Automated Post-combustion Flue Gas Membrane Performance Testing Skid at the National Carbon Capture Center
Outline

• The NETL Post-combustion Membrane Testing Skid (PCMS)
• Lessons learned
• Initial testing results
• Ongoing testing
• Future outlook
Our home at the PC4 Bench Scale Facility
Our home at the PC4 Bench Scale Facility
Post-Combustion Membrane Testing Skid

• The proving ground for our most promising membranes
• Compact footprint: can be moved as one unit
• Fully automated operation: set and forget
  – Designed for multiday/multiweek experiments for medium/long term testing
**Post-Combustion Membrane Testing Skid**

- **Based on standard constant pressure membrane testing system**
  - Mixed gas analysis using gas chromatograph
  - Pressurizes feed up to 30 psig, filters particulate matter, and dehumidifies feed to avoid condensation
  - Accepts hollow fiber modules and flat sheet modules

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**Flue**
- Chiller
  - MFC
    - HMT
      - PT
        - FI
          - ~2 slm feed bypass
  - MFC
  - Argon
  - Pumped to ~30 psig
  - +7 µm filters

**Primary membrane module**
- Water bath temp. control
  - 5-80 °C

**PTC**
- GC
  - 15-30 psia
Software and automation

- Embedded Windows 7 rugged computer/DAQ with custom LabVIEW-based software
- Enables “setup, start and walk away” operation philosophy
- Up to 5 pre-programmed pressure/flow steps
- Automated GC injections for mixed gas analysis from either feed or sweep
Membrane assembly

Flat sheet membrane assembly

Typical area: 0.6 to 2.7 cm²
Cross-current flow

Hollow fiber membrane assembly
(Single strand)

Typical length: 10 cm
Counter-current flow
December 2015 campaign timeline

- **Primary task: install and confirm PCMS operating properly**
  - Extensive tests at NETL minimizes installation and shakedown time at NCCC
- **Secondary task: test membranes (24-hour tests)**
  - 7 membranes successfully tested: 4 hollow fibers, 3 dense flat sheet membranes
December 2015 feed characteristics

PCMS-analyzed feed composition matches well with NCCC’s

Feed dewpoint depends on outside temperature (reduced by feed chiller to prevent condensation)
Reference membrane: PDMS

- **Commercial polydimethylsiloxane film**
- **Stable performance:**
  - ~3300 barrer CO₂
  - CO₂/N₂ around 8.5
  - O₂/N₂ around 1.9
  - All the above numbers agree with lit. data
- **Insufficient flowrate to measure water vapor permeability**
  - Around 16k-18k barrer
  - H₂O – much lower than literature ref. (40k)
  - 10 sccm not enough to avoid conc. polarization
Gen 1 mixed matrix coated hollow fibers

- Flat sheet TFE-PPZ/SIFSIX-2Cu₁ (not tested at PCMS)
- High surface porosity Torlon hollow fibers
- HF-1 (TFE-PPZ/30 wt% SIFSIX-2Cu₁ coating)

Polymer:

$$\begin{array}{c}
\text{OCH}_2\text{CF}_3 \\
\text{N} = \text{P} \\
\text{OCH}_2\text{CF}_3 \\
n
\end{array}$$

poly(bistrifluoroethoxy)phosphazene (TFE-PPZ)
Mixed matrix coated hollow fiber membranes

- PPZ/SIFSiX mixed matrix coated hollow fiber (HF-1)
  - Slight decline in all gas permeance over 22h: CO₂ from ~75 GPU to ~70 GPU
  - Insufficient time to determine decline significance

All hollow fibers are assumed: length = 10 cm, outer diameter = 540 µm
• **Three membranes planned this season**
  – Focus on medium term tests (3-4 weeks) to gain operational experience
  – Whenever available, test thin film hollow fiber composite membranes
  – One three-week test successfully concluded on a free-standing film
  – Ongoing testing on membrane #2
PDMS re-run to verify operation

- Confirmed PCMS still working after 6 months downtime
- Confirmed CO₂, O₂, N₂ results against previous run
- Higher apparent H₂O permeability (25k-28k barrer) with 50 sccm feed flowrate
- Hourly sampling frequency
Conclusions

- **We have an operational membrane testing skid at NCCC**
  - Versatile for testing different membrane formats
  - Short term testing gives data well corroborated with literature values and/or other testing equipment at NETL
  - Sufficient feed flowrate is required to quantify water vapor permeance accurately

- **Short term membrane performance at low humidity + contaminants consistent with performance under dry, ideal conditions**

- **The PCMS can be operated unattended for longer periods of time**
  - We have completed a three-week unattended test
  - The skid can tolerate several process upsets (e.g. temporary loss of feed flue gas)
  - Gas chromatograph maintenance imposes the biggest limitation on testing length
Future plan

• **Finish current testing season at NCCC**
  – Up to two additional membranes to be tested (4 week runs)
  – Upgrade feed flowrate capability to measure water vapor permeance

• **Design improvements**
  – More efficient water vapor removal to protect GC columns
  – Flow components to accommodate small multi-fiber modules

• **Continue developing membranes with focus on thin film composite hollow fiber membranes**
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