimized casting methodologies (complete)

Identified ideal substrate to cast 10 micron films and transfer on a porous support (complete)



Developed crosslinking chemistry to improve the mechanical as well as gas separation properties (complete)

Optimize UV-crosslinking chemistry (complete)

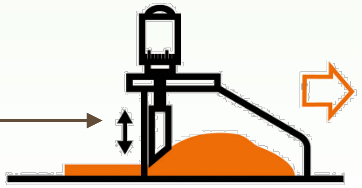
Task 5.1 and 5.2. Polymer Synthesis and Gen 1 ICE Membrane Fabrication (Membrane casting)

Hand casting

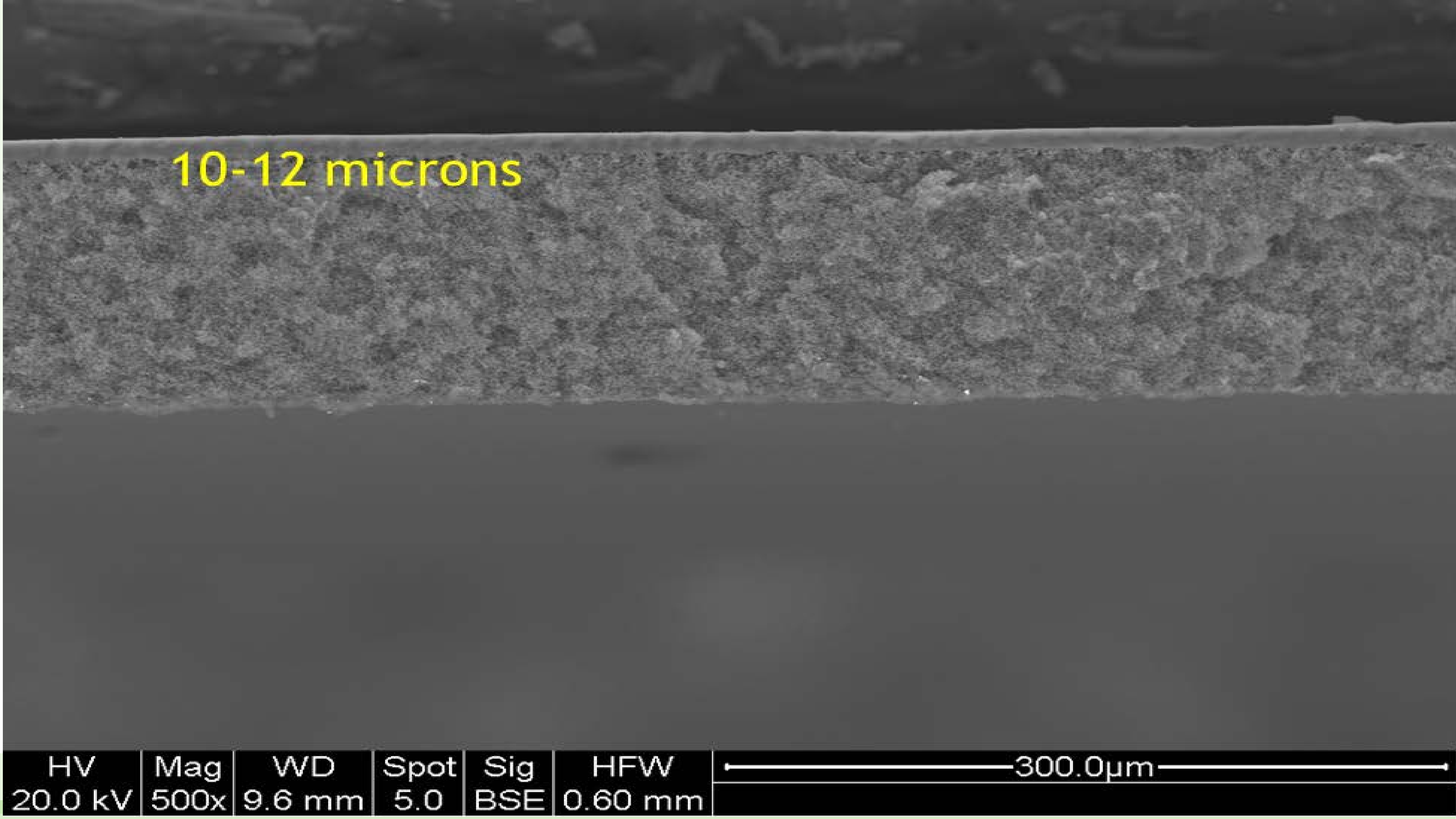
Screening is done by casting films on hand

Optimized support so the polymer can be released efficiently and transferred over a porous support

Knife



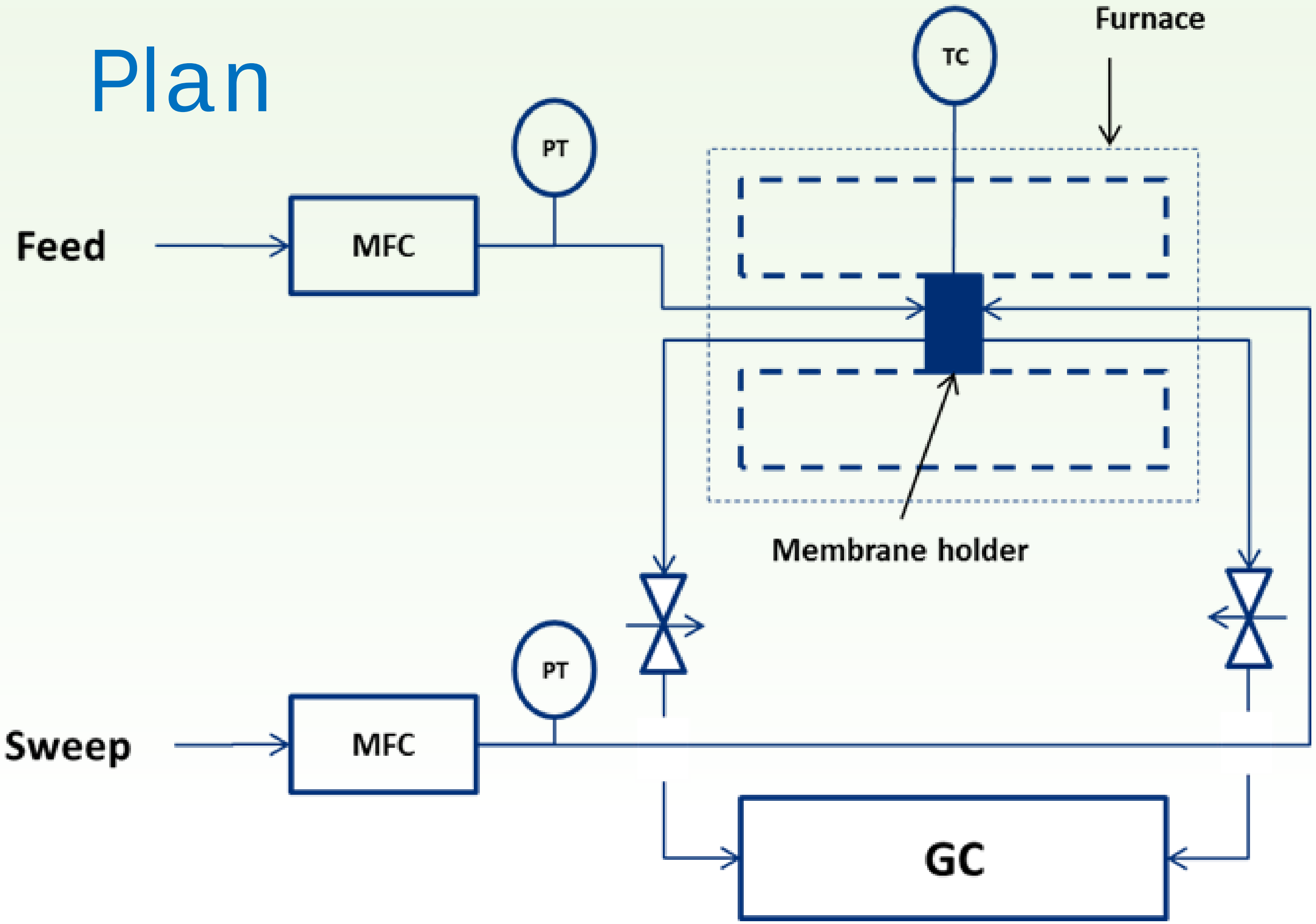
Polymer dope



10-12 microns
HV
20.0 kV
Mag
500x
WD
9.6 mm
Spot
5.0
Sig
BSE
HFW
0.60 mm
300.0μm

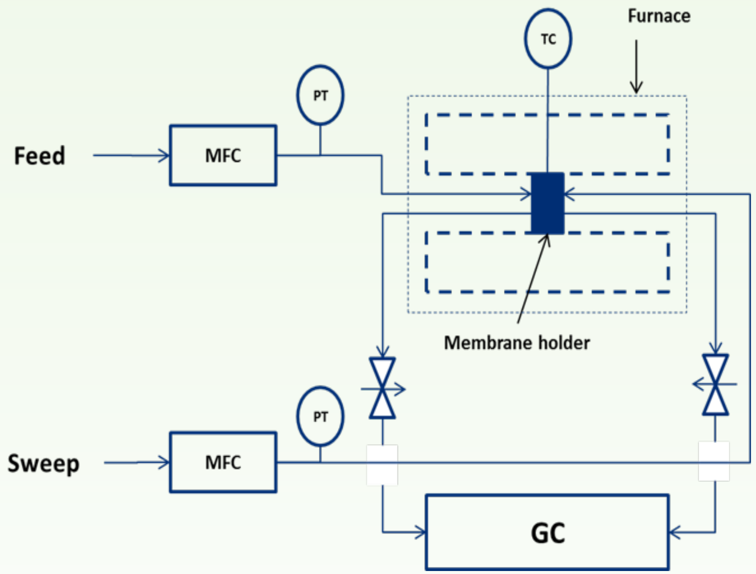
Tasks 5.3 and 5.4. Construction of Mixed Gas Testing Systems and Performance Testing

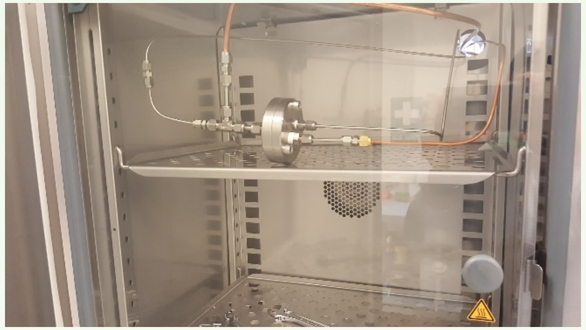
Plan

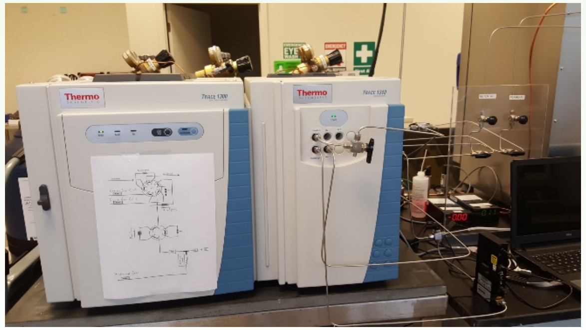


- Mixed Gas Selectivities
- Testing in Presence of Moisture
- Testing in Presence of Contaminants

Tasks 5.3 and 5.4. Construction of Mixed Gas Testing Systems and Performance Testing

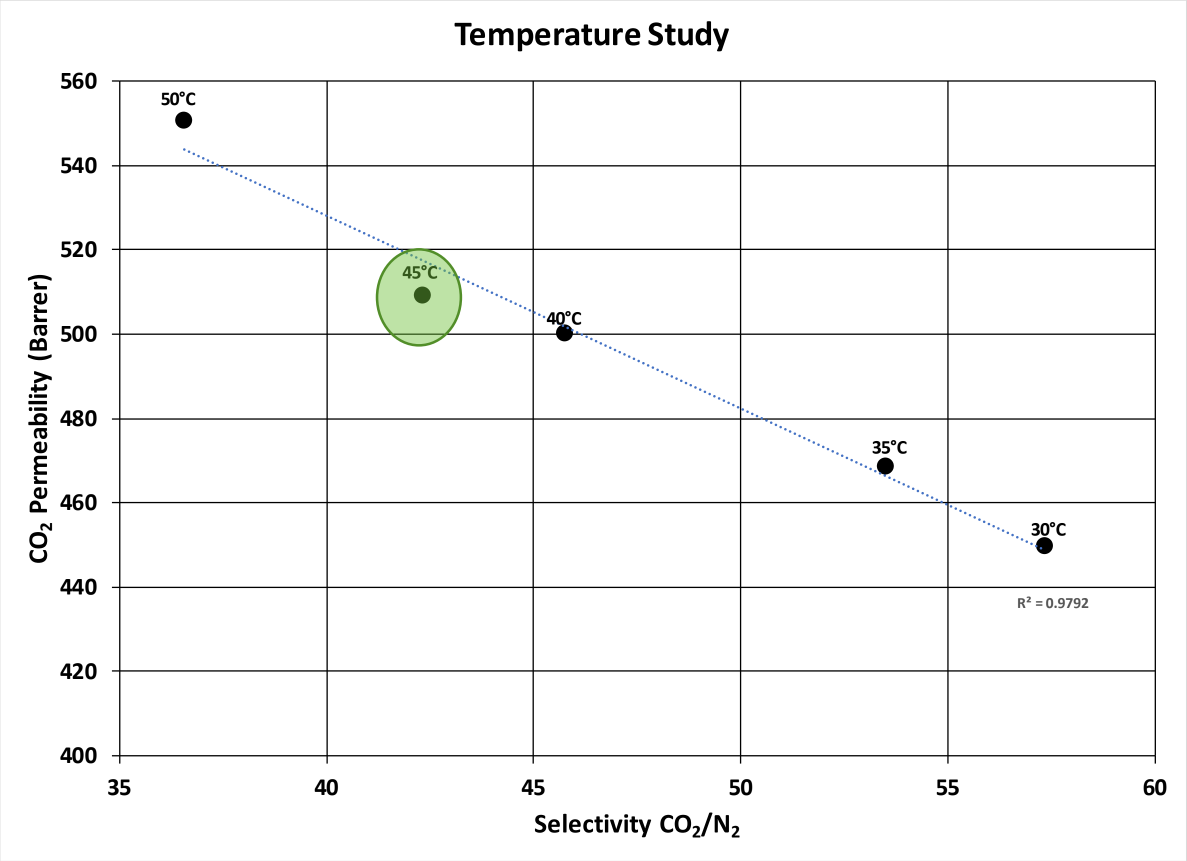






- Mixed Gas Selectivities
- Presence of Moisture (BP-3)

Tasks 5.3 and 5.4. Construction of Mixed Gas Testing Systems and Performance Testing

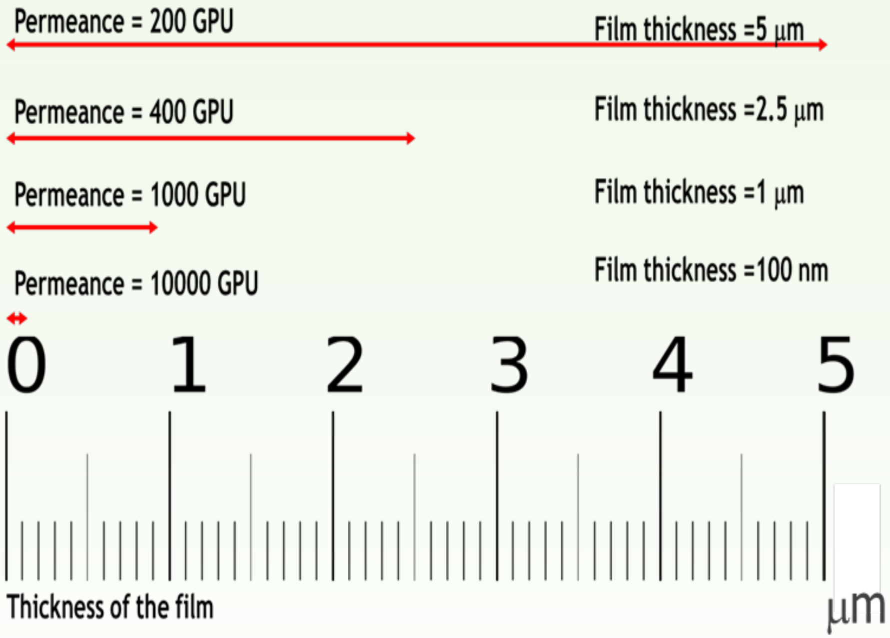


- Shows linear behavior for selectivity Vs. permeability
  - GEN-0
- Higher selectivity @ lower temperature
- Higher permeability @ higher temperature

Tasks 5.3 and 5.4. Construction of Mixed Gas Testing Systems and Performance Testing

%wt. Loading of Nanoparticles	Cast number	Characterization	Membrane results	
			Permeability	Selectivity
30% unmodified particles	LS-01-45A	Turned into a gel with white precipitates (not useable)	N/A	N/A
10% surface modified 10 nm particles	LIS-01-41 A	SEM, TGA, DSC, Membrane testing	659	41
20% surface modified 10 nm particles	LS-01-51 B*	Membrane testing	675-1025	20-33
40% surface modified 10 nm particles	**LIS-01-41 B, LIS-01-43**	**SEM, TGA, DSC, membrane testing**	**1609**	**44**
60% surface modified 10 nm particles	LIS-01-51A*	Membrane testing	250-400	25-30

Permeance Vs. Permeability



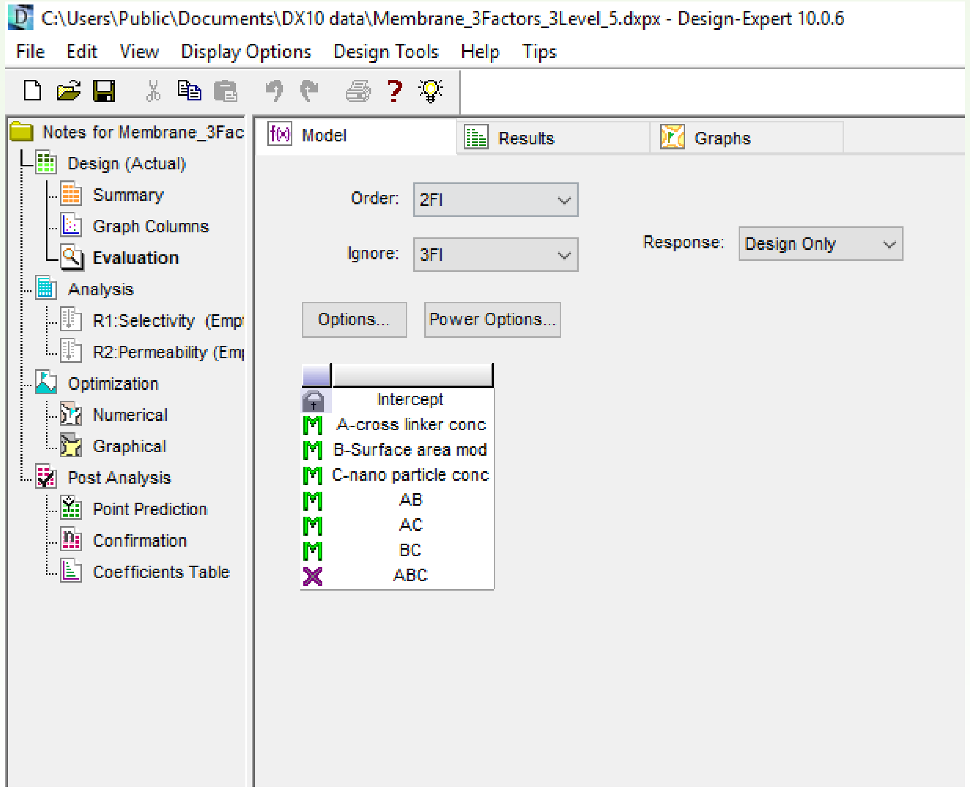
The effect of the film thickness on the permeance of the membrane. The scale bar is in microns to illustarte the point of perbeamity and permeance. The permeance numbers are based on a membrane material with the permeability of 1000 Barrer.

- Current state of the art membrane materials @ 250 permeability with selectivity of 35-45.
- These are casted @ 100nm giving permeance of 2500 GPU.

- Our best membrane has permeability of 1600 Barrers.
- Theoretically a membrane of 100 nm would result in permeability of 16000 GPU.
- A membrane of half a micron would result in permeance of 3200 GPU.

Task 5.1 and 5.2. Polymer Synthesis and Gen 1 ICE Membrane Fabrication

- Further optimization of membrane composition (In progress)
  - Design of Experiment matrix is being constructed on the following:
    - Optimized surface modification of the nano-particles
    - Optimized concentration of nan-particles
    - Optimized level of crosslinking
  - Control over nano-particle synthesis(complete)
  - Control over Polymer synthesis (complete)
    - Detailed studies of thermal characterization of the membranes



Using statistical analytical tools to optimize membrane composition

Task 6. Modification, Installation, and Testing of Air Stripper

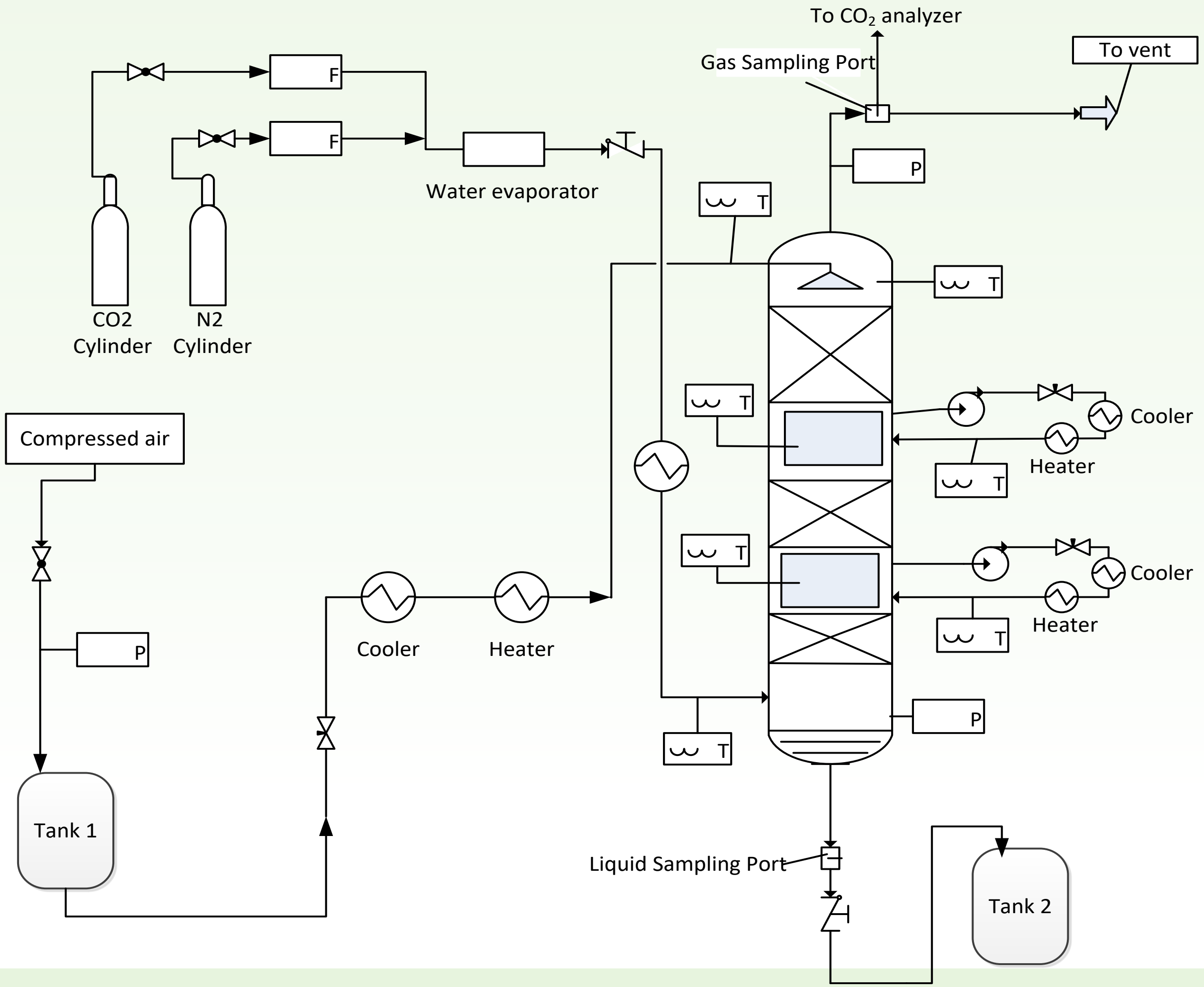
- Subtask 6.1 –Modification of Air Stripper
  - Complete modifications of the CO_2 air Stripping column have been successfully completed on schedule before March 31, 2017 and an associated milestone was achieved
- Subtask 6.2 – Installation of Air Stripper
  - Successfully completed by June 15, 2017.
- Subtask 6.3 – Parametric Testing of Air Stripper
  - have been in good progress and preliminary results have been obtained, which shows that the lean loading used in absorption tests can be achieved from the air stripping tests at an anticipated G/L ration and stripping temperature
  - The second milestone associated with subtask 6.3, "Complete parametric testing" is in good progress and will be completed on scheduled before September 30, 2017.

Task 6. Modification, Installation, and Testing of Air Stripper

Subtask 6.1 –Modification of Air Stripping System

Air stripping system modifications

- The schematic of air stripping column testing system, which is composed of:
  - Air stripping column with solvent circulation system for inter-stage heating.
  - Solvent feeding and discharging.
  - Gas feeding and discharging.
  - Temperature Control System .

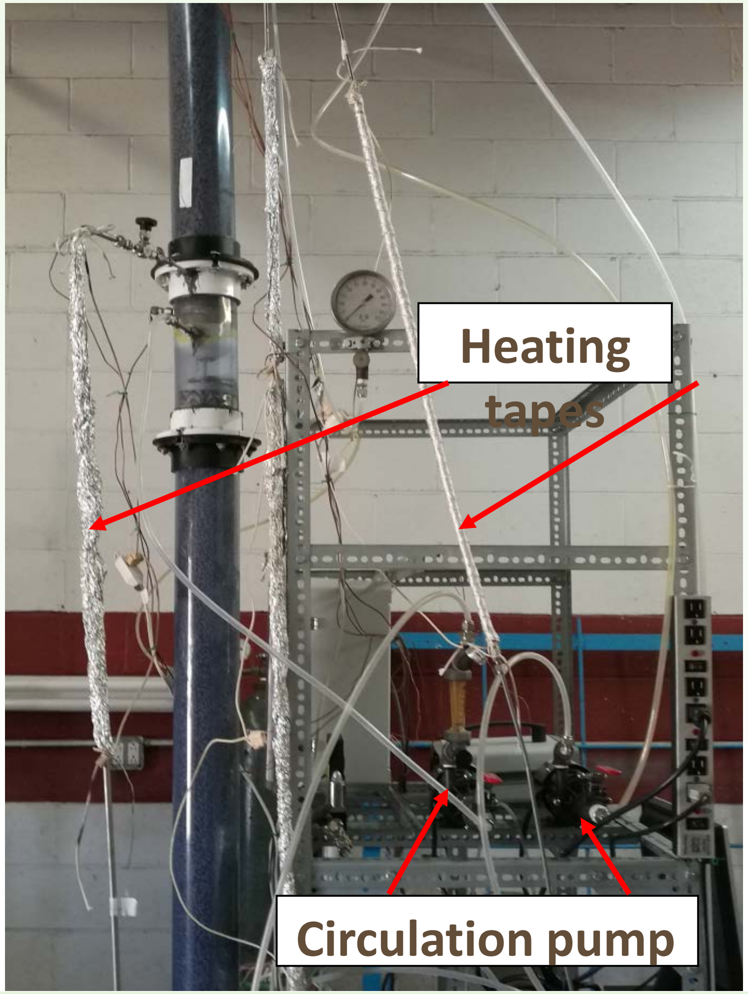


Task 6. Modification, Installation, and Testing of Air Stripper

Subtask 6.1 –Modification of Air Stripper

Air stripping column:

- 4 inch ID
- 4 mm θ ring random packing
- Total packing height of 96 inch
- Three packing sections with height of 21, 29 and 46 inches respectively
- Inter-stage heating loops between first/second and second/third packing section

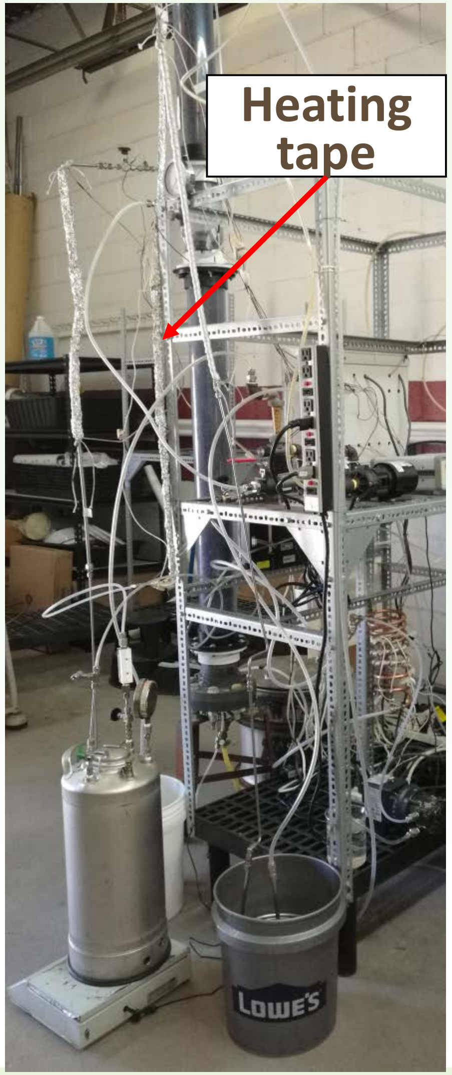


Task 6. Modification, Installation, and Testing of Air Stripper

Subtask 6.1 –Modification of Air Stripper

Solvent feeding System:

- 5 gallon solvent tank
- Tank was pressurized by compressed air
- Solvent goes through a flow meter and pre-heater to the stripper
- Solvent flow rate was calibrated by a scale

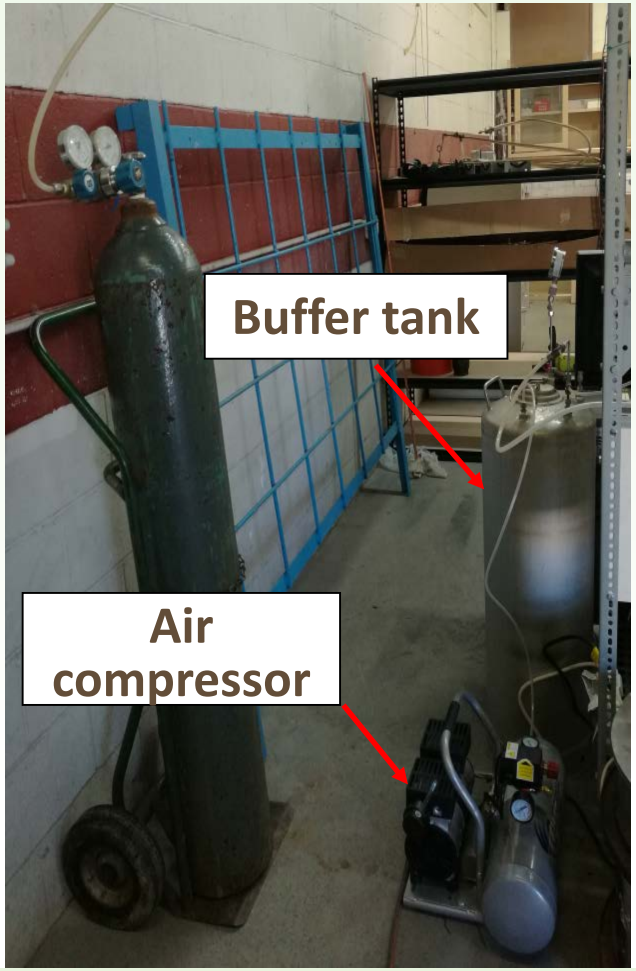


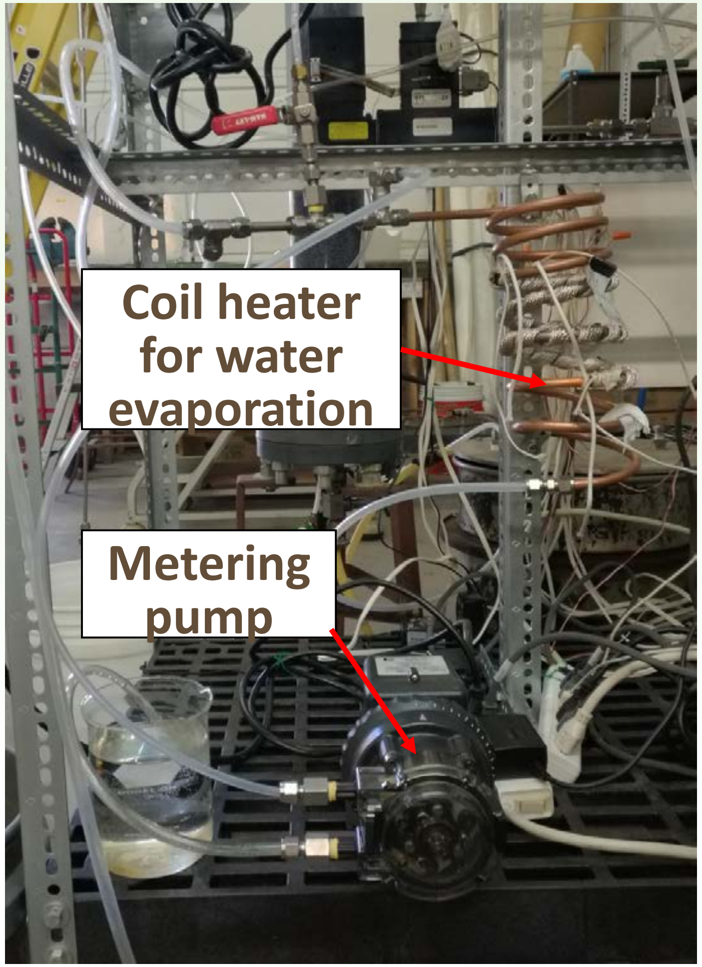
Task 6. Modification, Installation, and Testing of Air Stripper

Subtask 6.1 –Modification of Air Stripper

Air feeding system:

- An oil-free air compressor
- A 15 gallon buffer tank
- A water saturation system
- Air goes through air flow meter





Task 6. Modification, Installation, and Testing of Air Stripper

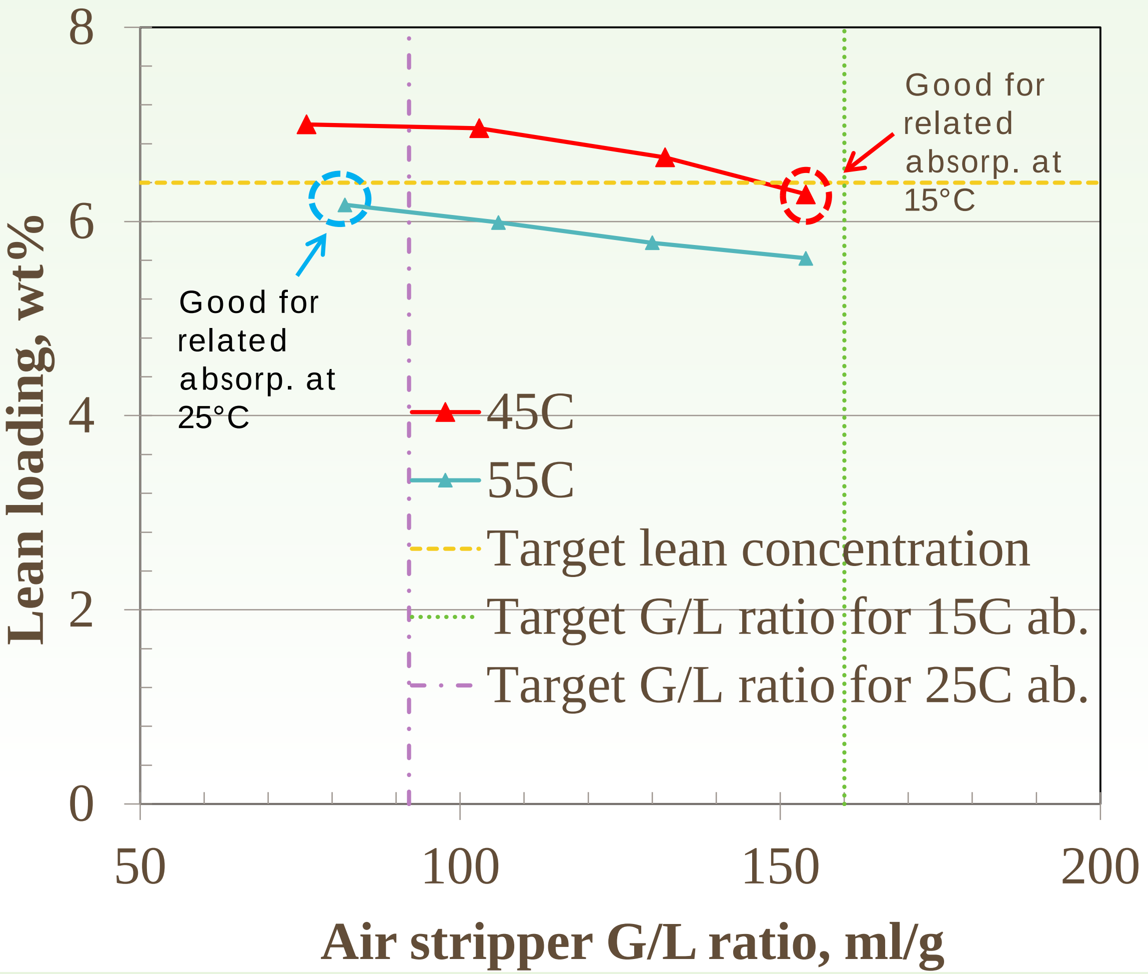
Subtask 6.2 – Installation of Air Stripper

- Shakedown tests with air and water
  - Performed leakage test to identify and repair leakage with epoxy welding. The leakage issue was resolved by mid of May 2017.
  - Modified the inter-stage heating units by replacing the cooling element with AC voltage transformer for better temperature control.
  - Established an alternative device for rich solvent preparation to expedite CO_2 loading in the lean solvent.
  - Established an independent solvent circulation bypass system with intensified heating capacity to overheat the lean solvent for packing material warm up.
- Shakedown tests with air and amine solvent
  - Detected and fixed any leaking points.
  - Calibrated air flow meter and CO_2 analyzer to obtain related working curves.
  - Improved the CO_2 loading measurement method by eliminating the pressure difference.

Task 6. Modification, Installation, and Testing of Air Stripper

Subtask 6.3 – Parametric Testing of Air Stripper (initial results)

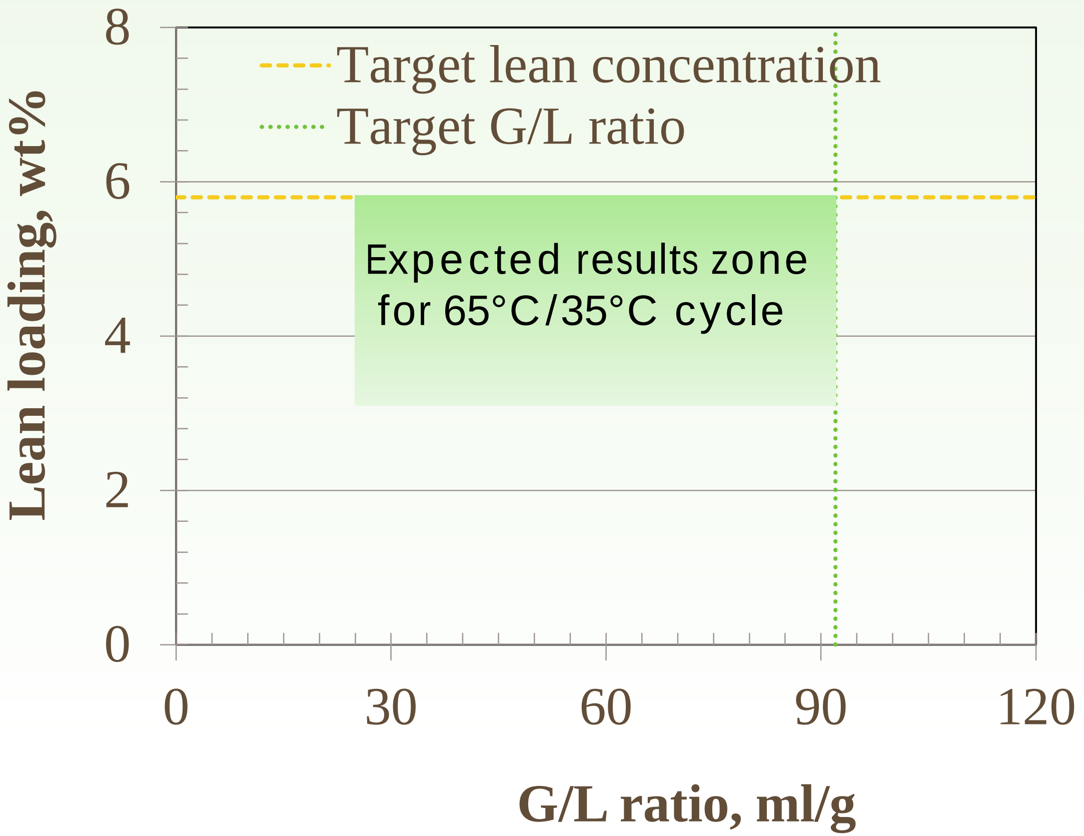
- Investigation of stripper operation parameters to regenerate solvent to targeted lean CO_2 loading
- Based on absorption tests in Task 4, the targeted lean loading and maximum air stripper G/L (air/solvent) ratio are:
  - 6.4wt% of lean loading for absorption at 15°C and 25°C
  - 160 ml/g G/L ratio correspond to 100% secondary air for 15°C absorption
  - 92 ml/g G/L ratio correspond to 100% secondary air for 25°C absorption
- Targeted lean loading and G/L ratio can be achieved with both 45°C/15°C and 55°C/25°C cycles
- For 55°C/15°C cycle, target lean loading can be achieved with as low as 58% of secondary air



Task 6. Modification, Installation, and Testing of Air Stripper

Subtask 6.3 – Parametric Testing of Air Stripper (continued)

- Further tests to be done include:
  - Air stripping test for 65°C/35°C cycle.
  - Air stripping at smaller G/L ratio for other cycles
- Expected results include:
  - For 65°C/35°C cycle, expected 5.8wt% of targeted lean loading (from 35°C absorption) by 65°C air stripping with G/L< 92 ml/g (corresponding to 100% secondary air)
  - Identify different air stripper operating conditions of cycles, include:
    - Air striping temperature
    - G/L ratio



Milestones: BP 2

- ✓ Fabricate 3 Gen 1 ICE Membranes – 3/31/2017
- ✓ Modify Air Stripping Column – 3/31/2017
- Select Up to Two Best Gen 1 Membranes – 9/30/2017
- ✓ Construct Isobaric Performance System – 9/30/2017
- Complete Parametric Testing of Air Stripping Column – 9/30/2017

Success Criteria: BP 2

- Completion of All Work Proposed
- Best Gen 1 ICE Membrane Achieves 2500 GPU Permeance and 25 CO_2/N_2 Selectivity
  - Current state of the ICE Gen-1 membrane is @ 1600 Barrer with selectivity of 44. This membrane @ a thickness of half a micron would result in permeance of 3200 GPU with a selectivity of 44.
- Successful Test of Air Stripping Column with Difference in Absorption and Stripping Temperatures of No More than 50^oC
- Submission of Continuation Application

Budget Status (BP2)

Description	BP2 budget	Actual spending June 2017	Remaining budget	Projected expenditure (BP2)	varian ce	%
Personnel	166,200	120,990	45,210	30,600	15,210	9%
Fringe Benefits	49,860	41,621	8,239	10,526	-2,425	-5%
Travel	4,990	4,952	38	1,500	-1,462	-29%
Equipment	76,100	15,438	60,662	40000	20,662	27%
Supplies	10,000	13,797	-3,797	1500	-5,349	-53%
Other direct cost	-					
Sub-recipient	407,564	328,147	79,417	105,000	-2,053	-6%
Total Direct Costs	714,714	524,944	189,770	189126	-1,856	0%
Cost matching	164,210	123,698	40,512	44,558	7,064	-2%
Indirect Charges	106,336	123698	12,791	33,664	-20,657	-20%
Total Project Costs	821,050	618,489	243,072	267,349	-24,276	-3%

Budget Period 3

Tasks for Budget Period 3 (2018)

- Task 7: ICE Membrane Scale-up and Simulated Flue Gas Testing
  - 7.1: Polyphosphazene Scale-up and Characterization
  - 7.2: Fabrication of Optimized Generation 1 ICE Membrane
  - 7.3: Simulated Flue Gas Testing
- Task 8: Initial Technical and Economic Feasibility Study
  - 8.1: System Performance Modeling
  - 8.2: Economic Analysis
  - 8.3: Sensitivity Analysis

Task 7. ICE Membrane Scale-up and Simulated Flue Gas Test

- Phosphazene Scale-up and Characterization
  - Optimize synthesis to minimize the batch to batch variations
  - Scale-up in the following order 100g and multiple 250g scales. (up to 1kg)
  - Characterization of polymers and compare with variations with batch to batch.
- Scale-up and Characterization of Gen 1 ICE Membranes
  - Perform detailed thermal characterization of the Gen 1 Ice membrane.
    - TGA and DSC and correlate with membrane performance data
  - Long-term thermal stability tests of the membranes and effect on membrane.
  - Cast 10x10 inch by 10 micron thickness films for the above study
- Simulated Flue Gas Testing
  - Isochoric Membrane Test System Will Be Upgraded to Handle Moisture
  - Selected Gen 1 ICE Membranes Will Be Tested in the Presence of Flue Gas Contaminants

Task 8. Initial Technical and Economic Feasibility Study

- System Performance Modeling
  - Hybrid process performance simulation
  - Heat pump cycle simulation
- Economic Analysis
  - Major equipment sizing and capital cost.
  - Electrical cost calculation
- Sensitivity Analysis
  - Sensitivity study of selected variables
  - Critical variables identification

Milestones: BP 3

- Model Performance and Conduct Mass and Energy Balances for Hybrid Process – 3/31/2018
- Scale-up Optimal Polyphosphazene to 1 kg – 6/30/2018
  - NMR, thermal and membrane performance data will be used to determine the consistency with small scale synthesis.
  - A 10% variance in membrane performance is acceptable.
- Conduct Simulated Flue Gas Testing of Gen 1 ICE Membrane – 9/30/2018
  - Simulated flue gas testing (containing CO_2, N_2, O_2, H_2O and SO_2)
  - Membranes used will be of @ 10 micron thickness of the optimized polymer composition material.
- Complete Technical and Economic Feasibility Study and Sensitivity Analysis – 9/30/2018

Success Criteria: BP 3

- Completion of All Work Proposed
- Scale-up Polymer Synthesis to 1 kg.
  - With the goal to have same chemical composition as the small batch (using NMR spectroscopy, GPC, and gas performance testing)
- Best Gen 1 ICE Membrane Achieves 5000 GPU Permeance and 30 CO_2/N_2 Selectivity
  - Cast thin membranes (half a micron) with the best ICE Gen-1 candidate to achieve 5000 GPU with a 30 CO_2/N_2 selectivity.
- Economic Analysis Shows Significant Progress toward Achieving a 30% Reduction in Cost of Electricity
- Submission of State Point Data Tables
- Submission of Final Report

Budget: BP 3

CATEGORY	Budget Period 3 Costs
a. Personnel	$169,200
b. Fringe Benefits	$50,760
c. Travel	$4,990
d. Equipment	$0
e. Supplies	$10,000
f. Contractual	
Sub-recipient	$373,023
Contractor	$0
FFRDC	$0
Total Contractual	$373023
g. Construction	$0
h. Other Direct Costs	$0
Total Direct Costs	$607973
i. Indirect Charges	$100481
Total Cost Share	$141,691
Total Project Costs	$708,454

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Questions?