



**National Carbon Capture Center Test Report:**  
**Membrane Module Testing at NCCC**

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CO<sub>2</sub> Capture from <1% CO<sub>2</sub> Sources

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## Executive Summary

The objectives of this project are to develop a cost-effective design and manufacturing process for new membrane modules that capture CO<sub>2</sub> from flue gas in coal-fired power plants. Using the modules for post-combustion CO<sub>2</sub> capture is expected to achieve the DOE target of <\$40/tonne CO<sub>2</sub> captured for 2025. The field test conducted at the National Carbon Capture Center (NCCC) in Wilsonville, AL was based on the testing collaboration agreement between The Ohio State University (OSU) and NCCC. The main task of the field test was to evaluate the spiral-wound membrane module performance with the actual flue gas produced at NCCC as the feed gas. Particularly, the effect of the impurities in the actual flue gas, such as O<sub>2</sub>, SO<sub>2</sub>, NO<sub>x</sub>, was investigated. Additionally, a long-term (500 hours) stability test of the spiral-wound membrane module was also conducted.

Each of the spiral-wound membrane elements was rolled with about 4" in diameter by 14.375" in length using four membrane leafs of 30" in length with a width of 14". The total membrane area was about 14,000 cm<sup>2</sup>. The membrane element was installed in a stainless-steel housing with a diameter of about 5" to constitute the membrane module. The module was placed inside the oven of a membrane testing unit, and the testing temperature was 57 – 67°C with a feed pressure of 1 – 4 atm and a vacuum of 0.2 – 0.3 atm on the permeate side. The OSU membrane module testing was performed in the Lab Scale Testing Unit (LSTU) building of NCCC from July 16<sup>th</sup> to August 17<sup>th</sup>, 2018. During this period, three spiral-wound membrane modules were tested. The flue gas composition was approximately 12% (± 1%) CO<sub>2</sub>, 7% (± 1%) O<sub>2</sub>, 81% (± 1%) N<sub>2</sub>, 0.5 – 1 ppm SO<sub>2</sub>, and 1.5 – 4 ppm NO<sub>2</sub> on dry basis; the flue gas under testing contained about 7.4% H<sub>2</sub>O for a feed pressure of 4 atm.

The three modules tested at NCCC showed repeatable results with an average of 1450 GPU (1 GPU = 10<sup>-6</sup> cm<sup>3</sup> (STP)·cm<sup>-2</sup>·s<sup>-1</sup>·cmHg<sup>-1</sup>) CO<sub>2</sub> permeance and 185 CO<sub>2</sub>/N<sub>2</sub> selectivity. Those results agreed well with the modules tested in our OSU lab using a simulated flue gas consisting of about 17.1% CO<sub>2</sub>, 68.5% N<sub>2</sub>, 7.4% H<sub>2</sub>O, 7% O<sub>2</sub>, and 3 ppm SO<sub>2</sub> at a feed pressure of 4 atm. The first module was tested for 500 hours and showed a stable performance at ~ 1450 GPU and ~ 185 CO<sub>2</sub>/N<sub>2</sub> selectivity at 67°C, 4 atm feed pressure and 0.3 atm permeate vacuum. At 57°C, 1 atm feed pressure and 0.2 atm permeate vacuum, the module showed a CO<sub>2</sub> permeance of 980 GPU and a CO<sub>2</sub>/N<sub>2</sub> selectivity of 164. At 57°C, 4 atm feed pressure and 0.2 atm permeate vacuum, the module showed a CO<sub>2</sub> permeance of 931 GPU and CO<sub>2</sub>/N<sub>2</sub> selectivity of 153 as a result of the carrier saturation phenomenon at the elevated feed pressure. This module showed a good 500-hour stability of performance, despite the disturbances such as low flue gas flow rates and two flue gas shutdowns. The second and third modules were tested to reproduce the results at 67°C, 4 atm feed pressure and 0.3 atm permeate vacuum. The second module was tested for 24 hours and showed a CO<sub>2</sub> permeance of ~ 1455 GPU and CO<sub>2</sub>/N<sub>2</sub> selectivity of ~ 175. The third module was tested for 30 hours and exhibited a CO<sub>2</sub> permeance of ~ 1450 GPU and CO<sub>2</sub>/N<sub>2</sub> selectivity of ~180. Therefore, the membrane module performance was reproducible consistently. There was no stability issue incurred by the impurities in the actual flue gas such as O<sub>2</sub>, SO<sub>2</sub>, and NO<sub>x</sub> during the field testing.

Overall, the membrane module testing at NCCC went smoothly, and the membrane module results achieved the same level of performance as compared to our OSU lab test results with the

simulated flue gas. In other words, the results showed that the modules tested at NCCC behaved similarly to those in the OSU lab, indicating the great potential of the membrane modules for CO<sub>2</sub> capture from flue gas in coal-fired power plants.

The smooth membrane testing at NCCC would not be possible without the great efforts of NCCC team members, particularly Tony Wu and Bob Lambrecht. We are grateful for their excellent analytical and mechanical supports, including the arrangements of lifting our membrane unit into the LSTU building, setting it up, connecting all tubing and electrical parts outside of our unit, installing air flow to our oven for accurate temperature control, and providing us the gas concentration data log. NCCC support and cooperation for our membrane module testing were wonderful and professional, and we truly enjoyed working with them.

# 1 Project Objectives and NCCC Test Objectives

## 1.1 Project objectives

The objectives of this project are to develop a cost-effective design and manufacturing process for new membrane modules that capture CO<sub>2</sub> from flue gas in coal-fired power plants. The membrane consists of a thin selective layer of a polymer structure so that it can be made in a continuous manufacturing process. It is incorporated in membrane modules (e.g., spiral-wound), for bench scale tests at simulated flue gas conditions. Using the modules for post-combustion CO<sub>2</sub> capture is expected to achieve the DOE target of <\$40/tonne CO<sub>2</sub> captured for 2025.

Currently, the project is in the Budget Period 2 (BP 2), which includes the fabrication of the scale-up membranes into three prototype membrane modules for continuous testing with simulated or real flue gas. In order to achieve the project objectives in BP2, the following tasks are defined:

- Task 8 – Optimized Prototype Membrane Fabrication
- Task 9 – Optimized Prototype Membrane Characterization
- Task 10 – Optimized Prototype Module Fabrication
- Task 11 – Optimized Prototype Module Characterization
- Task 12 – Construction of the Pilot Testing Unit
- Task 13 – Installation/Shakedown of the Pilot Testing Unit
- Task 14 – Module Testing with the Pilot Unit
- Task 15 – Final Techno-economic Analysis
- Task 16 – Quarterly Progress Reports
- Task 17 – Final Project Report

## 1.2 NCCC test objectives

The field test conducted at the National Carbon Capture Center (NCCC) at Wilsonville, AL was based on the testing collaboration agreement between The Ohio State University and NCCC and mainly related to Tasks 12, 13, and 14 of the project. The main task of the field test was to evaluate the spiral-wound membrane module performance with the actual flue gas produced at NCCC as the feed gas. Particularly, the effect of the impurities in the actual flue gas, such as O<sub>2</sub>, SO<sub>2</sub>, NO<sub>x</sub>, was investigated. Additionally, a long-term (500 hours) stability test of the spiral-wound membrane module was also conducted.

## 2 Technical Background

The average carbon dioxide level in the earth's atmosphere has increased from 280 ppm in the pre-industrial period to 379 ppm in 2005 [1]. A recent study estimates the present concentration at greater than 390 ppm [2]. The increase in CO<sub>2</sub> level is widely accepted as the biggest contributor to increasing global temperatures [1,3]. The report of the Intergovernmental Panel on Climate Change clearly attributes the major cause of CO<sub>2</sub> increase to anthropogenic fossil fuel use [1].

According to the International Energy Agency report on world energy statistics, coal accounted for 25% of the world electricity and 42% of the world CO<sub>2</sub> emissions at about 29 billion metric tons in 2007 [4]. In the absence of any international agreement on greenhouse gas (GHG) reduction, the coal use is projected to increase by more than 55% in 2035 with about 95% of the increase contributed by China and India [5]. The coal-fired electric power sector contributes about 33% of CO<sub>2</sub> emitted by all the fossil fuel sources (coal, petroleum and natural gas) in the U.S. [6]. Thus, it is amply obvious that cost-effective reduction of CO<sub>2</sub> emissions from the coal power plants will be the most important GHG reduction activity in the coming decades.

The recent progress of this project reported elsewhere has shown a good potential to achieve the target set by DOE [7-9]. If the project goals are met, the proposed technology will become available for the cost-effective capture of CO<sub>2</sub> from coal-fired power plants. The developed membrane modules and system can be applied in existing and new conventional coal-fired power plants.

### Spiral-Wound Membrane Element Fabrication

The design of the spiral-wound membrane module was divided into 3 sections, including the spiral-wound membrane element, Plexiglas FRP (fiber reinforced plastic) tube, and membrane housing. All modules were tested at OSU with simulated flue gas prior to the field test.

Each of the spiral-wound membrane elements was rolled with about 4" in diameter (using a 1.5" OD central tube) by 14.375" in length using four membrane leaves of 30" in length with a width of 14". The membrane leaf consisted of two pieces of the scale-up membrane (each at 30" in length with a width of 14"), a feed spacer in-between. The length of the membrane element was about 14.375", and the total membrane area was about 14000 cm<sup>2</sup>.

The image of one of the fabricated spiral-wound membrane elements is shown in Figure 1. The spiral-wound membrane element after fabrication was consequently inserted inside and glued to the Plexiglas FRP tube. The Plexiglas FRP was a Plexiglas tube that was analogous to and served as the fiber reinforced plastic (FRP) outer-wrap in commercial spiral-wound membrane elements. The Plexiglas FRP was 1/4" thick. The presence of 1/4" thick Plexiglas FRP aided in sealing by virtue of face-compression of the "O" rings on the flanges. The face compression "O" rings successfully helped in ensuring the elements without gas leakage and hence enhancing the CO<sub>2</sub> transport in each of the elements. Figure 2 shows the picture of a spiral-wound membrane module consisting of the membrane element inside a housing with a diameter of about 5 inches and a length of about 14 inches.

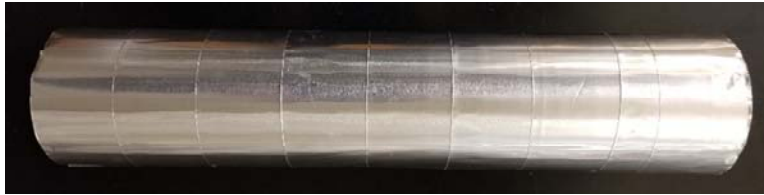


Figure 1. Spiral-wound membrane element fabricated.



Figure 2. Spiral-wound membrane module consisting of the membrane element inside a housing.

### 3 Experimental

A membrane-based testing unit was constructed and installed at NCCC in July 2018. The membrane system shown schematically in Figure 3 was used to measure the transport performance of the spiral-wound membrane module described above. The flue gas used for the testing of the prototype spiral-wound modules and other third-party developer technologies was drawn from Gaston Unit 5, a base-loaded, 880 MW<sub>e</sub> gross supercritical pulverized coal boiler fired with Alabama medium-sulfur bituminous coal and outfitted with selective catalytic reduction (SCR) technology for NO<sub>x</sub> control, and a wet flue gas desulfurization (FGD) unit. A NaOH scrubber was installed to further reduce the SO<sub>2</sub> down to ppm level. The flue gas that was sent to the testing unit was taken downstream of the NaOH scrubber, and it typically contained 12% ( $\pm 1\%$ ) CO<sub>2</sub>, 7% ( $\pm 1\%$ ) O<sub>2</sub>, 81% ( $\pm 1\%$ ) N<sub>2</sub>, 0.5 – 1 ppm SO<sub>2</sub>, and 1.5 – 4 ppm NO<sub>2</sub>, with balance of N<sub>2</sub>. Since OSU shared the flue gas slipstream with other technology developers, the flue gas was fully dried by NCCC to suit for the need of other testing campaigns. A compressor was provided by NCCC to deliver up to 4 slpm dried flue gas. Due to the intensive cooling prior to the compressor, the flue gas temperature to the membrane unit was in the range of 30 – 40°C.

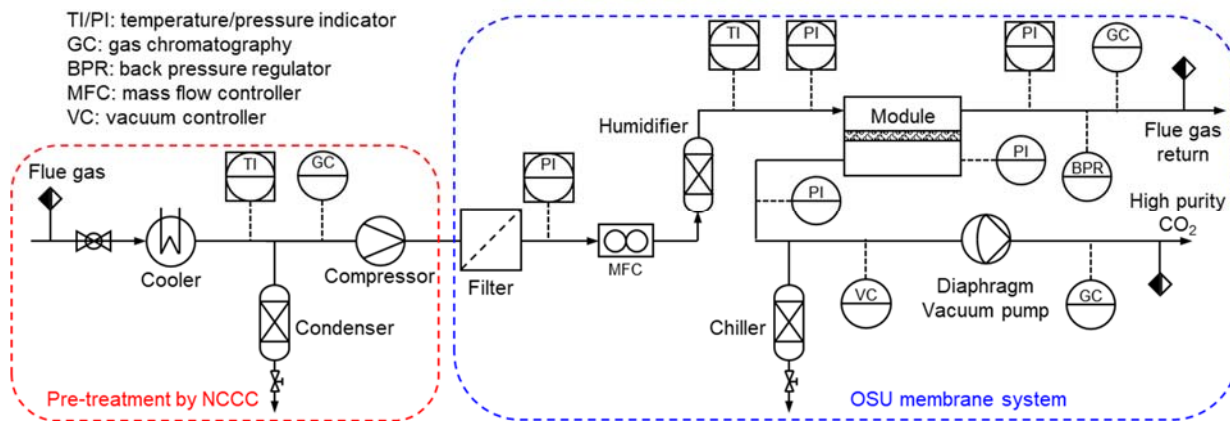


Figure 3. Schematic of the gas permeation unit for gas transport measurements.

For OSU’s membrane testing unit, the flue gas firstly passed through a 25- $\mu\text{m}$  disc filter to fully remove any residual particulate matter. The flue gas was then saturated with water vapor at the corresponding testing temperature and passed to the spiral-wound module as the feed gas. The treated retentate was vented. On the permeate side of the module, a vacuum was pulled by a diaphragm vacuum pump. A vacuum pressure down to 0.2 atm was precisely controlled by a vacuum regulator. Before the permeate stream passed to the vacuum pump, the moisture was knocked out by a chiller at 0°C. The compositions of the flue gas, the CO<sub>2</sub>-lean retentate, and the CO<sub>2</sub>-rich permeate were monitored by gas chromatography in real time. The pressures at the feed inlet, retentate outlet, permeate dead end, and permeate outlet were measured by pressure indicators, respectively, for the determination of the pressure drops on the feed and permeate sides, respectively.

Figure 4 shows the setup of the OSU membrane testing unit at NCCC. Due to the compact nature of the membrane unit, it was located indoor in the Lab-scale Testing Unit (LSTU) at NCCC. The unit consisted of 2 mass flow controllers, a humidifier, water pump, chiller, vacuum regulator, diaphragm vacuum pump, and gas chromatography. The membrane module to be tested was placed inside the oven of the unit as shown in Figure 5. The feed gas, flue gas, entered the module after passing through the humidifier, where the feed gas was humidified with water vapor by injecting the controlled amount of water into it to obtain the controlled concentration of water vapor.





Figure 4. The setup of the gas permeation unit at NCCC (photo credit: Tony Wu, NCCC).

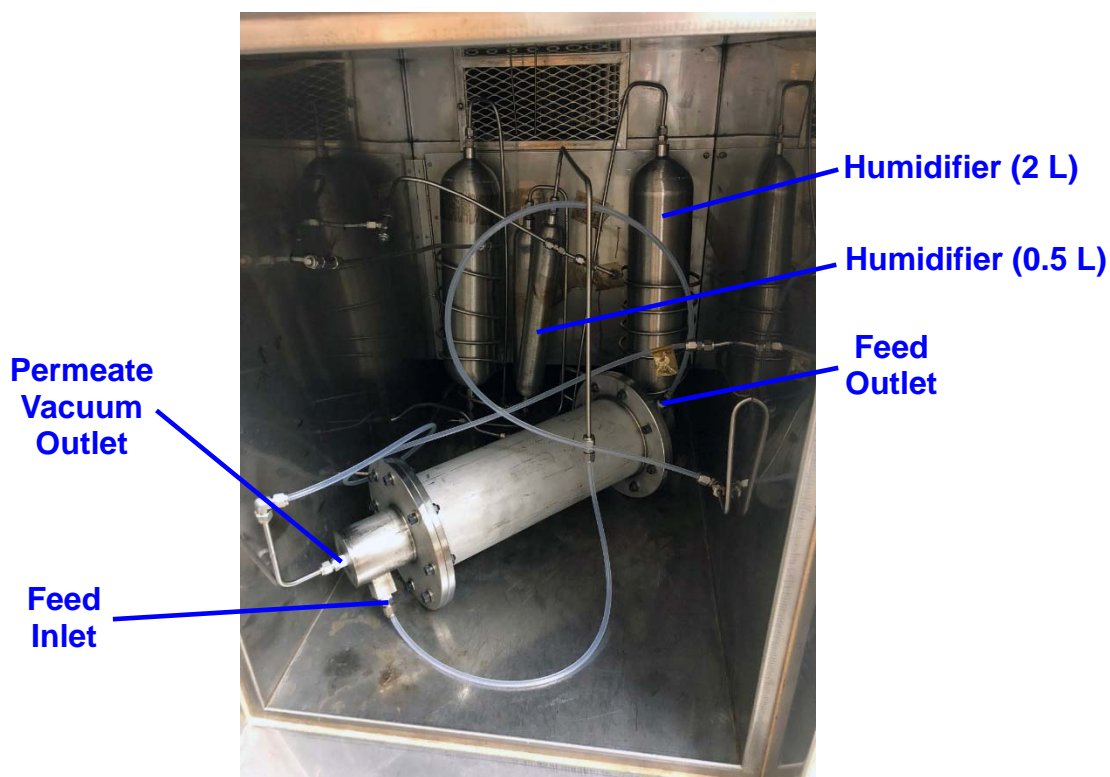


Figure 5. A spiral-wound membrane module placed inside the oven of the gas permeation unit for gas transport measurements.

## 4 Results and Discussion

### 4.1 Membrane module testing with simulated flue gas at OSU

Prior to the commissioning of the membrane module testing unit at NCCC, the performances of the spiral-wound membrane modules were characterized at OSU with simulated flue gas. Figure 6 shows the separation performance and stability of a spiral-wound membrane module with an active membrane area of 10000 cm<sup>2</sup>. Initially, the module demonstrated a CO<sub>2</sub> permeance of 1450 GPU and a CO<sub>2</sub>/N<sub>2</sub> selectivity of 185 at 67°C using the simulated flue gas (with ~17% CO<sub>2</sub>, 7% O<sub>2</sub> and 3 ppm SO<sub>2</sub>). At 400 hours into the run, the feed CO<sub>2</sub> concentration was reduced to 1% to simulate the retentate (simulated residual flue gas), resulting in an increased CO<sub>2</sub> permeance of 2010 GPU and an enhanced CO<sub>2</sub>/N<sub>2</sub> selectivity of 225. These performance improvements were mainly due to more available amine carriers for CO<sub>2</sub> transport at the lower CO<sub>2</sub> concentration. At 870 hours into the run, the feed returned to the simulated flue gas, and the original performance was obtained, showing an excellent stability for a total run time of 1900 hour.

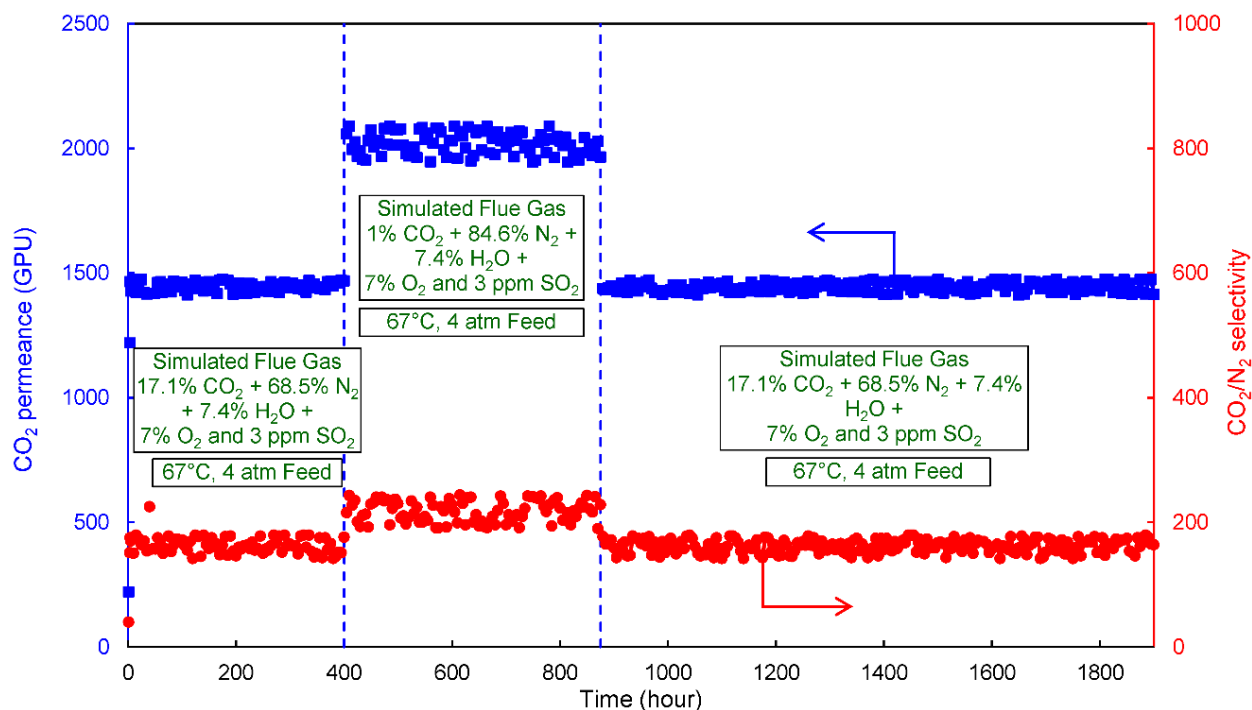


Figure 6. Spiral-wound module stability with simulated flue gas at 67°C.

### 4.2 Membrane module field testing with actual flue gas at NCCC

#### Separation performance and module stability

The separation performance of a 14000 cm<sup>2</sup> spiral-wound module (SW-65) is shown in Figure 7. The module was initially tested at 57°C with 1 and 0.2 atm of feed and permeate pressures,

respectively. Over the course of 24 hours, the module showed an average CO<sub>2</sub> permeance of 980 GPU with a CO<sub>2</sub>/N<sub>2</sub> selectivity of 164. The feed pressure was then increased to 4 atm, which led to an average permeance of 931 GPU with a selectivity of 153 for another 24 hours. Finally, the temperature was increased to 67°C with a vacuum pressure of 0.3 atm. The module demonstrated a good permeance of ~1450 GPU with a selectivity of ~185 for about 6 hours. However, the load of Unit 5 at NCCC dropped overnight, which led to an insufficient flue gas delivery pressure to OSU's testing unit. Due to the concentration polarization on the feed side, the CO<sub>2</sub> permeance reduced to ~1210 GPU with a selectivity of ~128. Once the flue gas delivery rate was re-established, the module performance was recovered and remained stable for 62 hours. After this, the flue gas flow rate reduced again due to the malfunction of a pressure regulator in the LSTU building. With NCCC team's help, the pressure regulator was replaced; the module performance was recovered and remained stable for 41 hours. Subsequently, a flue gas shutdown incurred by the malfunction of Unit 5 at NCCC, which led to a 14-h pause of the module test. During this flue gas shutdown, the original flue gas was replaced with a humidified N<sub>2</sub> to maintain the hydration of the membrane at 67°C and 4 atm. Once the flue gas returned, the separation performance of the module was recovered. A slightly higher CO<sub>2</sub> permeance was observed right after the flue gas returned, since the CO<sub>2</sub> concentration was only ~8%. After 128 hours of continuous testing with stable performance, Unit 5 was scheduled for a reserve shutdown. During the onset of the flue gas shutdown, the CO<sub>2</sub> concentration delivered to the module gradually reduced from 11% to 1.3%. This process lasted for 15 hours, and the CO<sub>2</sub> permeance eventually increased to ~1911 GPU. Afterwards, N<sub>2</sub> was used as the feed to replace the flue gas, and a stable N<sub>2</sub> permeance had been observed. After this 160-h flue gas outage, the module test was resumed, and a stable performance was observed. At the end, a cumulative 500-h test demonstrated the module stability when tested with actual flue gas at NCCC, despite the disturbances such as low flue gas flow rates and two flue gas shutdowns.

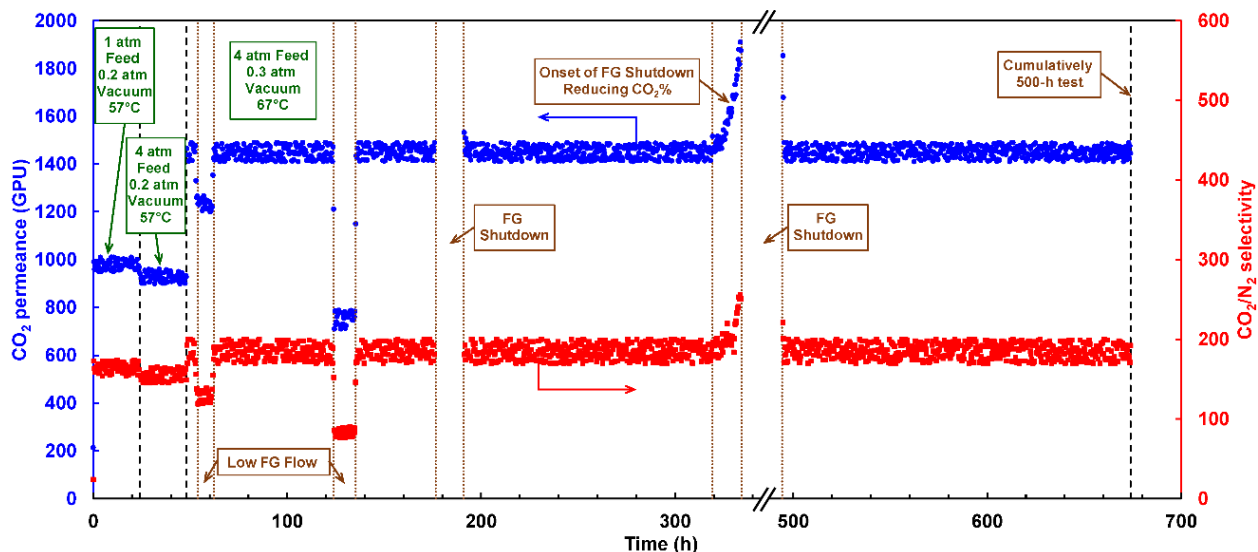


Figure 7. Stability plot of the spiral-wound module tested with actual flue gas at NCCC for 500 hours.

## CO<sub>2</sub> recovery and purity

Figure 8 shows the CO<sub>2</sub> recovery and permeate CO<sub>2</sub> purity of the spiral-wound module since the field trial was launched with actual flue gas at NCCC. At 57°C and 1 atm of feed pressure, a relatively low CO<sub>2</sub> recovery of ~7.5% was measured due to the low transmembrane driving force. The CO<sub>2</sub> recovery increased to ~30% by increasing the feed pressure to 4 atm. A further increased recovery of ~44% was obtained when the temperature was elevated to 67°C, owing to the increased permeance of the facilitated transport membrane. When the flue gas flow rate was low between the 124<sup>th</sup> and 135<sup>th</sup> hours, the CO<sub>2</sub> recovery increased to ~64% in spite of the concentration polarization on the feed side, presumably due to the reduced amount of CO<sub>2</sub> in the feed gas but with the same membrane area of the module for its removal. The highest CO<sub>2</sub> recovery, however, was registered during the onset of the second flue gas shutdown, where nearly 90% of the CO<sub>2</sub> was removed. The captured CO<sub>2</sub> in the permeate stream was about 95% or higher purity on dry basis during the normal operating conditions. However, a lower CO<sub>2</sub> purity was incurred when the flue gas flow rate or the feed CO<sub>2</sub> content was reduced.

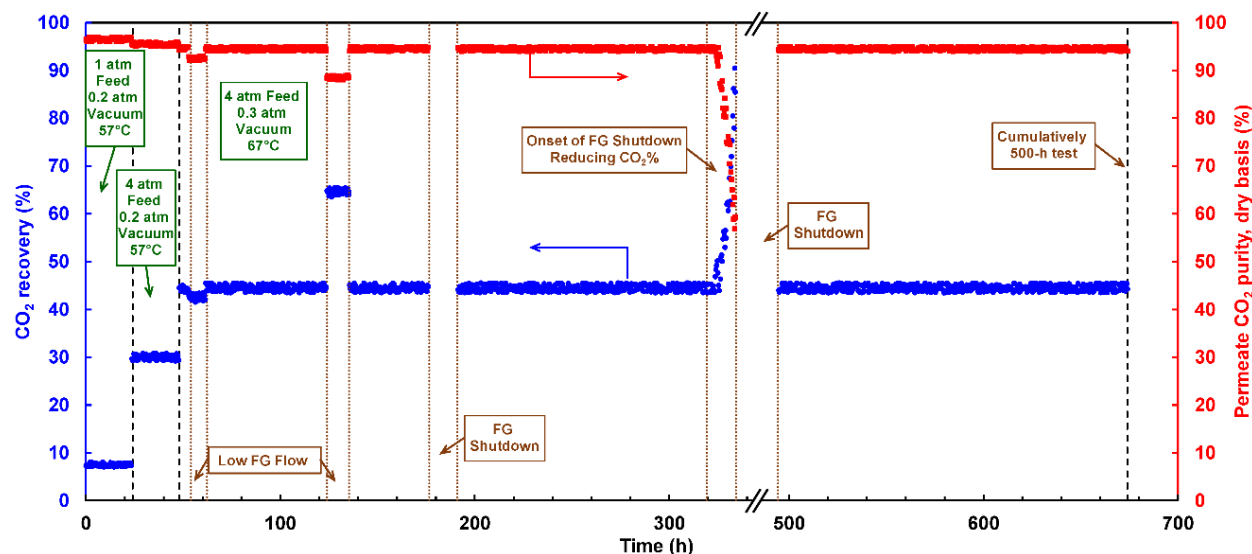


Figure 81. CO<sub>2</sub> recovery and permeate CO<sub>2</sub> purity of the spiral-wound module tested with actual flue gas at NCCC.

## Module separation performance reproducibility

The separation performance of the 14000 cm<sup>2</sup> spiral-wound module (SW-65) was reproduced with two other modules (SW-67 and SW-63) as shown in Figures 9 and 10, respectively. The modules were tested at 67°C with a vacuum pressure of 0.3 atm. The second module was tested for 24 hours and showed a CO<sub>2</sub> permeance of ~1455 GPU and CO<sub>2</sub>/N<sub>2</sub> selectivity of ~175. The third module was tested for 30 hours and exhibited a CO<sub>2</sub> permeance of ~1450 GPU and CO<sub>2</sub>/N<sub>2</sub> selectivity of ~180. These figures also indicated that these two modules were able to reach similar performances compared to the 100-hr test with simulated flue gas at OSU. In other words, the modules were successfully preserved well after the completion of the testing at OSU, and they showed similar performances with the actual flue gas at NCCC to those with the simulated flue gas at OSU.

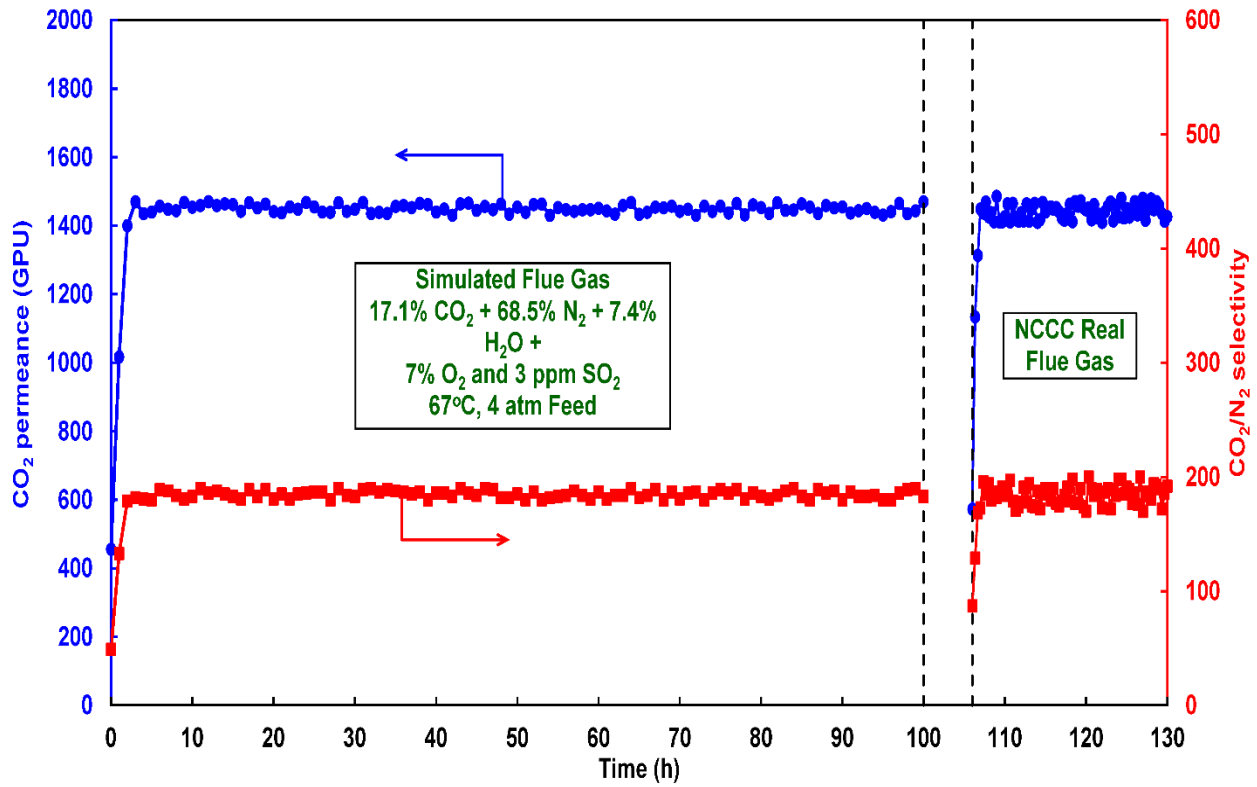


Figure 9. The stability plot of the spiral-wound membrane module SW-67 tested at NCCC.

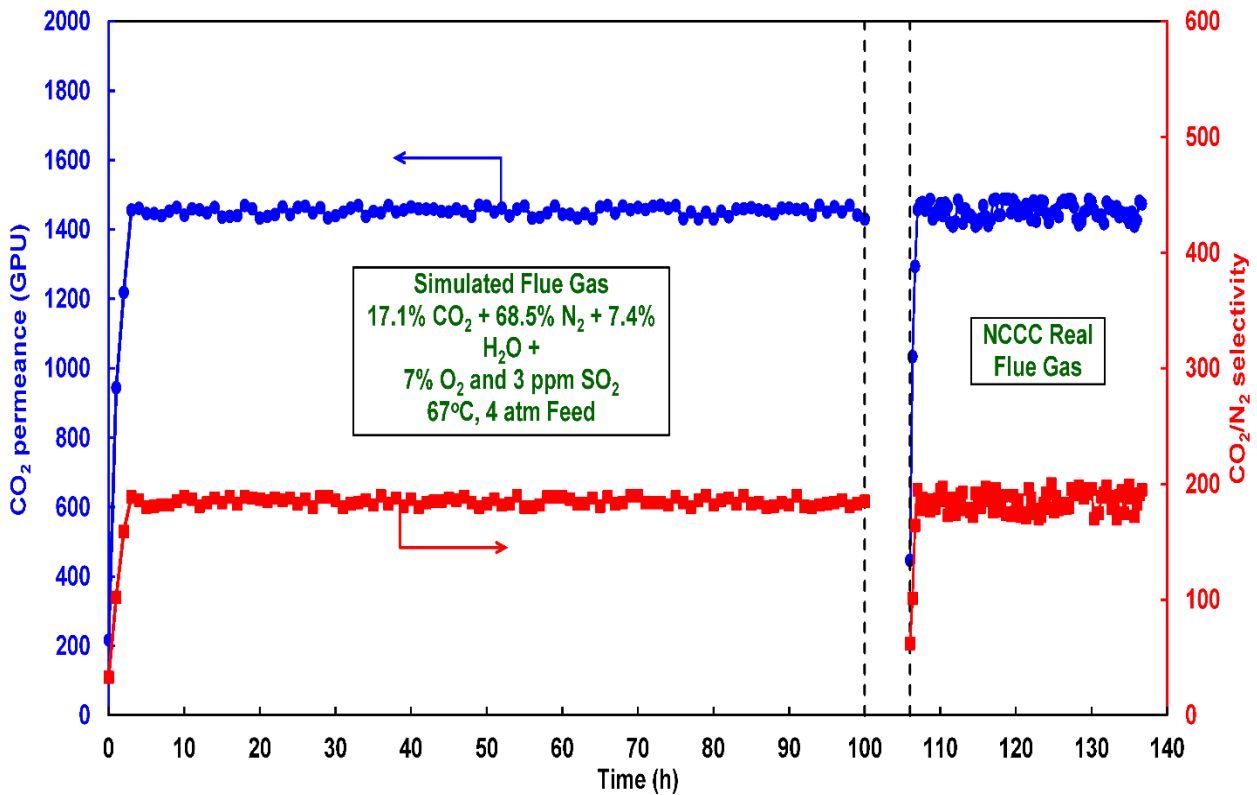


Figure 10. The stability plot of the spiral-wound membrane module SW-63 tested at NCCC.

## 5 Conclusions and Future Work

During July – August 2018, a 500-h field test campaign at NCCC, Wilsonville, AL showed that stable facilitated transport membrane module performance for carbon capture from real flue gas was achieved. Prototype spiral-wound modules, each with a membrane area of 1.4 m<sup>2</sup>, were developed for this project and were shown to operate efficiently and stably. Importantly for multi-year operations, the modules also demonstrated stable performance over multiple flue gas shutdowns and restarts of the membrane testing system.

The separation performance of the spiral-wound modules well reproduced that of the lab-scale flat-sheet membrane in terms of CO<sub>2</sub> permeance and CO<sub>2</sub>/N<sub>2</sub> selectivity. More importantly, a relevant CO<sub>2</sub> recovery (90%) with a high CO<sub>2</sub> purity (95%) was realized by the developed facilitated transport membrane in the commercial spiral-wound membrane module configuration for CO<sub>2</sub> capture from the actual flue gas at NCCC. Carbon capture rates of more than 40% were achieved by a single spiral-wound module with the coal-derived flue gas. Except for a few flue gas upsets, the CO<sub>2</sub> purities in the capture stream were about 95% or better on dry basis.

Significant responses to various operating conditions have been noted, particularly with respect to the feed pressure, feed CO<sub>2</sub> concentration, operating temperature, and flue gas shutdowns. The data obtained from this project have provided a basis for the design and fabrication of the full-scale spiral-wound module with a membrane area larger than 50 m<sup>2</sup>. Lastly, lessons learned from the field trial will be useful for the design and construction of an integrated membrane skid consisting of 2 membrane stages for 90% capture of the CO<sub>2</sub> with ≥95% CO<sub>2</sub> purity from a 10 TPD slipstream.

## 6 Acknowledgments

The smooth membrane module testing at NCCC would not be possible without the great efforts of NCCC team members, particularly Tony Wu and Bob Lambrecht. We are grateful for their excellent analytical and mechanical supports, including the arrangements of lifting our membrane unit into their Lab-scale Testing Unit (LSTU) at NCCC, setting it up, connecting all tubing and electrical parts outside of our unit, installing air flow to our oven for accurate temperature control, and giving us the concentration data log (including CO<sub>2</sub>, O<sub>2</sub>, SO<sub>2</sub>, NO<sub>2</sub>, and NO concentration from the flue gas, and O<sub>2</sub> concentration from the retentate stream). NCCC support and cooperation for our membrane module testing were wonderful and professional, and we truly enjoyed working with them.

We would also like to thank José Figueroa and David Lang, DOE/NETL and Matt Usher and Dean Bell, AEP (American Electric Power) Engineering Services for their great efforts and strong inputs for this membrane module test. We acknowledge the financial support from the Ohio Development Services Agency (ODSA, Project OER-CDO-D-15-09) with AEP cost share for this test. We also acknowledge DOE/NETL (Project DE-FE0026919) for providing/serving as the cost share to the ODSA Project.

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