Pilot-Scale Development of a PEEK Hollow Fiber Membrane Contactor Process for Post-Combustion CO$_2$ Capture

DOE Contract DE-FE0012829

Shiguang Li, Travis Pyrzynski, Naomi Klinghoffer, Timothy Tamale, James Aderhold, S. James Zhou, and Howard Meyer, *Gas Technology Institute (GTI)*

Yong Ding and Ben Bikson, *Air Liquide Advanced Separations (ALaS)*

Katherine Searcy, Andrew Sexton, *Trimeric*

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Project overview

- **Performance period**: Oct. 1, 2013 – June 30, 2018
- **Total funding**: $13.7MM (DOE: $10.6MM, Cost share: $3.1MM)
- **Objectives**:
  - Build a 0.5 MWₑ pilot-scale CO₂ capture system and conduct tests on flue gas at the National Carbon Capture Center (NCCC)
  - Demonstrate a continuous, steady-state operation for ≥ 2 months
- **Goal**: achieve DOE’s goal of 90% CO₂ capture rate with 95% CO₂ purity at a cost of $40/tonne of CO₂ captured by 2025

**Team**:

<table>
<thead>
<tr>
<th>Member</th>
<th>Roles</th>
</tr>
</thead>
<tbody>
<tr>
<td>gti</td>
<td>• Project management and planning</td>
</tr>
<tr>
<td></td>
<td>• Process design and testing</td>
</tr>
<tr>
<td>ALaS</td>
<td>• Membrane and module development</td>
</tr>
<tr>
<td>Porogen</td>
<td>• Techno-Economic Analyses (TEA)</td>
</tr>
<tr>
<td>Trimeric Corporation</td>
<td>• Site host</td>
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<tr>
<td>NCCC</td>
<td></td>
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</tbody>
</table>
Timeline and scope

**Current in BP2:**
- Funding: DOE: $5.6MM; Cost share: $1.0MM
- Delivery: 0.5 MW_e (10 tonne/day) pilot plant
What is a membrane contactor?

- High surface area membrane device that facilitates mass transfer
- Gas on one side, liquid on other side

- Membrane does not wet out in contact with liquid
- **Separation mechanism**: CO$_2$ permeates through membrane, reacts with the solvent; N$_2$ does not react and has low solubility in solvent
Process description

PEEK = polyether ether ketone
HFMC = hollow fiber membrane contactor
Technical challenges of applying HFMC to existing coal-fired plants

- **Performance** – Overall mass transfer resistance consists of three parts
  - Minimize each resistance
- **Durability** – Long-term membrane wetting in contact with solvent may affect performance
  - Make membrane surface super hydrophobic
  - Improve membrane potting to provide good seal between the liquid and gas sides
- **Scale-up and cost reduction**
  - Make larger diameter modules

\[
\frac{1}{K} = \frac{1}{k_g} + \frac{1}{k_m} + \frac{H_{adim}}{E \cdot k_l}
\]

- Overall mass transfer coefficient \( K \) (cm/s)
  - In the gas phase, \( k_g \)
  - In the membrane, \( k_m \)
  - In the liquid phase, \( k_l \)

\( H_{adim} \): non-dimensional Henry’s constant
- \( E \): enhancement factor due to reaction

GPU = Gas Permeation Unit
PEEK (\[
\begin{array}{c}
\text{O} \\
\text{C} \\
\text{C} \\
\text{O} \\
\end{array}
\]) membrane characteristics

- Exceptional thermal & mechanical resistances

<table>
<thead>
<tr>
<th>Polymer</th>
<th>Tensile modulus (GPa)</th>
<th>Tensile strength (MPa)</th>
<th>Max service temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Teflon™</td>
<td>0.4-0.5</td>
<td>17-21</td>
<td>250</td>
</tr>
<tr>
<td>PVDF</td>
<td>0.8</td>
<td>48</td>
<td>150</td>
</tr>
<tr>
<td>Polysulfone</td>
<td>2.6</td>
<td>70</td>
<td>160</td>
</tr>
<tr>
<td>PEEK</td>
<td>4</td>
<td>97</td>
<td>271</td>
</tr>
</tbody>
</table>

- Hollow fibers with high CO₂ flux, and thus high packing density and small equipment size

- Surface modified to be super hydrophobic

- Good chemical resistance to amine
  - Exposure of fibers to MEA solution (30%) at 120°C for 1,500 hours had no adverse effect on the mechanical or gas permeation properties

Hydrophilic \(\rightarrow\) Hydrophobic

Ethanol
## Preliminary TEA based on bench-scale field tests: HFMC costs 16% less than Case 12

<table>
<thead>
<tr>
<th>Item</th>
<th>Unit</th>
<th>DOE benchmark technology amine plant (Case 12)</th>
<th>PEEK HFMC field test data*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Increase in LCOE</td>
<td>%</td>
<td>69.6</td>
<td>57.0</td>
</tr>
<tr>
<td>Cost of CO₂ capture</td>
<td>$/tonne</td>
<td>56.47</td>
<td>47.53</td>
</tr>
</tbody>
</table>

* Bench-scale field tests with 4-inch-diameter module and aMDEA solvent: mass transfer coefficient of 1.2 (sec)⁻¹ at 93.2% CO₂ removal

## R&D strategy to meet DOE’s cost target ($40/tonne by 2025)

- Increase mass transfer coefficient from 1.2 to 2 (sec)⁻¹: $42.48
- Advanced solvents/new regeneration process design: < $40.00

*aMDEA = Activated methyl diethanolamine  
LCOE = Levelized Cost Of Electricity*
Intrinsic CO$_2$ permeance of the PEEK fiber improved to 2,500 GPU

1 GPU = 1 x 10$^6$ cm$^3$ (STP)/cm$^2$ • s • cmHg
Delamination of the fiber/epoxy interface observed during startup/shutdown tests, membrane potting improved recently.

Old modules: clear color differentiation between the epoxy and the fiber; the epoxy surrounds the fiber rather than infusing into the fiber.

Recent modules: fibers and epoxy are the same color; epoxy penetrated into the wall of the fibers.
Recent modules showed good startup/shutdown stability
Module scaled to 8-inch, which was tested at GTI with aMDEA solvent using air/CO₂ mixed gas as feed

Improved mass transfer coefficient of 2.0 (sec.)⁻¹ obtained
Lab parametric tests: CO$_2$ flux and capture rate increase with increasing feed pressure, solvent velocity and temperature.
Planning tests at the NCCC

- EPIC System was down selected from five bidders to construct the 0.5 MWₑ plant due to their:
  - Reasonable bid in terms of costs and technical approaches
  - Experience at NCCC
  - Modular construction experience
  - Experience with membrane skid design
- Detailed engineering and HAZOP completed
- Testing site (Pilot Bay 2 West) and layout of skids determined
Pilot plant for 0.5 MW_e (10 tonne/d) CO_2 capture

12 m (L) x 7.5 m (W) x 3.5 m (H) + a blower
Plant is under construction

Skids: frames complete, all equipment received, components are being installed

Control system: 75% complete
## BP2 milestones and overall testing schedule

<table>
<thead>
<tr>
<th>Milestone No.</th>
<th>Milestone Description</th>
<th>Planned Completion</th>
<th>Actual Completion</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Submit Budget Period 1 Report</td>
<td>11/30/15</td>
<td>12/23/15</td>
</tr>
<tr>
<td>2</td>
<td>Complete 8-inch module fabrication</td>
<td>12/31/15</td>
<td>10/09/15</td>
</tr>
<tr>
<td>3</td>
<td>Technical information for 8-inch module delivered</td>
<td>01/31/16</td>
<td>01/28/16</td>
</tr>
<tr>
<td>4</td>
<td>Complete initial solvent process determination</td>
<td>02/29/16</td>
<td>02/25/16</td>
</tr>
<tr>
<td>5</td>
<td>Achieve ≥90% CO₂ removal, contactor mass transfer coefficient ≥1.7 (sec)^{-1} in 8-inch modules</td>
<td>04/30/16</td>
<td>03/31/16</td>
</tr>
<tr>
<td>6</td>
<td>Complete procurement for the 0.5 MWₑ system</td>
<td>05/30/16</td>
<td>04/30/16</td>
</tr>
<tr>
<td>7</td>
<td>Complete construction of the 0.5 MWₑ pilot system</td>
<td>09/30/16</td>
<td>EPIC scheduled to complete by 9/30/16</td>
</tr>
</tbody>
</table>

### Overall testing schedule at NCCC

- **Plant fabrication and acceptance testing completed**
  - Sept. 2016

- **Plant delivered to NCCC**

- **Installation/shakedown completed**
  - March 2017 (PO-6)

- **Parametric testing completed**
  - Sept. 2017 (PO-7)

- **Long-term testing completed**
  - March 2018 (PO-8)
PEEK HFMC-based technology development path

Year


Scale

PoroGen Lab scale

DE-FE-0004787 (bench-scale, 25 KW)

DE-FE0012829 (small-pilot scale 0.5 MW)

Future 10 MWₑ large pilot scale

Future 100 MWₑ demonstration
Summary

- Preliminary TEA: PEEK HFMC costs 16% less than Case 12, can be furthered reduced by improving contactor performance.
- Intrinsic CO₂ permeance of the fiber improved to 2,500 GPU.
- Wicking of solvent at fiber/epoxy interface observed during startup/shutdown tests, membrane potting improved recently.
- Module scaled to 8-inch diameter, lab tests showed mass transfer coefficient of 2.0 (sec⁻¹) with aMDEA solvent.
- EPIC System was selected to construct the 0.5 MWₑ plant.
- Detailed engineering and HAZOP completed.
- Pilot plant construction to be completed by 9/30/16, testing at NCCC planned.
Acknowledgements

- Financial support

- DOE NETL José Figueroa