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Development of a Pre-Combustion CO_2 Capture Process Using High-Temperature PBI Hollow-Fiber Membranes

Final Report
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PROJECT DESCRIPTION

Introduction

SRI International (SRI) performed a research project sponsored by the Department of Energy (DOE) to demonstrate the polybenzimidazole (PBI) polymer hollow-fiber membrane (HFM)-based carbon dioxide (CO_2) capture and purification technology in pre-combustion applications. The project was carried out in two budget periods, BP1 and BP2. This report documents entire research work performed for both BP1 and BP2. The team completed the BP1 and BP2 scope of work and achieved the associated milestones and success criteria on schedule and within the budget.

Project Objectives

The overall objective of this project was to evaluate the advantages of transformational PBI HFM-based, CO_2 capture and purification technology at bench scale using an actual coal-derived syngas stream from an oxygen-blown coal gasification facility. The technical objectives in BP1 included preparing HFMs, fabricating modules, and upgrading the available skid for field testing. The technical objective for BP2 was to field-test the skid unit with actual coal-derived syngas from an oxygen-blown gasifier, obtain dynamic and steady state performance data, and update the Techno-Economic Analysis (TEA) that would assist with future process scale-up and design information of a small pilot-scale test unit.

Project Tasks

The research program was designed with a project management task and five progressive technical tasks and leading to both dynamic and steady-state testing of the PBI-HFM skid with actual coal-derived syngas. The work plan was to: (1) fabricate sufficient GEN-2 fibers for module fabrication; (2) upgrade the fiber skid to accommodate new fiber modules for bench-scale field testing; (3) conduct dynamic and steady-state testing with coal-derived syngas from an oxygen-blown gasifier and obtain system performance data; (4) perform a TEA and environmental, health, and safety (EH&S) assessment; (5) update the State-Point Data Table, Technology Gap Analysis, and Technology Maturation Plan; (6) uninstall and return the test skid to the Recipient’s facilities; and (7) submit a Final Report that describes the results and analysis of the project research effort.

PROJECT STRUCTURE AND TEAM

SRI assembled a diverse team to perform the above-mentioned project tasks. The team combines: a premier R&D institute (SRI International) with expertise in innovating technology, scaling up, and commercializing new technologies; firms specializing in system design/

integration and energy commercialization strategies (Enerfex, Inc. and Energy Commercialization, LLC); a company that fabricates polymer formulations and fibers (PBI Performance Products, or PBI PP); and a host site gasifier facility (University of Kentucky - Center for Applied Energy Research, or UKy-CAER).

ACCOMPLISHMENTS

BP1

The main research work performed in BP1 included the following activities:

- The fiber test skid (50 kWth) from the field test campaign at the National Carbon Capture Center (NCCC) was updated and modified. Modifications involved installation of new Coriolis gas flow meters, N_2-sweep gas flow line plumbing, heat tracing, new insulation, and installation of two new fiber module vessels. With this addition, there were four fiber module vessels on the skid. The two new vessels were plumbed with an option to use the N_2 sweep for the permeate flow. A photograph of the updated skid is shown in **Figure 1**.
- A new fiber tube elements casing was designed. In the new design, fibers were enclosed in a 9/16-in.-diameter and 28-in.-long stainless-steel tube (fiber tube element). Fibers were potted and sealed to the tube at each end of the tube. A set of holes on the side of the tube just below the potting area allowed entry and exit of feed gas at each end. This design supported *one-end-closed* and *both-ends-open* configurations for the tube elements. Each fiber tube element contained 175-200 individual fibers, and 19 such tube elements were in a fiber module vessel. Fiber tube elements were fixed in a tube sheet array at each end to form a fiber module assembly.

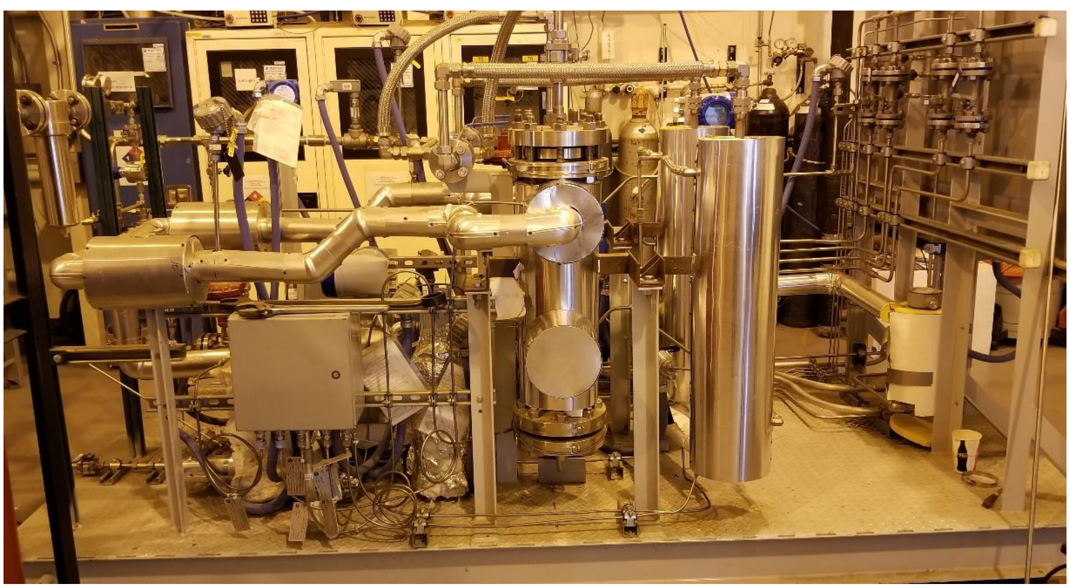


Figure 1. A photograph of the updated skid.

- New fiber module vessels were designed to reduce gas bypass, increase ease of replacing fiber tube elements, and reduce overall external vessel dimensions. The new vessel design was very similar to industrial units for reverse osmosis.
- A large quantity of fibers was spun to fabricate tube elements, and for quality control testing. The total length of fiber generated was more than 150 km. Individual tube elements for both previous and new vessel designs were made. The previous design tube elements were installed in older vessels, and the new tube elements with the option for N_2-sweep gas were installed in the new vessels.
- Slight variations in solid content (e.g., 20 to 21 wt%) of the PBI dope were seen from batch to batch and had a significant effect on the viscosity of the dope and hence the spinneret extruding pressure, which affected the fiber strength and microstructure. SRI worked with the PBI PP technical team and resolved the issue to improve the batch-to-batch consistency of the solid content.
- The PBI skid control program was updated to include new sensors, flow controllers, and valves. New safety interlock logic was incorporated to prevent over-pressurization of the onboard gas analyzer. The gas analyzer was further protected by installing a pressure regulator in the upstream of the detector cell.
- An engineering package of the skid detailing utility requirements, transportation, and installation was prepared and provided to the host site. The host site technical team reviewed the engineering package and provided feedback, and all field site issues were addressed before the skid was transported to the field test site. The host site agreement for testing was prepared and executed in January 2020.
- Shakedown testing of the modified skid was completed with simulated gases and installed the skid in a container (20 x 19 feet) that would permanently house the skid for transportation and field testing. The lighting and air circulation were installed to the container for maintenance and safety.

The testing of the skid with simulated syngas was planned for June 2020. However, the laboratory work was delayed due to COVID-19 shelter-in-place restrictions. Testing commenced as soon as the restriction was lifted. We completed 50 hours of skid testing and prepared for transportation of the skid to the field test site.

BP2

The main research work performed in BP2 included the following activities:

- Parametric tests were performed, and optimal process conditions were identified for steady-state testing. The field test host site provided the syngas from an oxygen-blown gasifier with the gas composition (H_2: 24.5%, CO: 28.94%, CO_2: 40.89%, N_2: 2.93%). The maximum syngas flow rate is 40 m^3/hr, and the maximum pressure is about 30 bar.

- The PBI-HFM bench skid was operated for over 500 hours and data was collected depending on field test host site availability. Feed, permeate, and retentate gas flow compositions were monitored by the field test host site using a UKy-CAER two-channel gas analyzer and/or the onboard skid-mounted analyzer interfaced with process control hardware and software.
- Data collected from the parametric and steady-state operation of the skid with syngas from the oxygen-blown gasifier were shared with team members for interpretation and analysis. Charts, plots, and tables were prepared to present and interpret the data.
- A final technology assessment was conducted following bench-scale field testing with actual coal-derived syngas from an oxygen-blown gasifier. The results were incorporated into the following deliverables:
    - The preliminary TEA was updated based on the results from bench-scale field testing and prepared a final TEA in accordance with DOE guidelines as stated in the PMP.
    - The preliminary Technology Maturation Plan (TMP) was updated following bench-scale field testing and prepared the final TMP in accordance with the Statement of Project Objectives (SOPO).
    - The State-Point Data Table was updated based on the test results from actual testing and the updated heat and mass balance data from modeling. The updated State-Point Data Table was submitted in accordance with the SOPO.
    - The Technology Gap Analysis (TGA) was prepared in accordance with the SOPO. The TGA included technical risks and risk mitigation strategies for advancement of the technology to the next scale of development.
    - The EH&S process risk assessment report was prepared and submitted in accordance with the SOPO. The assessment provided a full overview of the risks and hazards associated with the PBI-HFM CO_2 capture process and detailed the risks and mitigations for the technology based on data and information collected throughout the project.
- The skid was decommissioned and prepared for transport. This task involved disconnecting the tie-ins to the syngas feed and other utility connections at the field test host site. The skid was transported from the field test host site to SRI at Menlo Park, CA.
- The skid was prepared for long-term storage. This work involved removing the PBI membrane fiber elements from the pressure vessels and any foreign deposits on surfaces

as a result of their exposure to coal-derived syngas, and flushing the skid components (e.g., valves, flow meters, pressure gauges) with nitrogen. The PBI bench skid is currently stored at SRI and it may be used in future field test programs.

- The Final Report was prepared for the DOE and documented the complete research activities performed in both BP1 and BP2.

PROJECT SUCCESS CRITERIA FOR BP1 AND BP2

All specific project success criteria were successfully met on schedule. These criteria included: (1) completing the update the 50 MWth unit; (2) installing a second fiber spinning line to double the fiber spinning capacity from 4 to 8 m/min; (3) spinning of >100 km good quality fibers with high baseline performance; (4) shakedown testing of the skid with simulated gas mixtures; (5) transporting and installing the skid at the field site; and (6) completing at least 300 hr. of field testing with syngas and demonstrating the targeted performance.

FUTURE WORK

The PBI HFMs can be successfully used for high-temperature gas separation applications. Specifically, PBI HFMs have many advantages over current HFM technology in H_2/CO_2 separation as demonstrated in this project, and hence the technology has a clear edge in H_2 production industry. We have also demonstrated that PBI HFMs can be used in desalinations and ultra-filtration applications. Recently, we completed a project to demonstrate the use of PBI HFMs to reuse of process water from a powerplant. Future projects should be focused on H_2 production and water desalination/purification applications.

PROJECT OBJECTIVES AND GOALS

The overall objective of this project was to evaluate the advantages of transformational polybenzimidazole (PBI) polymer hollow-fiber membrane (HFM)-based, carbon dioxide (CO_2) capture and purification technology at bench-scale using an actual coal-derived syngas stream from a coal gasification facility. The project was carried out over two budget periods. The technical objectives in Budget Period 1 (BP1) included preparing HFs and modules and upgrading the available skid for field testing. The technical objectives for BP2 were to field-test the skid unit with actual coal-derived syngas from an oxygen-blown gasifier to obtain performance data, update the Techno-Economic Analysis (TEA) that would assist with future process scale-up, and provide information on the design of a small pilot-scale test unit. The goal was to advance the PBI-HFM CO_2 capture and gas separation system for pre-combustion applications beyond second-generation economic performance predictions and make progress toward meeting overall fossil energy performance goals of CO_2 capture with 95% CO_2 purity at a cost of electricity (COE) 30% less than baseline capture approaches.

The research program was designed with progressive technical tasks leading to both dynamic and steady-state testing of the PBI-HFM skid with actual coal-derived syngas. The work plan was to: (1) fabricate sufficient Generation-2 (GEN-2) fibers for module fabrication; (2) upgrade the fiber skid to accommodate large fiber modules for bench-scale field testing; (3) conduct dynamic and steady-state testing with coal-derived syngas from an oxygen-blown gasifier and obtain system performance data; (4) perform a TEA and environmental, health, and safety (EH&S) assessment; (5) update the State-Point Data Table, Technology Gap Analysis (TGA), and Technology Maturation Plan (TMP); (6) uninstall and return the test skid to the Recipient's facilities; and (7) submit a Final Report that describes the results and analysis of the project research effort.

PROJECT MANAGEMENT AND PLANNING

The project was carried out over two budget periods, BP1 and BP2. Task 1, which ran through the entire project period included all planning, budgeting, reporting, subcontract management, and communications with DOE. The details of activities performed are given below.

TASK 1.0 - PROGRAM MANAGEMENT AND PLANNING

Task Description: *The Recipient shall manage and direct the project in accordance with a Project Management Plan to meet all technical, schedule and budget objectives and requirements. The Recipient will coordinate activities in order to effectively accomplish the work. The Recipient will ensure that project plans, results, and decisions are appropriately documented, and project reporting and briefing requirements are satisfied.*

The Recipient shall update the Project Management Plan (PMP) 30 days after award and as necessary throughout the project to accurately reflect the current status of the project. Examples of when it may be appropriate to update the Project Management Plan include: (a) project management policy and procedural changes; (b) changes to the technical, cost, and/or schedule baseline for the project; (c) significant changes in scope, methods, or approaches; or (d) as otherwise required to ensure that the plan is the appropriate governing document for the work required to accomplish the project objectives.

Management of project risks will occur in accordance with the risk management methodology delineated in the Project Management Plan in order to identify, assess, monitor and mitigate technical uncertainties as well as schedule, budgetary and environmental risks associated with all aspects of the project. The results and status of the risk management process will be presented during project reviews and in Progress Reports with emphasis placed on the medium- and high-risk items.

Summary of Accomplishments

The following project requirements were completed in BP1 under Task 1: (1) Project kickoff meeting at DOE; (2) submission of project property report; (3) submission of the preliminary TEA; (4) skid modification to include two newly designed module vessels; (5) finalization of the project TMP (a summary is provided herein); (6) a site visit to the University of Kentucky - Center for Applied Energy Research, (UKy-CAER) to discuss field test details and requirements; (7) execution of host site test agreement; (8) submission of the continuation report and presentation for BP1. The project continuation review for the BP1 was held on 11 June 2020 via WebEx, and SRI received the approval to continue the project to BP2.

In BP2, project management and planning activities performed under Task 1 included coordination with the host site on details for preparing the skid for its acceptance at their site, SRI team travel logistics, arrangements of cranes for skid lift, and finalizing the test schedule.

SRI issued subcontract awards for Enerfex and University of Kentucky for BP2 research activities. SRI prepared the EH&S report, Technology Gap Analysis, and the Final report, and delivered the project close-out presentation on March 30th, 2023, with the DOE project manager.

BUDGET PERIOD 1 (BP1) RESEARCH ACTIVITIES

SUMMARY

The main research activities in BP1 were carried out under Tasks 2 and Task 3. These activities included: updating and modifying the fiber test skid with new flow meters, insulation, and plumbing; redesigning and fabricating fiber tube elements and vessels; spinning a large quantity of PBI hollow fibers for module fabrication; improving quality control and performance of fibers from batch to batch; updating software and hardware for data acquisition and control; preparing an engineering package of the skid for the host site; executing the host site test agreement; and conducting shakedown testing of the skid.

Complete testing of the skid with simulated syngas was originally planned for June 2020. However, the laboratory work was delayed due to COVID-19 shelter-in-place restrictions and the testing was commenced once the restriction was lifted. The research activities performed in BP1 are discussed below in detail.

TASK 2.0 - BENCH SKID MODIFICATIONS AND ACCEPTANCE TESTING

Task Description*: The Recipient will refurbish and update an existing PBI test skid constructed during a previous project (FE0012965), design a new pressure vessel to house the modules, and fabricate the Recipient's GEN-2 fibers and modules. The Recipient will also evaluate the performance of the updated skid using a simulated gas stream closely resembling shifted syngas composition (e.g., H_2:40-45%, CO:2-3%, CO_2:40-42%, N_2:2-4%). The experimental results will be compared with the process model predictions derived in Task 3.*

The Recipient will also update the Basic Engineering Design Package (BEDP) of the skid test unit and incorporate all the required mechanical details of the system, including an updated equipment specifications and layout plan. The Recipient will provide utility requirements for the field test skid to facilitate preparation of a skid installation plan by the field test host site, which will be executed during Task 4.

Summary of Accomplishments

Research work in Task 2 was performed under five subtasks. The subtasks were divided by different activity groups as: (1) skid refurbishment; (2) installation of new fiber line and high-volume fiber spinning; (3) design of fiber modules and vessels; (4) skid shakedown testing; and (5) review of the piping and instrumentation diagram (P&ID) and hazard and operability (HAZOP) evaluation. These subtasks activities are described in detail below.

Subtask 2.1 - Refurbishing and Upgrading of the Existing Bench Skid

Task Description*: After the previous test under FE0012965, the fiber test skid (4'-8" x 10'-6" in size installed with 4 pressure vessels designed to operate at 225℃) was returned to the Recipient's facilities. Currently the skid is installed at SRI's Menlo Park Campus High Bay Facility. This skid was designed to handle the 500 slpm shifted syngas flow rate and 300 slpm hydrogen flow rate. The Recipient will examine the skid and replace and update the system components, as needed. The Recipient will also add two new large module vessels (approximately 4" diameter and 36" length), and install Coriolis flow meters to accurately measure permeate and retentate gas streams. The fiber elements will be replaced with the Recipient's GEN-2 fiber elements produced during Subtask 2.2.*

Skid Modification for New Vessels and Heat Tracing

The skid refurbishment work completed in BP1 included redesign and fabrication of new fiber module vessels, removal of the two Generon® vessels to make room for the new module vessels, installing a Coriolis flow meter to measure the inlet gas flow, installing heat tracing and insulation for new lines and vessels, upgrading the electrical control panel, and servicing and calibrating the NOVA gas analyzer. A photograph of the skid is shown in **Figure 2**. Two new SRI-designed 4-in. vessels were installed in the skid (shown in **Figure 2** without insulation covering). The details of the new vessel design are given in Task 2.3.

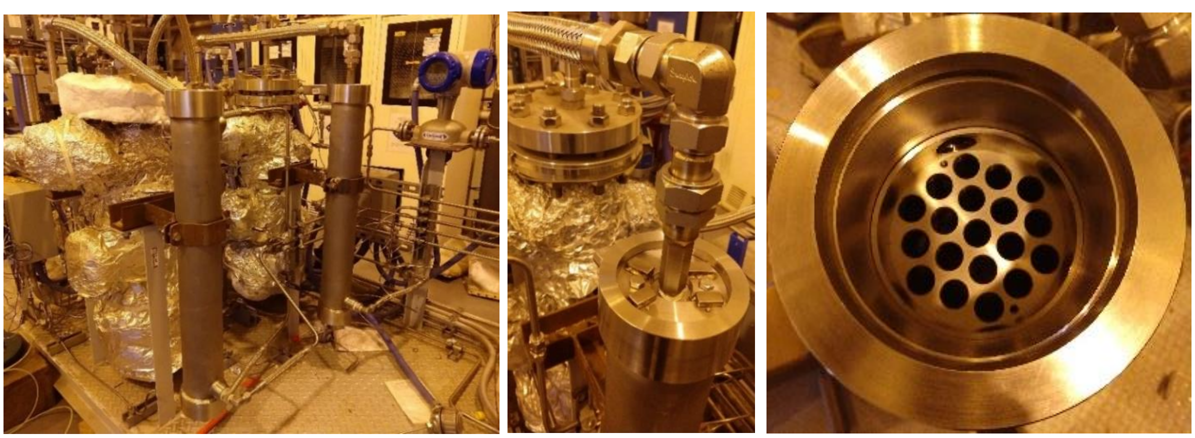


Figure 2. Two new modules (in foreground, shown uninsulated) installed in the skid at SRI. Top views of new vessels, both closed and opened, also pictured.

To service the new fiber module vessels, we added new piping lines in the skid. Heat tracing elements were installed to these new piping lines and vessels. We also installed a nitrogen gas sweep line and a gas heater, providing the flexibility of using a sweep gas in the permeate side during testing. The use of sweep gas in the permeate side depends on the module design as it needs open-ended fiber modules. The new 4-in. vessels support *both-ends-open* fiber modules as well as *one-end-closed* modules, as described in detail under relevant tasks.

Gas Flow Meters and Measurements

One of the challenges we experienced at the National Carbon Capture Center (NCCC) was ensuring the accuracy of gas mass flow meters. Common mass flow meters use the thermal conductivity of the gas to determine the flow rate, and thus they are not suitable for gas mixtures with varying composition. As the gas composition changes, mass flow meters fail to measure accurate flow rates. Coriolis flow meters are a much better alternative because they measure the total mass flow regardless of the composition and, with known gas composition from online analyzers, the volumetric gas flow can be determined. We had installed a Coriolis gas flow meter in the retentate line for the test at NCCC. We installed a second Coriolis flowmeter in the skid to measure the inlet syngas flow rate. Combined with the Coriolis meter already installed on the retentate line and the gas composition analyzer, the new Coriolis meter will allow us to significantly improve the accuracy of the mass balance and evaluation of membrane performance.

The wiring of the skid was also updated to accommodate the new Coriolis flow meter, the nitrogen mass flow controller for sweep gas, and the heat tracing system. The electrical control panel and associated electronics were modified to support the changes to heat tracing, flow controls, and relays. We also updated the control program to service changes of instrumentation in the skid. The skid piping and reactor vessels (two old-style vessels and two new vessels) have been re-insulated with more durable materials. The new insulation also allows for easier removal and reinstallation around the tops of the vessels to facilitate easier replacement of fiber modules in the vessels. The series of photos in **Figure 3** shows the progress of the skid modification and insulation work.

Figure 3. Reactors and piping on the skid were first insulated with fiberglass (top left) and then sheathed in aluminum.

Gas Composition Analyzers

During the SRI visit to UKy-CAER in May 2019, we discussed the availability of analytical instruments at their site for online monitoring of feed, permeate, and retentate streams. Based on these discussions, it appeared that an additional online flow monitoring device on the skid would be useful because the existing monitoring systems at the UKy-CAER may sometimes be unavailable or have limited access. The skid unit was originally equipped with a NOVA analyzer for online flow composition monitoring. However, it did not function properly during the NCCC run campaign, and thus we used the NCCC's analyzers instead. We have isolated the problem with the NOVA analyzer to sudden pressure changes in sampling lines as we switched from inlet or retentate to permeate flow. We installed new pressure regulators in the sampling lines and modified the software for a new valve-switching sequence to avoid pressure fluctuations in the analyzer. Taking measures such as these to protect the analyzer from upset conditions is important because the analyzer is installed in a purge cabinet and cannot be accessed for troubleshooting during operation in a Class I, Division 2 environment. With these changes, the analyzer worked perfectly and was calibrated for CO_2 and hydrogen. We completed the shakedown tests of the skid with newly installed components.

Subtask 2.2 - Fabrication of Generation 2 (GEN-2) Fibers

Task Description*: The Recipient will fabricate and process 100-200 kilometers of GEN-2 fibers using existing production methods. The produced fibers will be made into submodules (each submodule with 0.5 to 0.75" diameter, 18 to 30 units). The submodules will be tested individually using the Recipient's existing test system to ensure their integrity before assembly into modules and installation in the bench-scale pressure vessels. The testing of the submodules will be performed using a bench-scale test system that has a capacity to test gas flow rates in the range 100 ml/min to 10 liter/min. The top-performing submodules that experimentally achieve H_2/CO_2 selectivities 30 or higher will be selected for installation into the bench-scale pressure vessel.*

New Fiber Spinning Line Installation

During BP1, we updated the fiber-spinning infrastructure and capabilities at SRI. We have procured replacement spinnerets as they wear out in continuous long-term operation. In addition, SRI installed a second spinning line to increase the fiber production capacity. **Figure 4** shows a photograph taken during the installation of the second hollow fiber spinning line. The new line is marked as Line 2. This system has identical components: pumps, spinneret holders, coagulation bath, washing bath, stretch rollers, and the take-up roller. The fiber spin lines were installed parallel to each other as shown in the picture. The PBI dope solution and the bore solvent were delivered to the spinneret using high-pressure pumps with an accuracy of +/- 0.01 ml/min.

Figure 5 and **Figure 6** show the schematic diagram and photographs of the spinning line, respectively. The drive wheels on the line can be independently controlled, allowing fibers to be precisely stretched at any stage. Using the spinning lines, we produced PBI hollow fibers for the modules and skid test demonstration.

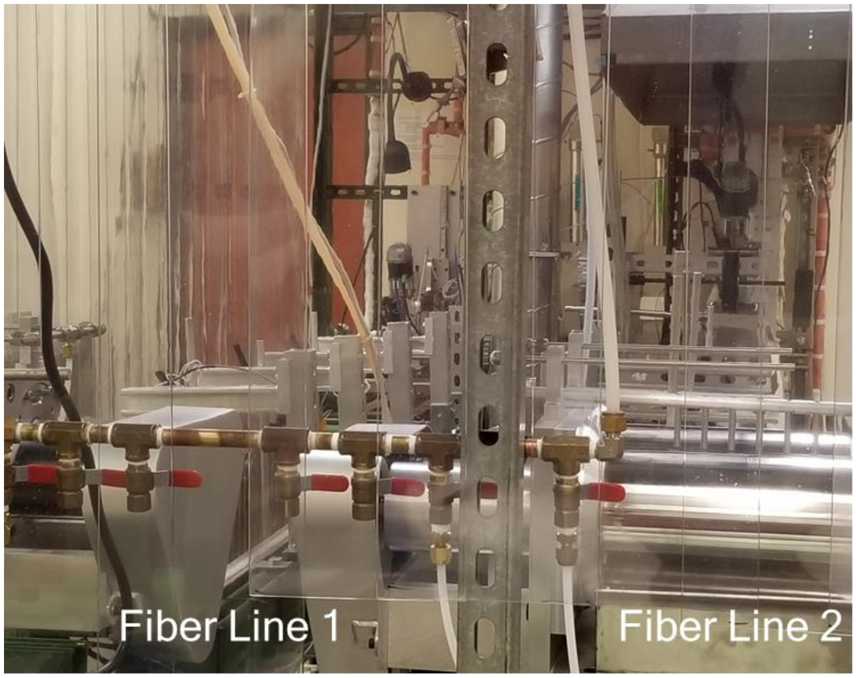


Figure 4. A photograph of the new fiber spinning line. Line 1 is the old line, and Line 2 is the newly installed line.

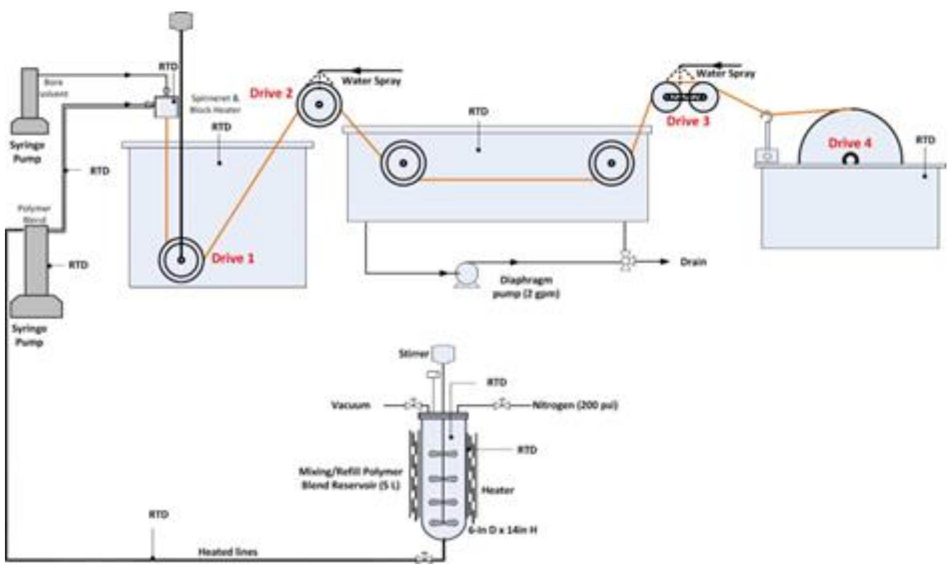


Figure 5. Schematic diagram of the SRI HFM spinning line.

Figure 6. Photographs of the spinning line.

PBI Hollow Fiber Quality Control

In the previous project, testing of high-performance fibers at the NCCC revealed the mechanical stability and flexibility of the fibers is important under high flux and pressure fluctuations. If the fibers lack stability, they can be damaged during handling and vibrations in the process. The goal of the current project is to fabricate a large stock of fibers that have high gas separation performance during field demonstration, as well as being mechanically stable and flexible enough to withstand handling, module making, and regular processing without damage to the fibers. The PBI is a glassy polymer, and the presence of macro-voids reduces the mechanical strength of the fiber. Thus, we optimized the spinning process to avoid macro-void formation inside the wall of HFM fibers.

To identify the key process parameters that would optimize the fiber spinning, separation performance, and mechanical stability, we used a generic design of experiment (DoE) approach to quickly determine the input factors that could be adjusted to improve the quality of the hollow fiber. As an example, **Figure 7** illustrates the relationship between controllable input factors, uncontrollable input factors, and responses, and Table 1 provides details about the factors related to the fiber spinning process.

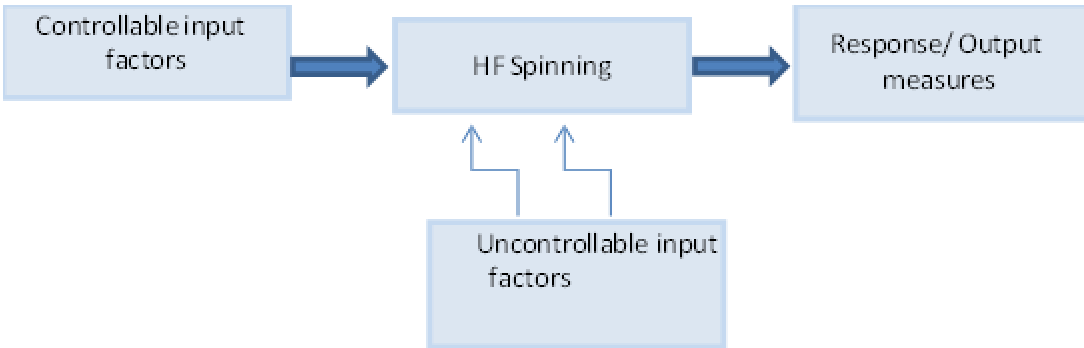


Figure 7. Fiber spinning process factors and responses flow chart.

Once the spinning conditions for producing a hollow fiber with the desired dense layer and separation performance are selected, the next step in the optimization process is to identify the process parameters that influence the formation of macro-voids. In the optimization process, the measured responses are the gas permeation rate and the density of macro-voids.

Table 1. Parameters for spinning optimization.

Parameter	Notes
Controllable parameters Dope solution (composition, flow rate, and temperature) Bore solution (flow rate, composition, and temperature) Coagulant (flow rate, composition, and temperature)	Spinneret design is fixed
Output Response Shell side dense layer thickness and defects Lumen side pore structure H_2 Permeance H_2/CO_2 selectivity Macro-voids Mechanical strength (Mandrill test)	Key screening parameters are density of macro-voids, dense layer thickness and the H_2/CO_2 selectivity

Dope composition variability:

Earlier in the project, we found that the dope provided by PBI Performance Products (PBI PP) exhibited a slight variation in solid content (e.g., 20 to 21 wt%) from batch to batch. The variation in the solid content has a significant effect on the viscosity of the dope and hence the dope line pressure through the spinneret, which changes the microstructure of the fiber. We worked with the PBI PP technical team to address the issue to improve the consistency of the solid content. PBI PP sent two variations of dope (SRI Blend 7A and Blend 7B) with 18.3 and

20.7% solids content to allow us to evaluate the effect of solids content on formation of macro-voids in the fibers and the overall fiber performance. Once the dope composition inconsistency was resolved, the fiber microstructure variation was significantly reduced. The dope composition was finalized for use in both spin lines.

During the fiber spinning, each fiber batch was characterized by examining the fiber porous area, surface and the cross-section using scanning electron microscope (SEM). The sample images in **Figure 8** show the lumen side and the cross section of the porous structure for fiber from lot 100C. The fibers after process optimization are macro-void free and are mechanically strong and highly flexible. **Figure 9** shows a fiber that was wrap-around of a ¼ inch tube. The amount of fiber spun in the project during troubleshooting, process optimization, and skid testing is given in **Table 2**. We have spun over 150 km of fibers in total length.

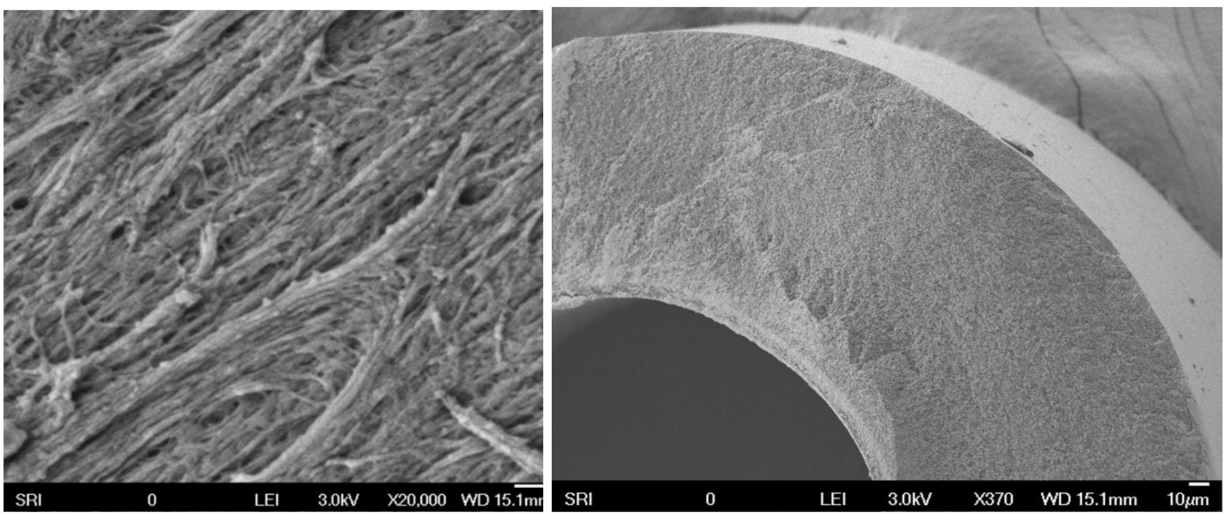


Figure 8. High-magnification photographs of the new fibers showing open porous lumen surface and support structure cross section.

Figure 9. Macro-void-free fiber that wraps around a ¼-inch tube.

Fiber washing and drying:

In BP1, we refined the process for post-treatment of spun fibers after observing that the washing process had some effect on the fiber microstructure. After the fibers are spun, they go through a multi-step washing process, beginning with a water wash and ending with an isopropyl alcohol wash (IPA), after which they are cross-linked. The early fibers spun on the current project were dried between the washing and cross-linking steps. However, this drying process, in which the fibers were hung to dry in the back of a fume hood, was faster than anticipated and we believe it caused collapse of some of the internal pores of the fiber and lower fiber permeance than expected. After this issue was identified, the intermediate drying step was eliminated, and the fibers were transferred directly from the final IPA wash bath into the crosslinking bath. Before heating the crosslinking bath, which was started immediately in previous runs, a soaking time of 24 hours was implemented to allow IPA to diffuse out of the fiber bore and the crosslinking solution to diffuse into the fibers. These changes improved fiber permeance and strength.

We operated both fiber spin lines in parallel and that doubled the fiber production capacity. **Table 2** summarizes our fiber spinning progress. As of 31 March 2020, we had spun and crosslinked over 150 km of fibers in total for module production to use in the skid.

Table 2. Fiber spinning progress in 2019 to reach goal of 100 km of fibers spun. Additional fibers were spun in 2020 with new dope formulations.

Spin Line	Batch	Total Fiber Length (km)	Cumulative Fiber Length (km)	Date Spun	Post-treatment Completed Date
1	**100A**	1.5	1.5	7/11/2019	7/18/2019
1	**100B**	1.5	3.1	7/17/2019	7/18/2019
1	**100C**	1.5	4.6	7/22/2019	8/8/2019
1	**100D**	1.4	6.0	7/26/2019	8/8/2019
1	**100E**	0.5	6.5	7/26/2019	8/8/2019
1	**100F**	1.4	7.9	7/30/2019	8/8/2019
1	**100G**	1.3	9.2	7/31/2019	8/23/2019
1	**100H**	1.5	10.6	8/1/2019	8/23/2019
1	**100I**	1.4	12.0	8/8/2019	8/23/2019
1	**100J**	1.5	13.6	8/14/2019	8/23/2019
1	**100K**	1.0	14.6	8/16/2019	10/5/2019
1	**100L**	1.4	16.0	8/19/2019	10/5/2019
1	**101A**	1.5	17.6	9/4/2019	10/5/2019
1	**101B**	1.5	19.1	9/5/2019	10/5/2019
1	**101C**	0.8	19.9	9/12/2019	10/19/2019
1	**102A**	1.7	21.6	10/1/2019	10/13/2019
1	**102B**	2.3	24.0	10/2/2019	10/13/2019
1	**102C**	2.6	26.5	10/3/2019	10/13/2019
1	**102D**	2.5	29.1	10/8/2019	10/19/2019
1	**102E**	2.5	31.6	10/9/2019	10/19/2019
1	**102F**	2.6	34.2	10/10/2019	10/27/2019
1	**102G**	2.5	36.6	10/15/2019	10/27/2019
1	**102H**	2.4	39.0	10/16/2019	10/27/2019
1	**102I**	2.3	41.3	10/17/2019	11/8/2019
1	**102J**	2.5	43.8	10/22/2019	not post-treated
1	**102K**	2.5	46.2	10/23/2019	11/11/2019
1	**102L**	1.8	48.0	10/24/2019	11/11/2019
2	**202A**	0.8	48.8	10/24/2019	not post-treated

Spin Line	Batch	Total Fiber Length (km)	Cumulative Fiber Length (km)	Date Spun	Post-treatment Completed Date
1	103A	2.0	50.8	10/25/2019	not post-treated
2	203A	0.5	51.3	10/25/2019	11/14/2019
1	103B	2.2	53.4	10/31/2019	11/14/2019
2	203B-E	2.0	55.4	11/1/2019	11/14/2019
1	103C	2.3	57.7	11/5/2019	11/25/2019
2	203F	1.6	59.3	11/5/2019	11/21/2019
2	203G	2.0	61.3	11/6/2019	11/21/2019
2	203H	2.3	63.6	11/7/2019	11/21/2019
2	203I	2.5	66.1	11/12/2019	12/2/2019
1	103D	2.2	68.3	11/13/2019	12/7/2019
2	203J	2.3	70.6	11/13/2019	12/2/2019
2	203K	2.3	72.9	11/14/2019	12/12/2019
2	203L	2.3	75.2	11/19/2019	12/7/2019
2	203M	2.0	77.2	11/20/2019	12/2/2019
2	203N	2.3	79.6	11/21/2019	12/12/2019
2	204A	2.0	81.6	11/26/2019	1/14/2020
2	204B	2.0	83.6	12/3/2019	12/17/2019
2	204C	2.0	85.6	12/4/2019	1/10/2020
2	204D	1.9	87.5	12/5/2019	12/20/2019
2	204E	2.0	89.5	12/10/2019	12/20/2019
2	204F	2.1	91.6	12/11/2019	1/14/2020
1	104A	1.0	92.6	12/11/2019	12/18/2019
2	204G	2.0	94.6	12/12/2019	12/19/2019
2	204H	2.0	96.6	12/17/2019	12/24/2019
2	204I	2.1	98.7	12/18/2019	12/25/2019
2	104B	1.9	100.6	12/19/2019	12/26/2019
2	204J	2.0	102.6	12/19/2019	12/26/2019
1	105B	2	104.6	2/11/2020	2/18/2020
2	205D	1.9	106.5	2/12/2020	2/19/2020
2	205A	2	108.5	2/14/2020	2/21/2020
2	205B	1.8	110.3	2/14/2020	2/21/2020
2	206A1	1	111.3	2/21/2020	2/28/2020
2	206A2	1	112.3	2/21/2020	2/28/2020
2	206B	2	114.3	2/17/2020	2/24/2020
1	106A	1	115.3	2/17/2020	2/24/2020
1	106B	1	116.3	2/27/2020	3/5/2020
2	206C	2	118.3	2/25/2020	3/3/2020
2	206E2	1	119.3	3/4/2020	3/11/2020
1	106C	2	121.3	3/5/2020	3/12/2020
2	204F	2	123.3	1/22/2020	1/29/2020
2	204G	2	125.3	2/5/2020	2/12/2020
2	205E	2	127.3	2/6/2020	2/13/2020
2	206E1	1	128.3	3/3/2020	3/10/2020
1	107B	2	130.3	3/11/2020	3/18/2020
1	107A	1.8	132.1	3/12/2020	3/19/2020
2	207B	2	134.1	3/12/2020	3/19/2020
1	107C1	1	135.1	3/16/2020	3/23/2020
1	107C2	2	137.1	3/16/2020	3/23/2020

Potting of fibers into a bundle for lab test:

The HFM module used for the laboratory gas separation performance test was made up of 100 fibers, each with a length of 40 cm, for a total surface area of approximately 690 cm^2. These fibers were placed in a metal sleeve and secured in place using a high-temperature epoxy. The epoxy was also used to create a "dead-end" seal at one end of the module. **Figure 10** shows HFM modules made for the laboratory test.

Figure 10. Potted hollow fiber membrane modules for lab test.

Gas permeation test:

Gas permeation testing was performed at SRI using a bench-scale testing system. The H_2- and CO_2-pure gas were used to measure the pure gas permeance and pure gas selectivity of the fibers. The fiber testing modules were comprised of 100 fibers and the active length of the fibers was 45 cm. The temperature can be adjusted 25 to 250°C and pressure 10 to 200 psig. The system is equipped with a gas flow meter to measure the permeate flux. **Figure 11** a shows photograph of the laboratory test system.

Figure 11. Bench-scale fiber performance testing station.

We tested the performance of fibers produced on Lines 1 and 2 to identify those that would be suitable for use in large modules. The gas permeance measurement was conducted at 200°C and 200 psig. **Figure 12** shows pure gas H_2/CO_2 selectivity and pure H_2 permeance of produced fibers. Every point in the figure represents gas separation performance of a fiber batch, and for each batch we produced ~2 km of fibers.

In **Figure 12**, batches in the red region show low H_2 permeance. Using a design of experiment (DoE) approach, we investigated the root cause of the low permeance. The lower permeance of the fibers in that region is because of pore shrinkage during post-treatment process. Optimizing the fiber washing and drying process during post-treatment of the fibers as mentioned above allowed us to avoid pore shrinkage of the fibers.

The gray area in **Figure 12** presents fibers with relatively high pure H_2 permeance and low H_2/CO_2 selectivity. As the selective layer of PBI HFMs is thin, it sometimes becomes defective during spinning, post treatment, and handling of the fibers for module making, thus the selectivity drops and permeance increases. To avoid defects, we filtered the coagulation bath solution to capture particles that may cause defects when the fibers are not solid in coagulation bath. We also smoothed out the surfaces the fibers come in contact with during the process.

Applying the improvements that we learned in the fiber development process allowed us to spin fibers batches with the desired pure H_2 permeance and H_2/CO_2 selectivity; the performance data are shown by green area in **Figure 12**.

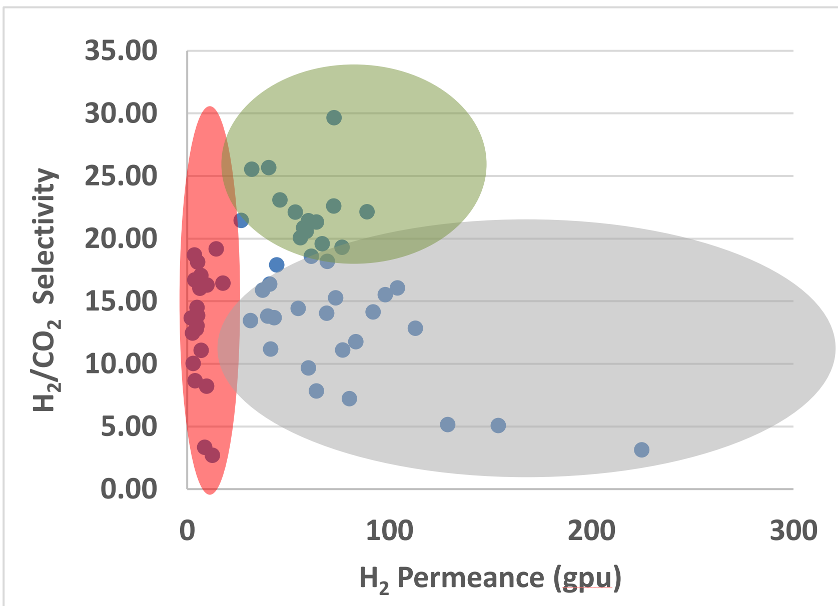


Figure 12. Pure H_2 permeance and H_2/CO_2 selectivity of PBI HFMs batches at 200°C and 200 psig. The red area shows performance of batches with low permeance, and the gray area displays the gas separation performance of batches with low selectivity. The green area shows the performance of batches with desire pure H_2 permeance and H_2/CO_2 selectivity.

We measured the performance of the fibers spun in Lines 1 and 2 to select the fibers suitable for large module making. As an example, the temperature effect on gas permeabilities for H_2 and CO_2 through PBI HFMs (204B) was measured over a temperature range of 120–180°C in the pressure range of 50 to 200 psi (see **Figure 13**). **Figure 14** depicts the permeabilities of CO_2 and

H_2 as a function of inverse absolute temperature at a 200-psi pressure differential. The permeability coefficient of H_2 increases significantly with temperature while that of CO_2 shows only a slight increase or stays flat. Therefore, H_2/CO_2 selectivity increases with temperature (about 30 for 204B at 225°C and 200 psi). The gas transport through a membrane generally follows Arrhenius rule. The data are plotted in Arrhenius form as given in Equation 1 to evaluate the activation energies for H_2 and CO_2 permeation through the membrane.

$$P = P_0 \, Exp\,(-E_a/RT) \qquad (1)$$

where P is the transport coefficient, P_0 is the pre-exponential factor [(cm^3(STP)·cm)/(cm^2·s·cmHg)], E_a is the activation energy of permeation (J/mol), T is the temperature (K), and R is the ideal gas constant (8.314 kJ/(mol·K)). The values of E_a for the transport of H_2 and CO_2 through the PBI HFM were determined from the slopes ($-E_a/R$) of the best curve-fits through the permeation data in **Figure 14** (https://www.ncbi.nlm.nih.gov/pmc/articles/PMC6316465/figure/membranes-08-00132-f007/). Estimated E_a values for H_2 and CO_2 are 20.8 and 17.4 kJ/mole, respectively. The surface area of the HFM (0.055 –in OD, 18-in. length, 100 fibers) used in the lab testing was about 800 cm^2 (~ 0.5 kW$_{th}$).

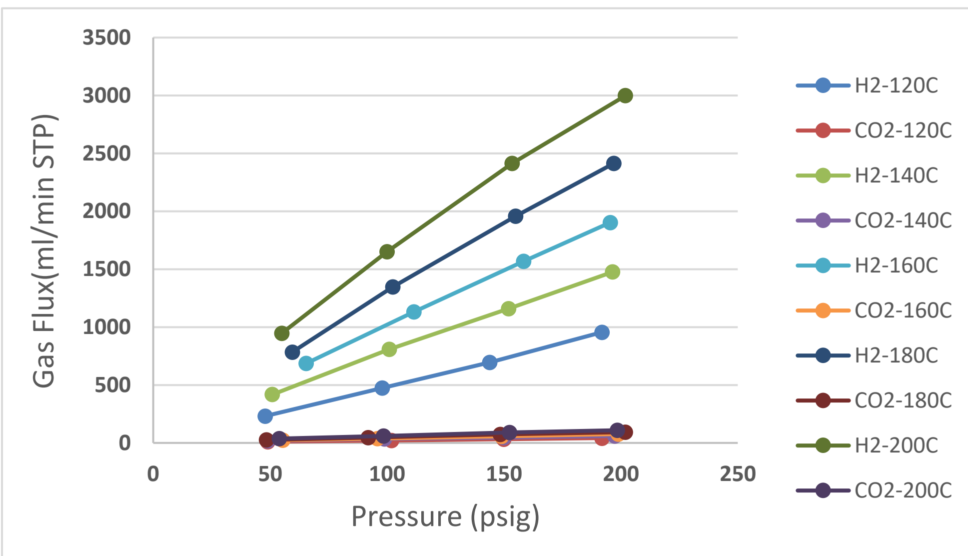


Figure 13. H_2 and CO_2 gas flux of batch 204B at various pressure at 120, 140, 160, 180, and 200ºC.

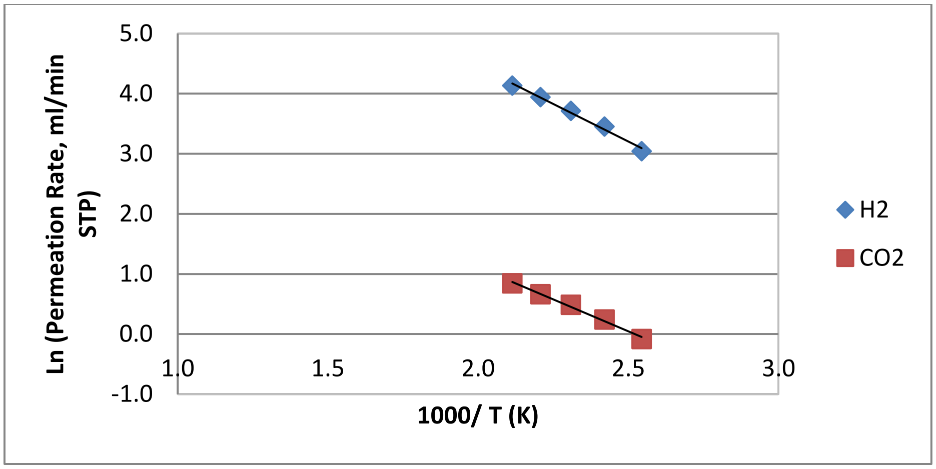


Figure 14. Permeability of H_2 and CO_2 as a function of absolute temperature at 200 psi.

Figure 155 shows the permeance data for fiber lot 108A. Similar data were collected for various fiber batches such as 107D, 107E, 207D, and 207F. The estimated total gas flow rate through the membrane vessel (new design) with fibers from various batches (107D, 107E, 108A, 207D, and 207F) were evaluated and compared with the data from the NCCC campaign in April 2017 that were measured at varying temperatures at a constant pressure (150 psi pressure differential). The measured data for the selected fiber lots was consistent with the performance of the GEN-2 membrane during the NCCC run. These data are presented in **Figure 166** and were derived from permeance plots similar to ones shown in **Figure 15**. The expected average performance per module vessel seems to follow the same trend as observed at the NCCC.

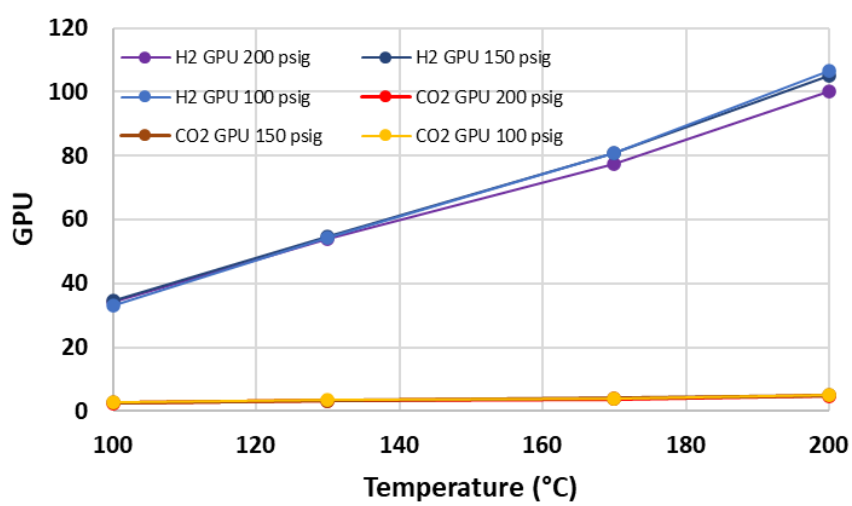


Figure 15. Performance of fiber series 108A from single gas testing.

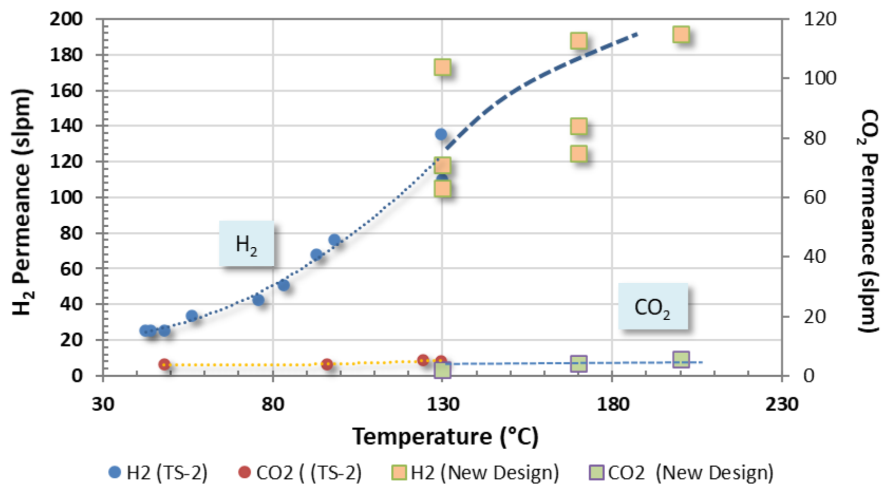


Figure 16. Comparison of expected performance of new vessels compared to the performance observed at the NCCC for the TS-2 module (GEN-2) in 2017. The data for the new design was based on the small element testing in April 2020.

Subtask 2.3 - Module Design and Installation of the Modules (4" diameter)

***Task Description**: During the previous test campaign (FE0012965), the Recipient observed that the gas flow arrangement in the pressure vessels needed to be redesigned to minimize gas bypass and improve performance. The Recipient will address the gas flow design changes to existing 4" vessels and will design and fabricate two new 4" vessels (total of four vessels in the skid). The Recipient will install fiber submodules into the newly designed 4" fiber module assemblies for steady-state and dynamic testing.*

Module Design

New module and vessel designs were considered based on the lessons learned from the previous skid testing at the NCCC. In the new module design, the 9/16-in. stainless steel casing (tube) in the sealing area of the individual elements is extended for the full length of the fibers (28-in.), as shown in **Figure 17**. A set of holes on the side of the tube just below the potting area allows entry and exit of feed gas at each end. The new test module assembly was comprised of 19 fiber bundle tube elements with 9/16-in. OD and 28-in. length. Each tube element contained 175 PBI hollow fibers having an O.D. of 0.55 mm, yielding a 30% fiber packing density. The fibers were potted at each end of the fiber bundle tubes, and the fiber bundle tubes were fixed in a tube sheet array at each end to form a fiber bundle module assembly. The dual O-ring seal arrangement at the end the tube forces the feed gas to flow along the fiber element, minimizing the gas bypass. This arrangement also prevents the fibers from flapping and vibrating under high gas flow as the fibers are confined to a smaller volume inside the 9/16-in. diameter tube. **Figure 18** shows the 4-in. vessel with 19 elements and the sealing arrangement. The new vessel and sealing were designed to accommodate the module assembly, gas feed inlet, and retentate exit changes. The new design was reviewed and approved by the fabricator. The vessels were fabricated, pressure-tested, and shipped to SRI by the vendor in October 2019.

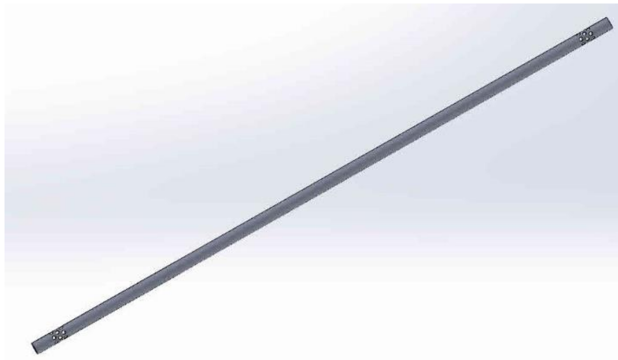


Figure 17. Individual fiber tube element that holds over 175 fibers.

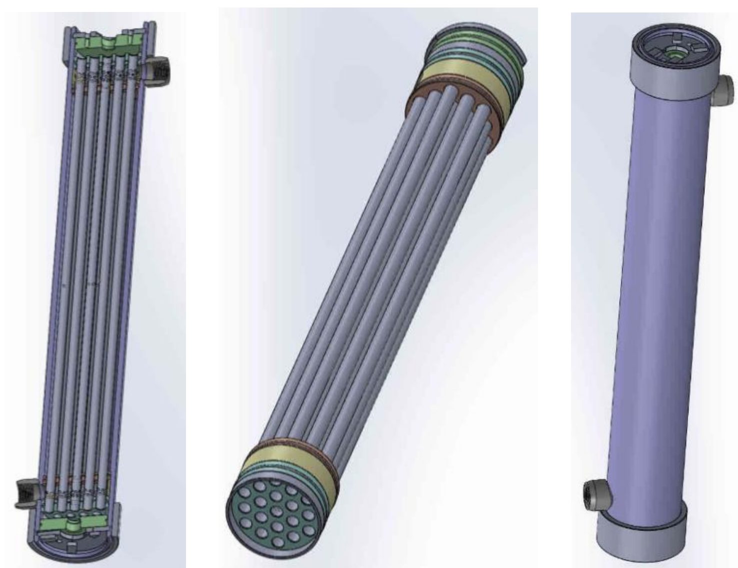


Figure 18. Cross section of the fiber module assembly (*left*). The complete assembly consists of 19 elements (*middle*). New 4-in. fiber module vessel that houses the fiber module assembly (*right*). Syngas gas entry/exit ports are on the sides, and permeate ports are at each end.

Fiber Shrinkage Test

We conducted a brief study on changes in fiber length and diameter under different heating conditions. After heating the fibers to 200°C under nitrogen for periods of 3-16 hours and then cooling over a period of 2 hours, the fiber length decreased by 2-3% (e.g., from 31-7/8 in. to 31-1/4 in.) and the diameter by 1%. However, after leaving the fibers at ambient temperature in air for 24 hours post-heating, the fibers increased slightly in length, e.g., to 31-1/2 in. The two fiber batches tested exhibited similar behavior (within 1%). The changes to fiber diameter with heating are small enough in magnitude that they are not expected to cause leaks between the fibers and the epoxy potting. This type of leak did not occur in long-term testing of the fibers in the skid at the NCCC. In the previous project, changes in fiber length were not problematic because the fiber bundles were only held in place at the open end, and the closed end of the fiber bundles hung free in the reactor. However, the new module design is a *both-ends-open* configuration that requires the fiber bundles to be glued into a metal sleeve at both ends. Therefore, any fiber shrinkage after potting could cause the fibers to break. Based on our findings of the shrinking/relaxing behavior of the fibers during and post-heating, we developed the potting method carefully to pre-shrink the fibers by heating under nitrogen before potting the first end to ensure that some slack remained in the fibers after potting the second end.

Module Making

Fiber modules were prepared for testing in old and new design vessels. The individual fiber bundle elements were potted, assembled, and mounted in the pressure vessel module housing with feed, retentate, and permeate lines connected. The old design with tube elements were composed of GEN-2 fibers left over from SRI's previous PBI project, and they were potted with the *one-end-closed* configuration used in the old design vessel.

Initially, five new design fiber tube elements were potted within a metal sleeve using the *both-ends-open* configuration for testing in the two new reactors on the skid. Each reactor has space for 19 elements; however, since the potting procedure was new to the SRI team, we tested five bundles and the other 14 spaces were blocked off in one of the new reactors to validate the potting method and the tube element design. After the module design satisfactorily demonstrated the expected operability, durability, and leak-tightness in the testing on the skid, the potting-/module-making continued. Fiber batches were selected to make ~25 additional tube elements for shakedown testing. The SRI machine shop produced ~100 metal sleeves to be used in fabrication of fiber tube elements. Module potting was continued with more fibers to build up a strong supply of tube elements for testing at Uky-CAER.

Fiber Module Potting Procedure

The fiber bundles were potted using the method described below.

1. Gather bundles of 175 fibers each. Align the fibers and visually check them for damage or breaks.
2. Heat the fibers under nitrogen to 200°C for 3 hours to pre-shrink the fibers. Potting should begin the same day as the pre-shrink step so that the fibers remain at their shortest length during potting.
3. Prepare the metal tubes: Degrease with acetone, wrap 1-in. on each end with Parafilm so that glue doesn't stick to the outside of the tubes, lightly wax only the edges of the tubes to reduce friction and scraping of the fibers.

4. Slide the fiber bundle through the metal tube, leaving excess fiber length on both ends. Cut one end of the fiber bundle 1-2 in. from the end of the tube, so that all fibers are the same length. This end is potted first.
5. Dip the cut end quickly in hot wax to temporarily seal the fiber bores, so that glue does not penetrate in the fiber bores later. Once the wax cools, remove any excess wax between the fibers so that the fibers are not stuck to each other.
6. Spread the 1-in. of fibers closest to the end with epoxy, making sure that each individual fiber gets coated and there are no dry spots between fibers.
7. Fill a rubber cap halfway with epoxy and put the cap over the ends of the fibers. Clamp the metal tube vertically, so that the rubber cap is at the bottom. Gradually push the cap up over the metal tube until the end of the cap reaches the first set of holes. This step is done slowly so that air can escape the glued end.
8. Lightly squeeze the end of the rubber cap and use a wooden stick through the first set of holes to move the fibers and glue around and dislodge any air bubbles. Glue should be visible through the first set of holes but should not rise to the second and third row of holes, which must remain open for syngas flow.
9. Let the glue dry overnight. The next day, repeat the potting procedure on the other end of the modules. Because a day has passed since pre-shrinking the fibers at 200°C, the fibers may be up to 3/16” longer than the day before. To avoid breakage that could occur if the fibers shrink along their length again after being heated in the skid, some slack should be left in the fibers when potting the second end. An easy way to accomplish this is to fill the rubber caps the day before use with at least ¼ in. of wax or epoxy. The hardened wax or epoxy will push the fibers upward into the module slightly while the second end is being glued, so that the fibers are not stretched within the module once the glue dries.
10. Let the glue on the second end dry overnight. Then, cut the rubber caps off and slice the ends of the glued fiber bundle with a sharp blade so that they are flush with the end of the metal tube. The parts of the fiber that had been blocked with wax will have been removed, and the fibers will be open at both ends. The module is now ready for leak testing.

Figure 19 shows photographs of the potted new design fiber tube elements.

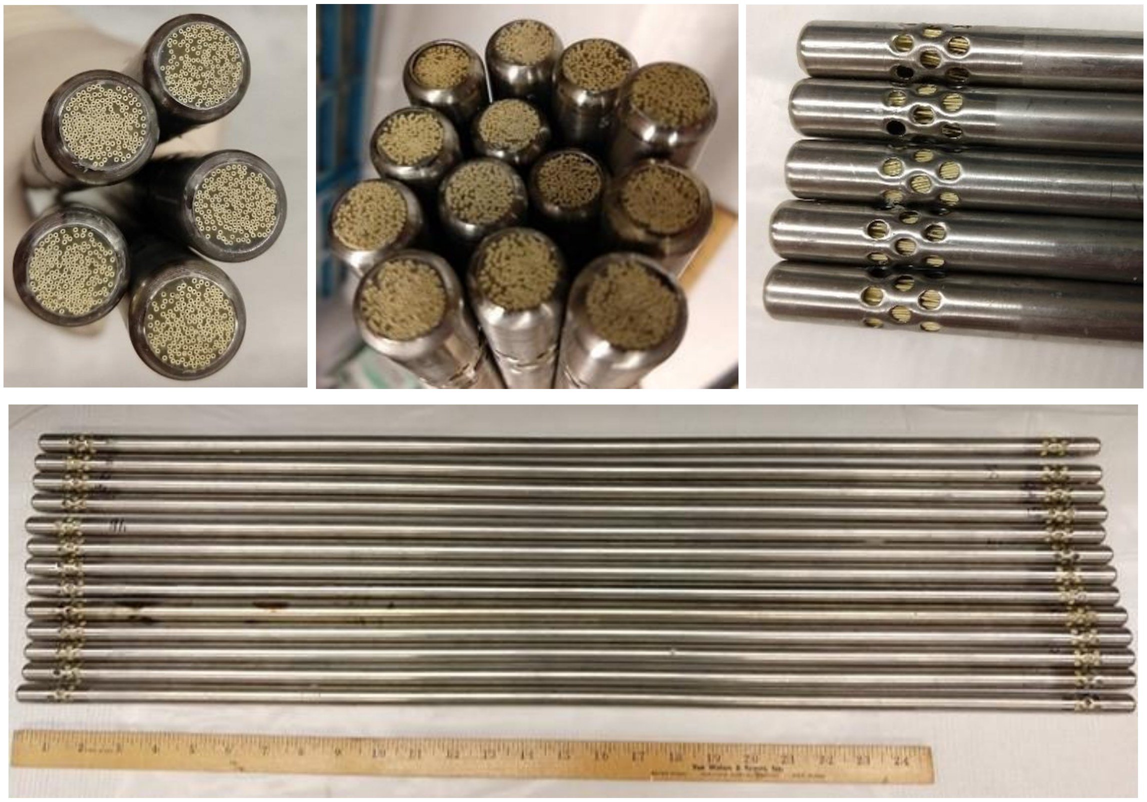


Figure 19. Completed fiber tube elements for installation in the new reactors on the skid.

Module Leak Testing

We designed and constructed a custom leak-testing apparatus that includes adapters for testing modules of both new and old reactor designs (**Figure 20**). This system provided a quick way to screen out modules with broken fibers or potting leaks before investing time to install a tube element into the vessels on the skid. Module elements were tested with 10 psig nitrogen applied to the outside of the fibers and the mass flow rate of the permeate was measured. Because the nitrogen permeability of the PBI fibers is very low, significant permeate flow indicates a leak in the module. The location of the leak can be pinpointed by removing the adapter to the mass flow meter so that the end of the module can be observed visually. For modules with *both-ends-open* configuration, each end was tested individually, with the other end blocked with a cap and O-ring seal. All closed-end and open-end modules were tested and found to be free of leaks. They were installed in the skid for testing.

Figure 20. Leak-testing apparatus for fiber modules.

Module Elements Quality Control

Membrane elements, Old-Style and New-Style, underwent the same quality control (QC) process, as shown in **Figure 21**. To summarize, all membrane cartridges were checked for leaks, and if leaks were found, an attempt was made to repair the cartridge. If the element couldn't be repaired, then the tube element was disassembled, and the element was re-potted with new fibers. If the element passed QC, then it was tested in the fiber skid. After testing in the fiber skid, all cartridges re-examined and QC'd prior to shipping to UKy-CAER.

Potting Delamination Issues

When the New-Style cartridges are subjected to multiple heating and cooling cycles (heated to above 80°C and then cooled to room temperature), the epoxy separates from the stainless-steel housing, resulting in delamination. We observed a similar occurrence in the Old-Style cartridges. The cause of delamination is due to the difference in thermal expansion of the metal sleeve and the epoxy used for potting. To prevent this delamination completely, epoxies with similar thermal expansion properties are needed. However, we did not find a better epoxy than the currently used brand.

While it is possible to repair the delaminated cartridges, the delamination process may occur again after the repaired cartridges are subjected to another heating and cooling cycle. To address this issue and ensure that delamination did not occur during skid test, we kept the fiber skid heated and pressurized for the entire duration of the test cycle. **Figure 22** provides a flowchart of the delamination observed during testing at the SRI campus and the delamination mitigation plan.

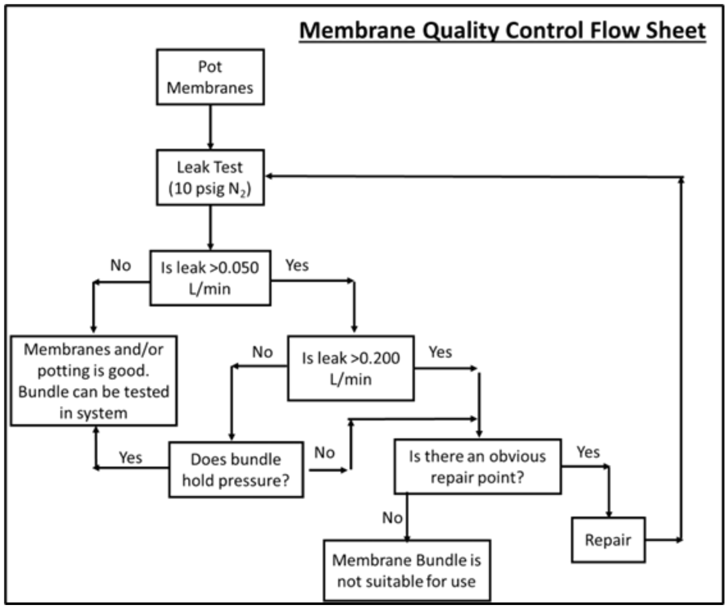


Figure 21. Quality control flow sheet. After potting, all membrane elements were checked for leaks, and if leaks were present, the membrane elements were either repaired or completely rebuilt.

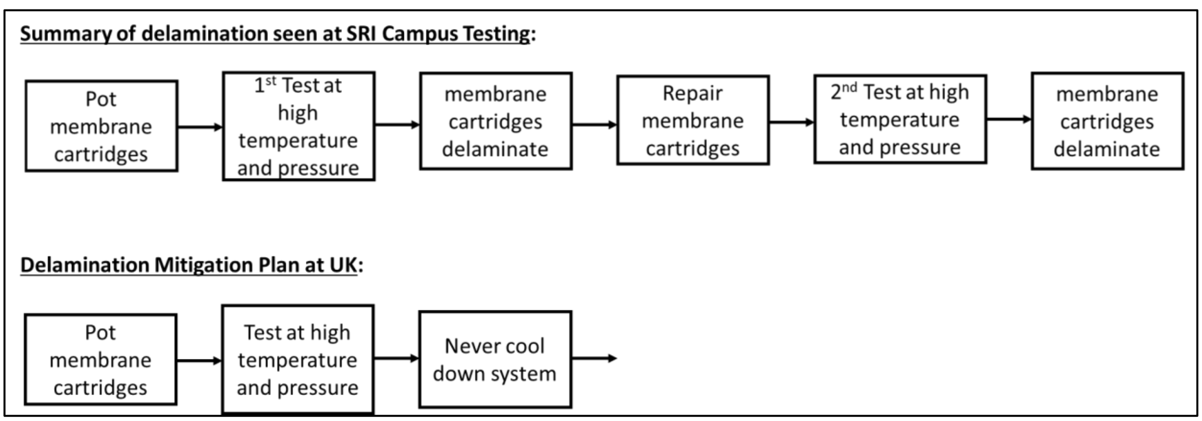


Figure 22. Delamination seen at SRI and mitigation plan to be implemented at UKy-CAER testing.

To repair delaminated the New-Style cartridges, we applied a vacuum to the epoxy plug and applied fresh epoxy to the holes (see **Figure 23**). This procedure has been tested and proven to be an effective method for addressing delamination issues. It involved using vacuum to draw the fresh epoxy through the delaminated area and seal the leaks. This technique can be used multiple times and some of the cartridges underwent this repair process three times before passing the QC test.

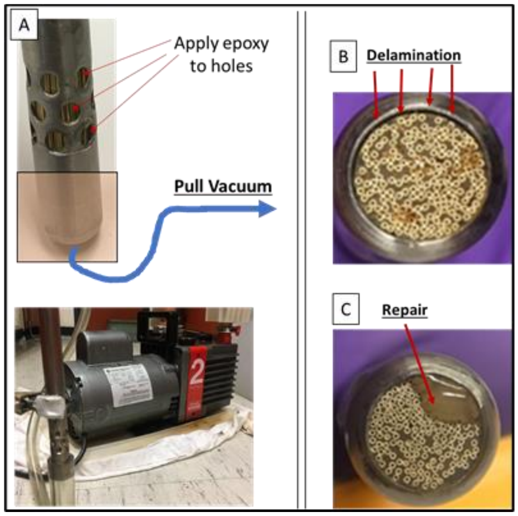


Figure 23. Example of vacuum repair technique on delaminated cartridges. (A) shows where the vacuum and epoxy are applied. (B) shows the delamination, (C) shows how the epoxy is pulled by the vacuum through the delaminated area. (Normally, this much epoxy does not come out; we used an image with excess epoxy for illustrative purposes.)

Subtask 2.4 - Bench Skid Acceptance Testing

***Task Description**: The Recipient will conduct preliminary skid testing for membrane performance using N_2, H_2, and CO_2 gases before shipping the skid and fiber modules to the field test host site. The duration per test may vary between 2 to 5 hours. The main parameter to vary during this testing is the stage cut (the ratio between gas feed flow rate and gas permeate flow rate) at 200℃ and 200 psi of pressure. During acceptance testing, the stage cut will be varied between 0.2 to 0.7 to ensure robust bench-skid operation.*

Shakedown Testing

During the periods ending 12/31/2020 and 3/31/2021, shakedown testing of the modified skid was completed. We tested both the Old-Style module-1 (Old-1) and New-Style module-1 (New-1). The investigation of the potting integrity after use, refurbishment of used membrane elements, and the fabrication of additional new membrane elements were completed. The membrane element potting QC and long-term testing information prior to shipping is described above in subtask 2.3.

Subtask 2.5 - Preliminary Hazard and Operability (HAZOP) and P&ID Review

***Task Description**: The Recipient will provide the existing piping and instrumentation diagrams (P&IDs) to the field test host site so they can conduct a preliminary HAZOP evaluation of the P&ID and also have an understanding of the process utility requirements. Upon completion of bench-skid modifications, the Recipient will send the final BEDP, P&IDs, utility requirements, and electrical load diagrams to the field test host site for final review and incorporation in the field test host site agreement. During this task, the Recipient will provide utility requirements for* *the field test skid to facilitate preparation of the skid installation plan by the field-test host site and incorporation in the field-test host site agreement.*

Field Visit and Meeting Summary

The SRI team visited the field test site at UKy-CAER to see the gasifier and discuss a timeline for the required information exchange, and the skid transfer. We prepared a document containing key information describing the utilities requirements of the PBI skid for UKy. This document included line drawings specifying inlet gas lines, vent lines, and electrical requirements, as well as skid dimensions and layout. We sent the skid information document to UKy in December 2019. This was also forwarded to the DOE project manager as a separate document. UKy and SRI resolved a few issues related to the location of the skid installation and the skid enclosure.

SRI decided to install the skid in a shipping container for ease of transportation, protection from the elements, and to allow the flexibility to locate the skid indoors or outdoors. We purchased a 20-ft container and installed lighting and exhaust fans that comply with Class 1 Div. 2 requirements. We placed the skid in the container and prepared it for transport after completing testing at SRI (**Figure 24** and **Figure 25**).

Figure 24. Skid placement and installation inside the container for shipping to UKy.

Figure 25. Shipping container in which the skid is installed for transportation to the University of Kentucky and field operation.

TASK 3.0 - MODELING OF THE MEMBRANE PERFORMANCE

***Task Description**: The Recipient will update a membrane performance model to determine the optimal module arrangement and predict the performance of the PBI bench skid.*

Summary of Accomplishments

Research work in Task 3 was performed under three subtasks. The subtasks were (1) modeling of the module arrangement; (2) modeling of skid performance; and (3) completion of the preliminary TEA and TMP. These subtasks activities are described in detail below.

Subtask 3.1 - Modeling of the Module Arrangement

***Task Description**: The overall flow distribution within the PBI fiber vessels will be different compared to the previously tested design (information on the design change is given in Subtasks 2.1 and 2.3). The Recipient will model the new bench-skid module arrangement for: (1) flow arrangement (i.e., co-current, counter-current, cross flow, or combination), (2) membrane size (in m^2/tonnes CO_2 or H_2 separated), (3) estimated membrane packing density (membrane area per unit volume in units of m^2/m^3), and (4) estimated pressure drops (in units of bar) for the membrane retentate and permeate under normal operating conditions.*

Module Assembly and Flow Configurations

In BP1, the new module design was modeled by Enerfex to assess the required minimum selectivity and the stage cut required to increase the H_2 recovery to 99%. The variation of module performance due to change in feed flow sides—shell side vs. bore side—was also modeled, including process flow dynamics of the two configurations. The UKy oxygen-blown gasifier-shifted syngas PBI membrane feed composition was used in the simulation for a 6.0 m^2 test module configuration. For modeling purposes, we used the new test module design for testing GEN-2 fibers. As shown in **Figure 26**, the new test module comprised 19 fiber bundle tube elements that have an O.D. of 9/16 in. and a length of 28 in. Each tube element contained 285 PBI hollow fibers having an O.D. of 0.5 mm, yielding ~ 40% fiber packing density. The fibers were potted at each end of the fiber bundle tubes, and the fiber bundle tubes were fixed in a tube sheet array at each end to form a fiber bundle tube module.

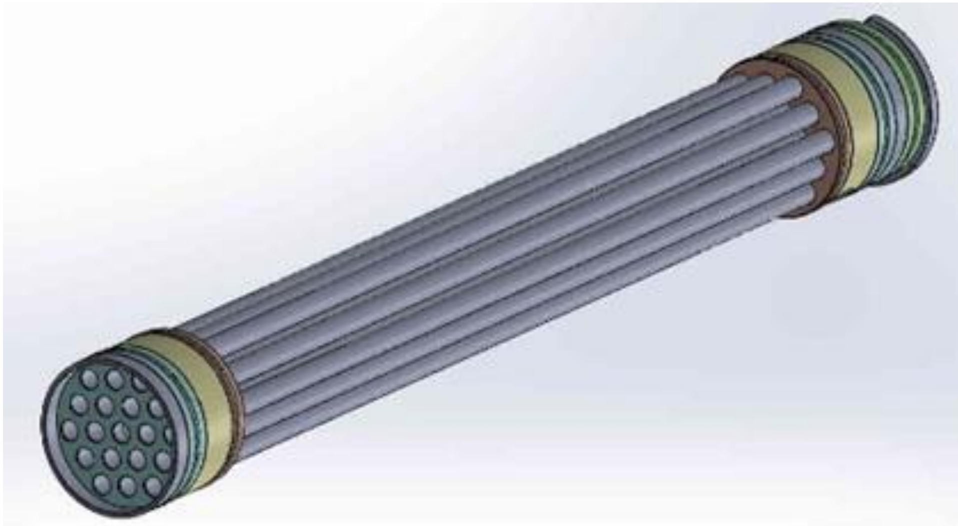


Figure 26. Test module assembly for GEN-2 fibers

The fiber bundle tube module is placed in a pressure vessel housing with feed, retentate, and permeate ports. The test module specifications, modeling parameters, and performance for the given specification and the modeling parameters for two cases considered are given in **Table 3**, **Table 4**, and **Table 5**, respectively.

Table 3. Test module specifications.

Test module specifications:		
Fiber packing density	40%	40%
No. Fibers per tube	285	285
No. of tubes / module	19	19
Effective open tube dia., mm	10.329	10.329
Effective tube length, m	0.7112	0.7112
Membrane area, m^2	6.0	6.0

Table 4. Test module modeled parameters.

Test module modelled parameters:		
Feed pressure, psia	300	300
permeate press., psia	15	15
Feed flow, slpm	198	600
Retentate flow, slpm	85	417
Stage-cut	56.80%	30.50%
Feed to retentate average, slpm	141.5	508.5
Feed to retentate avg. / tube, slpm	7.45	26.76
Selectivity	35	35
H_2 GPU	80	80

Table 5. Predicted test module performance.

Test module modelled perfromance:		
H_2 recovery	98.4%	55.0%
CO_2 capture	90.0%	97.4%
Carbon capture	90.4%	97.5%
Presure drop, psi	0.01	0.11
Tube flow velocity, ft/s	4.86	17.46
Reynolds number	16,904	60,719

Increasing a membrane's H_2 recovery to 99% is the key to achieving the lowest levelized cost of energy (LCOE) with CO_2 capture. Increasing stage-cut increases permeate H_2 recovery but decreases retentate CO_2 capture. Increasing the pressure ratio at the same stage-cut and feed pressure maintains permeate H_2 recovery while increasing retentate CO_2 capture a little over 1%. **Table 6** shows the results from two cases modeled by Enerfex.

Table 6. Predicted test module performance for 99% H_2 recovery.

Case 1:	At 300 psia feed pressure, 99.0% H_2 recovery yields 88.7% CO_2 capture. Stage-cut is 57.3%.
Case 2:	At 200 psia feed pressure, 99.0% H_2 recovery yields 89.8% CO_2 capture. Stage-cut is 57.7%.

In Case 1, the PBI HF membrane's H_2/CO_2 selectivity of 35, a stage-cut of 57.3%, a feed pressure of 300 psia, and a pressure ratio of 13.3 yields 99.00% H_2 recovery and 88.7% CO_2 retentate capture. In Case 2, the pressure ratio is increased to 20 with about the same stage-cut and 200 psia feed pressure, then the CO_2 retentate capture is increased to 89.8% and the H_2 recovery remains at 99.00%. Marginally relieving CO_2 capture from the 90.0% enables 99.0% H_2 recovery and the lowest possible LCOE for the given H_2/CO_2 selectivity. The pressure ratio of 20.0 enables a smaller CO_2 capture relief differential below 90.0% to get 99.0% H_2 recovery compared with the pressure ratio of 13.3. Total carbon capture as CO_2 (after conversion of captured CO and CH_4 to CO_2) for the 13.3 pressure ratio case and the 20.0 pressure ratio case is 89.1% and 90.2%, respectively. Comparison of Cases 1 and 2 shows that lowering the feed pressure from 300 psia to 200 psia while keeping the stage-cut and pressure ratio the same will yield the same CO_2 retentate capture, H_2 recovery, and total carbon capture.

Enerfex also modeled the variation of module performance due to change in feed flow sides; shell side vs. bore side. **Figure 27** shows the schematic of the fiber module with labeling indicating bore side feed. In the shell side feed configuration, feed flow enters through side holes in one end and exits from the side holes at the opposite end. The permeate flow exits from bore side of one end while optional nitrogen sweep gas is introduced from the other end to facilitate the permeate flow.

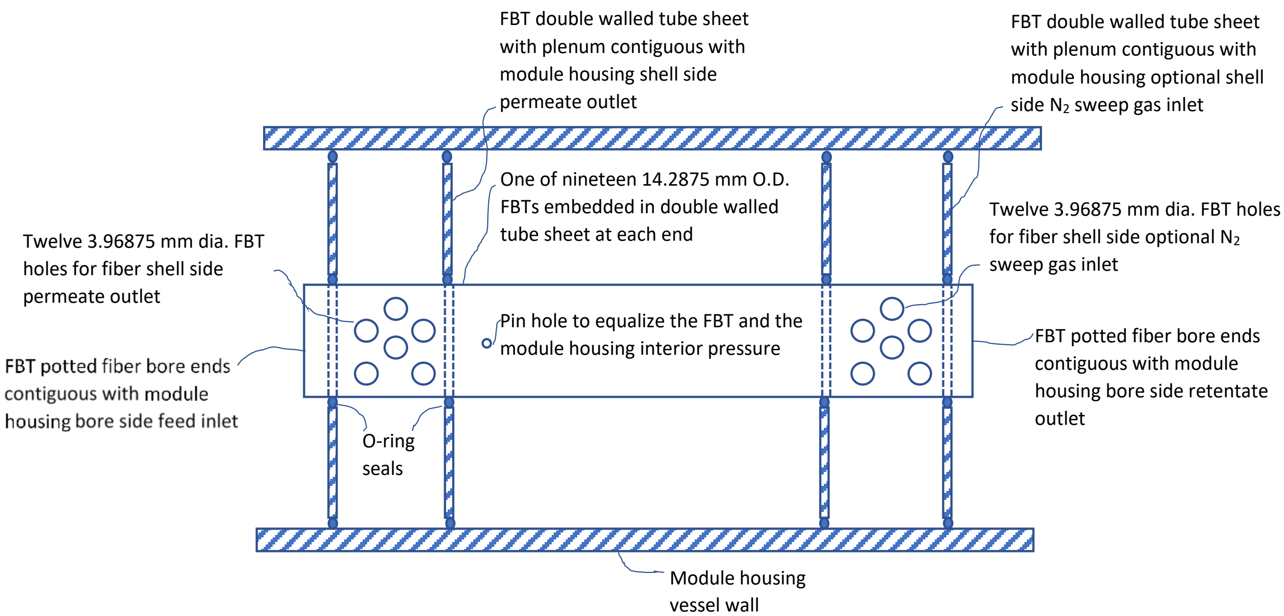


Figure 27. PBI pre-combustion CO_2 capture gas separation test module bore side flow configuration schematic.

In **Figure 27**, each fiber bundle tube (FBT) element contains 285, 0.5 mm O.D., asymmetric PBI hollow fibers with an effective length of 71.120 cm, yielding an effective thin-film shell side surface area of 3,177 cm^2 per FBT. The PBI gas separation module in **Figure 26** with 19 FBT elements would then have a total shell side thin-film membrane surface area of 60,360 cm^2 or approximately 6.0 m^2 of membrane surface area.

The FBT has an O.D. of 14.2875 mm and an I.D. of 13.3350 mm, giving a cross-sectional bore area of 139.590 mm^2. The fiber bundle of 285 fibers of 0.05 mm O.D. in the FBT has a total cumulative cross-sectional area of 55.84 mm^2, yielding a fiber packing density of 40% and leaving 60% of the FBT cross-sectional bore area open, equivalent to a clear and unobstructed tube bore of 10.329 mm I.D. with an 83.754 mm^2 cross-sectional area; accordingly, 83.754/139.590 = 60%.

In the bore side feed, the twelve 3.96875-mm diameter holes at each end of the FBT accommodate the permeate outlet at one end and the optional N_2 sweep gas inlet at the other end; each has a total cumulative cross-sectional open area of 148.374 mm^2. Accordingly, the largest pressure drop will be in the hollow fiber bore side feed to retentate flow side.

Test Module Modeled Performance

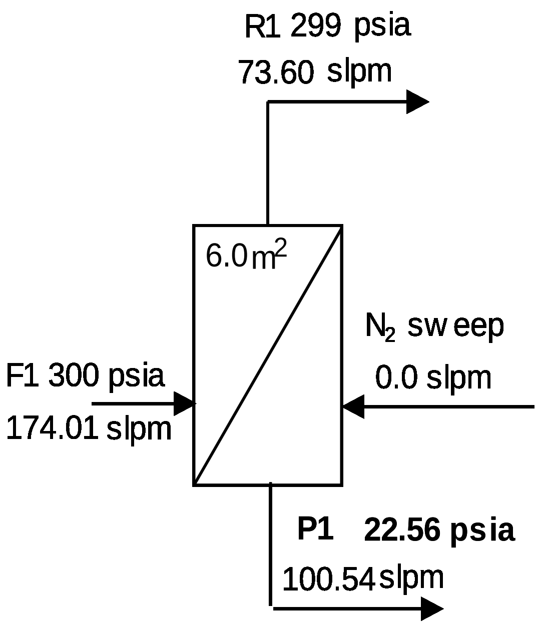
The UKy oxygen blown gasifier shifted syngas composition shown in **Figure 28** was simulated for the 6.0 m^2 PBI membrane feed (F1) test module configuration.

	UKY Comp. SHIFTED
H_2	43.50
CO	2.50
CO_2	41.00
N_2	3.00
H_2O	10.00
	100.00

Figure 28. University of Kentucky oxygen blown gasifier shifted syngas composition (%).

The modeled mass balance and schematic are presented in **Figure 29**. The flow streams R1 and P1 are the carbon capture stream (retentate) and the hydrogen fuel stream (permeate), respectively.

The test module modeled performance shows high H_2 recovery at 99% in the permeate fuel stream. The CO_2 capture in the membrane retentate is 88.7% and increases to 89.1% after residual retentate CO and CH_4 are converted to CO_2 catalytically in a post-membrane treatment process. The test module modeled process parameters show this performance is possible with a PBI H_2/CO_2 selectivity of 35, which is at the low end of the PBI selectivity spectrum with a feed pressure of 300 psia and a pressure ratio of 13.3.



PBI Test Module Specifications:

Fiber packing density	40%
No. Fibers per FBT	285
No. of FBTs / test module	19
FBT open tube dia., mm	10.329
Effective PBI fiber length, m	0.7112
PBI membrane area, m^2	6.0

PBI Test Module Process Parameters:

Feed pressure, psia	300
permeate press., psia	22.56
pressure ratio	13.3
Feed flow, slpm	174.01
Retentate flow, slpm	73.60
Stage-cut	57.77%
Feed to retentate average, slpm	123.81
Feed to retentate avg. / fiber, slpm	0.0229
Selectivity	35
H_2 GPU	80

PBI Test Module Modeled Performance:

H_2 recovery	99.0%
CO_2 capture	88.7%
Total carbon capture*	89.1%

* Includes CO and CH_4 in the feed converted to CO_2

Figure 29. Test module performance.

Process Flow Dynamics of Shell Side Feed versus Bore Side Feed

Table 7 shows the process flow dynamics comparing shell side feed and bore side feed in the PBI test module configuration presented in **Figure 27**.

Table 7. Comparison of dynamic flow parameters for bore side and shell side feeds.

PBI Test Module Modeled Dynamic flow Parameters:

	press, psia	ρ, lb/cf	visc., 10^{-5} Pa s	slpm	alpm	vel., ft/s	Ren. Nbr.	Δp, psi
FBT shell side feed	300	1.799	2.318	6.52	0.583	0.38	1489	0.00
Fiber bore* side permeate	22.56	0.023	1.835	0.019	0.035	27.08	50	6.47
* Bore I.D. = 0.5 mm								
	press, psia	ρ, lb/cf	visc., 10^{-5} Pa s	slpm	alpm	vel., ft/s	Ren. Nbr.	Δp, psi
Fiber bore* side feed	300	1.799	2.318	0.0229	0.0020	1.55	176	0.40
FBT shell side permeate	22.56	0.023	1.835	5.29	9.93	6.48	411	0.00
* Bore I.D. = 0.5 mm								

In the case of the FBT shell side feed, the fiber bore side permeate flow of 0.035 alpm results in a 6.47 psi pressure drop; the pressure drop will be higher if the N_2 sweep gas option is used. In fact, a pressure drop of 19.54 psi was calculated for the fiber bore side permeate when a N_2 sweep at 50% of the permeate volume flow was used.

In the case of the fiber bore side feed, the FBT shell side permeate flow of 9.93 alpm results in a 0.00 pressure and provides an allowance, if needed, for increased pressure drop when the N_2

sweep gas option is used. In fact, a pressure drop of 0.00 psi was calculated for the FBT shell side permeate case when a N_2 sweep gas at 50% of the permeate volume flow was used.

In the fiber bore side feed case the pressure drop is 0.40 psi. Accordingly, the fiber bore side feed/FBT shell side permeate flow pattern offering the lower permeate pressure drops is the more favorable flow scheme.

Subtask 3.2 - Modeling of Skid Performance

Task Description*: The Recipient will model the performance of the bench skid with the gas composition from the field-test host site. The process energy demand will be estimated. The membrane modeling will be performed in sufficient detail to facilitate process design and predict membrane performance.*

Stream Table

A syngas stream was provided by UKy-CAER for HFM skid testing. We used the syngas composition (shifted and tar removed) provided by UKy shown above in **Figure 28** for the model. **Table 8** shows the gas stream (feed, retentate, and permeate) mass balances for UKy-CAER gasifier feed syngas composition as predicted by Enerfex modeling.

Table 8. Mass balance for HFM process for two simulations.

Sim 1:	Mass balance for a 6.0 m^2 (65.0 ft^2) test PBI membrane module								
	psia feed =	300	permeate pressure =		22.56	pressure ratio =		13.3	
		CO_2	CO	CH_4	Ar/N_2	H_2	H_2S	H_2O	
		fract	fract	fract	fract	fract	fract	fract	totals
	Feed = FI	41.00%	2.50%	0.00%	3.00%	43.50%	0.00%	10.00%	100.0%
	Retentate = R1	85.97%	5.69%	0.00%	7.00%	1.03%	0.00%	0.24%	99.9%
	Permeate = P1	8.03%	0.16%	0.00%	0.07%	74.54%	0.00%	17.14%	99.9%
		CO_2	CO	CH_4	Ar/N_2	H_2	H_2S	H_2O	totals
	F1, slpm	71.35	4.35	0.00	5.22	75.70	0.00	17.40	174.01
	R1, slpm	63.27	4.19	0.00	5.15	0.76	0.00	0.17	73.54
	P1, slpm	8.07	0.17	0.00	0.07	74.94	0.00	17.23	100.47
		CO_2	CO	CH_4	Ar/N_2	H_2	H_2S	H_2O	
	Retentate capture	88.69%	96.20%	n/a	98.71%	1.00%	n/a	1.00%	
	Permeate recovery	11.31%	3.80%	n/a	1.29%	99.00%	n/a	99.00%	
	total carbon capture	89.12%	shell side pressure drop, psi			0.013889			
	stage-cut	57.74%	shell side avg. velocity, ft/s			5.98			
	feed flux, cm^3/s/cm^2		0.0480						
	retentate flux, cm^3/s/cm^2		0.0203						
	permeate flux, cm^3/s/cm^2		0.0277						
Sim 2:	Mass balance for a 6.0 m^2 (65.0 ft^2) test PBI membrane module								
	psia feed =	200	permeate pressure =		15	pressure ratio =		13.3	
		CO_2	CO	CH_4	Ar/N_2	H_2	H_2S	H_2O	
		fract	fract	fract	fract	fract	fract	fract	totals
	Feed = FI	41.00%	2.50%	0.00%	3.00%	43.50%	0.00%	10.00%	100.0%
	Retentate = R1	86.11%	5.70%	0.00%	7.01%	1.02%	0.00%	0.23%	100.1%
	Permeate = P1	8.05%	0.16%	0.00%	0.07%	74.66%	0.00%	17.16%	100.1%
		CO_2	CO	CH_4	Ar/N_2	H_2	H_2S	H_2O	totals
	F1, slpm	47.56	2.90	0.00	3.48	50.46	0.00	11.60	116.01
	R1, slpm	42.18	2.79	0.00	3.44	0.50	0.00	0.11	48.98
	P1, slpm	5.38	0.11	0.00	0.04	49.96	0.00	11.49	66.92
		CO_2	CO	CH_4	Ar/N_2	H_2	H_2S	H_2O	
	Retentate capture	88.68%	96.20%	n/a	98.71%	0.99%	n/a	0.99%	
	Permeate recovery	11.32%	3.80%	n/a	1.29%	99.01%	n/a	99.01%	
	total carbon capture	89.11%	shell side pressure drop, psi			0.006667			
	stage-cut	57.69%	shell side avg. velocity, ft/s			3.99			
	feed flux, cm^3/s/cm^2		0.0320						
	retentate flux, cm^3/s/cm^2		0.0135						
	permeate flux, cm^3/s/cm^2		0.0185						

Subtask 3.3 - Preliminary TEA and Technology Maturation Plan

Task Description*: The Recipient will prepare and submit a Preliminary TEA in accordance with SOPO Appendix A. The Recipient will prepare and submit a Technology Maturation Plan (TMP) in accordance with SOPO Appendix B.*

Preliminary Techno-Economic Analysis (TEA)

A preliminary TEA was developed following the analysis documented in the NETL report "*Cost and Performance Baseline for Fossil Energy Plants - Volume 1b: Bituminous Coal (IGCC) to Electricity (Rev 2b, July 31, 2015.* The pre-combustion capture technology analyses followed Case B5B. The preliminary TEA was submitted to DOE as a separate document. **Table 9** summarizes results comparing the baseline CO_2 capture technology (B5B) with PBI membrane-based capture.

Table 9. Technology comparison of baseline CO_2 capture with PBI membrane capture.

Case Name	IGCC-B1A Baseline No Capture	IGCC-B5B Baseline CO_2 Capture	SRI PBI Case 1 Membrane CO_2 Capture	SRI PBI Case 2 Membrane CO_2 Capture
CO_2 removal	No	Selexol	PBI membrane	
CO_2 purification	No		Yes	
Sulfur removal	Sulfinol	Selexol		
Performance and Economic Summary				
H_2/CO_2 Selectivity	n/a	n/a	40	50
H_2 GPU	n/a	n/a	80	80
CO_2 capture	n/a	92.25%	90.04%	90.02%
CO_2 purity	n/a	99.48%	95.62%	95.57%
H_2 recovery	n/a	99.98%	96.10%	99.64%
HHV plant efficiency	42.10%	32.60%	32.61%	34.39%
LHV plant efficiency	43.70%	33.80%	33.81%	35.65%
COE w/o T&S ($/MWh)	$107.10	$135.30	$120.76	$117.74
COE w/ T&S ($/MWh)	$107.10	$144.50	$129.96	$126.94
% Increase in COE	0.00%	34.92%	21.34%	18.52%

The integrated gasification combined cycle (IGCC) baseline case without CO_2 capture removes sulfur with a Sulfinol H_2S removal unit. The gas turbine fuel in that case is unshifted syngas. The IGCC baseline case with CO_2 capture uses a two-stage Selexol for CO_2 separation and H_2S removal. The two modeled SRI cases use the PBI membrane for CO_2 removal, and Selexol for H_2S removal. The Case-1, SRI PBI membrane 40 selectivity (upper limit for GEN-1) case is based on the current development status of the bulk PBI HF membrane. The Case-2, SRI PBI membrane 50 selectivity (upper limit for GEN-2) is the highest value measured on small-batch PBI HF parametric tests. Plants with Case-1 and Case-2 SRI PBI membrane systems recover 96.10% and 99.64% of the feed H_2 in the permeate, respectively. Thus, H_2 loss to the retentate is substantially reduced in Case-1 and Case-2, avoiding the need to recycle H_2 from a downstream CO_2 purification step with its attendant loss of captured CO_2 and giving a capture rate of less than 90%.

The main focus is to find a pathway for overall fossil energy performance goals of CO_2 capture with 95% CO_2 purity at a COE of 30% less than baseline capture approaches. Therefore, we modeled several additional cases with varying H_2/CO_2 selectivities and/or H_2 permeances and evaluated the corresponding COE. The data from this evaluation is presented in **Figure 30**. This analysis indicates that a PBI HFM with a selectivity of H_2/CO_2 of about 35 at H_2 permeance at 80 GPU is required to meet the DOE goals.

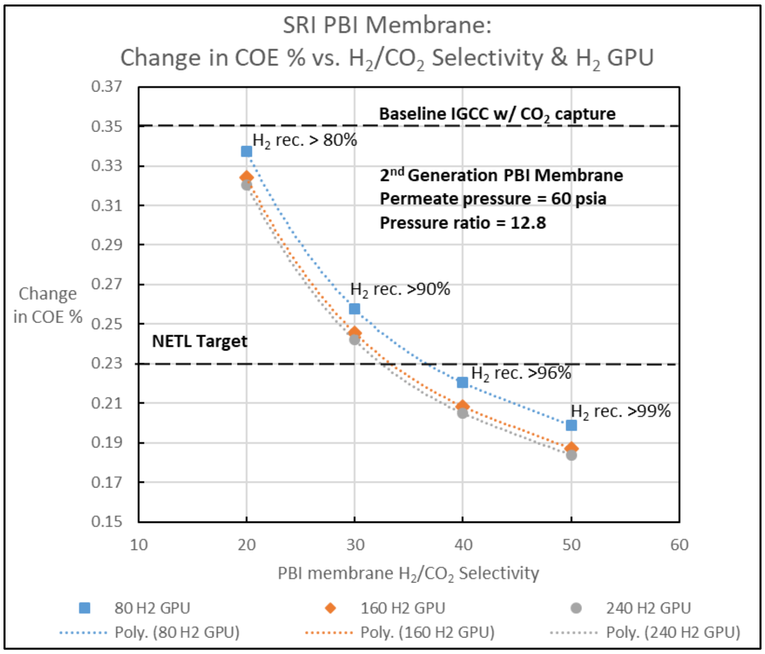


Figure 30. Change in COE as a function of H_2/CO_2 selectivity and H_2 permeance.

Preliminary Sensitivity Analysis

A preliminary sensitivity analysis was conducted to determine the minimum COE by adjusting CO_2 capture efficiency, which increases H_2 recovery. Based on the NETL technology screening report (DOE/NETL-2015/185), the estimated cost of CO_2 capture for an SRI PBI HF membrane-based capture technology is $39.20/tonne-$CO_2$. The 90% CO_2 capture established by NETL has been the performance goal parametric for CO_2 capture since the inception of the SRI PBI CO_2 capture membrane project. NETL now believes there may be overall performance benefits if the 90% capture is relieved or marginally lowered. However, CO_2 capture marginally lower than 90% must still capture CO_2 at a minimum purity of 95% and a minimum 30% reduction in LCOE compared with baseline capture LCOE.

Lowering the 90% CO_2 capture goal to between about 82% to 88% will enable higher H_2 recovery and, as a direct consequence, lower LCOE with lower H_2/CO_2 selectivity. Four PBI membrane selectivities, 20, 30, 40, and 50, were simulated to yield 99.7% H_2 recovery, and returned resultant CO_2 capture rates for each selectivity. Except for the 50 selectivity, which returned a 90.1% CO_2 capture rate, the selectivities of 20, 30, and 30 returned CO_2 capture rates of 76.3%, 83.9%, and 87.8%, respectively. Each selectivity had a LCOE about 32.9% less than the baseline capture LCOE. A summary plot of the simulation results is given in **Figure 31**.

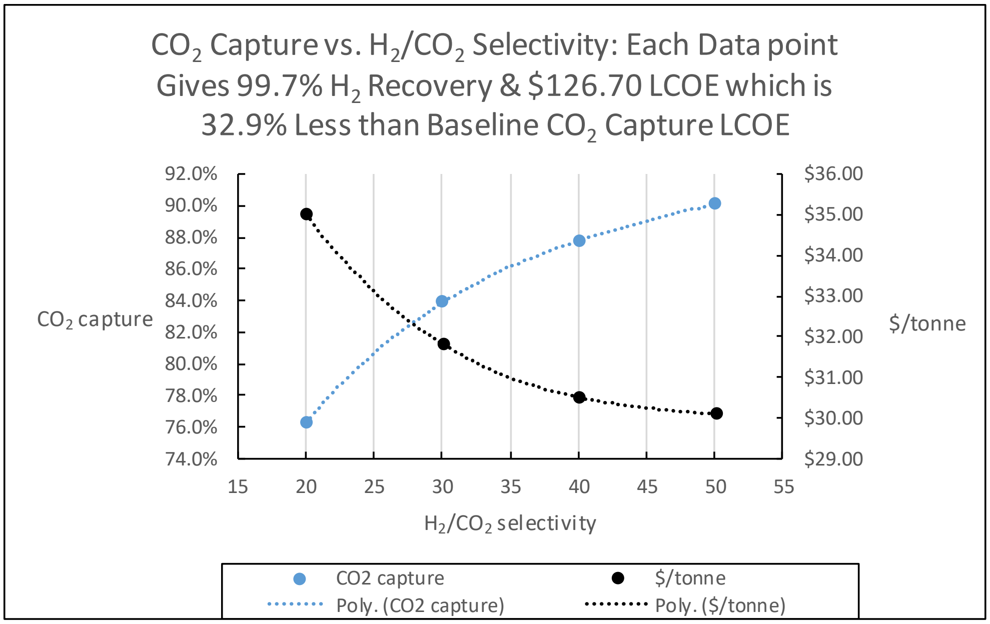


Figure 31. Summary of four PBI membrane simulation results.

As expected, the \$/tonne CO_2 captured trend is an exact inverse of the %CO_2 capture trend, i.e., as the %CO_2 captured decreases, the cost in \$/tonne-CO_2 capture increases in direct proportion. **Table 10** tabulates three selectivity data points in the plot: the selectivity at which the two plots intersect, and selectivities of 35 and 40.

Table 10. Three data points from **Figure 31** above.

Selectivity	27.8	35	40
CO_2 capture	82.40%	86.00%	87.80%
$/tonne	$ 32.46	$ 31.00	$ 30.50

From **Table 10**, it appears a selectivity of 35 would be optimal in terms of a reasonable average selectivity attainment in bulk fiber production and a reasonable (marginally lower) 86% CO_2 capture. The captured CO_2 progressive stream composition after membrane separation, H_2S removal, catalytic oxidation, and H_2O knock-out will yield a final captured CO_2 purity of 96.66%.

BUDGET PERIOD CONTINUATION

Federal Government Requirement

In accordance with the "Continuation Application and Funding" article in this Cooperative Agreement, DOE funding is not authorized beyond Budget Period 1 without the written approval of the Contracting Officer. DOE's decision whether to authorize funding for Budget Period 2 is contingent on (1) availability of funds appropriated by Congress for the purpose of this program; (2) the availability of future-year budget authority; (3) substantial progress towards meeting the objectives of your approved application; (4) submittal of required reports and other deliverables; and (5) compliance with the terms and conditions of the award.

DOE Approval

SRI completed all proposed activities for BP1 satisfactorily and requested DOE approval to proceed to BP1 with no change in scope. DOE approved the request, and the project continued to BP2.

BUDGET PERIOD 2 (BP2) RESEARCH ACTIVITIES

SUMMARY

The main activities performed in BP2 were carried out under Tasks 4, 5, and 6. These activities included installation of the skid at UKy site, operation of the skid with syngas under dynamic and steady-state conditions, demonstration of long-term stability of the fiber module under field conditions, and the technology assessment for future scale-up and technology maturation.

TASK 4.0 - OPERATION OF BENCH SKID AT FIELD TEST HOST SITE

***Task Description**: The Recipient will install the modified bench skid at the field test host site and complete testing activities. Efforts include bench skid transfer from the Recipient's facilities to the field test host site, bench skid installation, bench skid shakedown testing followed by system operation, and data collection and analysis.*

Summary of Accomplishments

Research activities in Task 4 were conducted under four subtasks that included skid installation at the site, development of a test plan, operation of the skid with actual syngas under dynamic and steady-state conditions, and data analysis. These activities were successfully completed with over 800 hours of skid operation, and the details are discussed below under respective subtasks.

Subtask 4.1 - Bench Skid Transport and Installation

The Recipient will transport the modified bench skid to the field test host site. The field test host site will be prepared for bench skid installation. The modified bench skid will be installed, and an assessment will be conducted to evaluate and update needs/requirements to test the skid at the host site. Initial shakedown testing of the modified bench skid will be conducted with inert gases.

The Recipient will address any required bench skid modifications based on the findings from Subtask 2.5 to complete skid installation. The Recipient will prepare two spare fiber elements. The Recipient will review the standard operating procedures (SOPs) and HAZOP/EH&S findings prior to preparing a field test plan. The Recipient will use available data relating to on-site conditions, process design conditions, feedstock, products, utilities, noise, measurement units, required codes and standards, and the specifications of effluents and waste streams in the preparation of SOPs and HAZOP evaluations.

Skid Installation

SRI coordinated with UKy for the skid transport and installation schedule. SRI contracted crane services for loading/unloading the skid and worked with the UKy staff for installing utility connections. **Figure 32** shows a picture of the skid being lifted to the foundation and the installed skid at the site at UKY with utilities connected.

Figure 32. Photographs of the skid installation at UKy.

Membrane Module Installation

We installed membrane module elements in the vessels because they were shipped separately to avoid damage during transportation. After completing the installation, leak tests were performed to ensure all seals were tight.

Stability of Membrane Module Elements in Long-term Testing

We sometimes experienced delamination problems with the New-Style membrane cartridges. When the New-Style cartridges are put through repeated heating/cooling cycles (heated to >80°C and then cooled to RT), the epoxy delaminates from the stainless-steel housing. Although we can successfully repair these cartridges, the delamination process sometimes repeats itself when repaired cartridges go through another heating/cooling cycle. To mitigate this issue and assure no delamination, we plan to leave the fiber skid heated and under pressure for the complete duration of the test cycle. Once the skid is up and running at UKy-CAER, we do not plan on cooling it down unless we need to change cartridges or there is a prolonged break in testing. **Figure 33** gives a flow sheet of the delamination seen during SRI campus testing and of the delamination mitigation plan. To test our mitigation plan, we conducted a 36-hour testing at SRI. We tested five different types of cartridges: (1) Old-Style, (2) Old-Style repaired, (3) New-Style, (4) New-Style vacuum repair, and (5) New-Style O-ring repaired. The Old-Style cartridges were shown to withstand thermal cycling without delaminating, while maintaining separation properties. Because of this, only the New-Style modules were tested long-term. **Figure 34** shows the membrane performance of repaired and new cartridges after being held in CO_2 at 100 psig and 125 °C for 72 hours. The test consists of: (1) 4x new cartridges, and (2) 2x vacuum repaired and 2x O-ring repaired cartridges. The results showed the membranes remained stable for 72 hours with high selectivity (>35) and did not delaminate. The small number of cartridges used, only 4x membranes per test, was to minimize the number of cartridges that would need to be repaired.

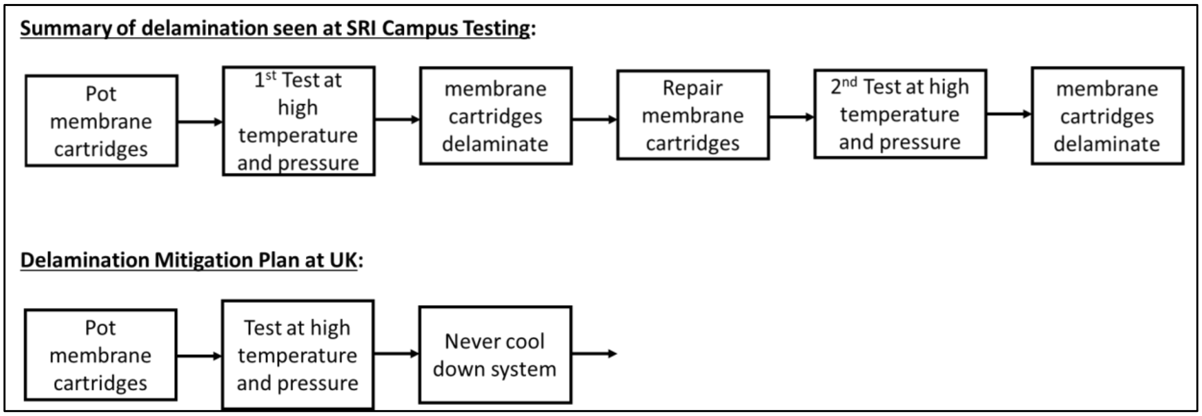


Figure 33. Summary of delamination seen at SRI Campus testing (top), and mitigation plan to be implemented at UKy-CAER testing (bottom).

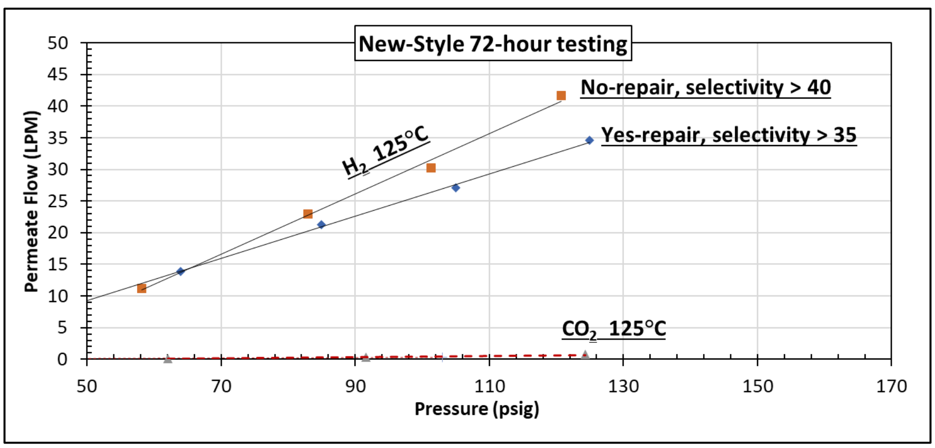


Figure 34. New-Style membranes remained stable for 72 hours without delamination. Selectivity remained >35 at 125°C and 120 psig.

Subtask 4.2 - Development of a Test Plan

The Recipient will develop a robust test plan to obtain the required experimental data during parametric and steady-state testing with coal-derived syngas at the field test host site (Subtask 4.3). The major process variables will be the feed gas flow, membrane temperature (100-225°C), trans-membrane pressure (100-300 psi range), and stage cut (0.2 to 0.7 range). The test plan will define the performance evaluation of the bench-scale fiber skid and membrane performance targets, and how the plan supports the DOE program goals.

Test Plan

SRI planned to perform testing on a modified bench skid using coal and natural gas-derived syngas at the UKy field host site. The major process variables included feed gas flow, membrane temperature, feed pressure, and stage cut. The test plan defined the performance evaluation targets for the skid and how it supports the goals of the DOE program. The test was submitted to the DOE Program Manager (PM) as a separate document. The following is a summary of the test plan.

Test Parameters

The objective of the planned testing is to demonstrate the PBI membranes 50 $kW_{thermal}$ skid performance at a field setting and multiple cycles. The parameters to be varied included gas composition, gas feed rate, pressure differential, temperature, stage cut, and the membrane morphology including the membrane dense-layer thickness. A sample parametric matrix for generating data for the performance database is given in **Table 11**.

Table 11. Sample parametric matrix for generating data for the performance database.

Test Parameter	Range	Unit
Temperature	100 to 200	°C
Pressure	50 to 200	psig
Gas composition	Variable	slpm
Stage cut	0.2-0.9	
Selectivity	10 to 30	

Membrane Performance Evaluation Criteria

The membrane formulation and configuration in the bench-scale system was planned to be evaluated as follows:

- Hydrogen separation efficiency and hydrogen flux of the membrane during testing in constant multi-gas loading (calculated at the gas inlet and outlet locations to the membrane unit).
- Measurement of flux and separation factor as a function of gas composition and contaminant level.
- Mechanical and physical properties of the fresh and exposed membranes (e.g., morphology changes and chemical degradation resistance).

Testing Protocol for the Skid

Given below is the overall membrane testing process:

- A leak test was conducted on each membrane to be tested.

- Hydrogen flux was determined at constant temperature and flow conditions over a 4- to 8-hour period and reported as an hourly average.
- Each membrane was tested to 120 hours to determine whether there were long-term performance or structure changes.

The test schedule was planned for each campaign with 2 weeks of testing followed by data analysis. A total minimum of 600 hr. of testing with syn gas was scheduled.

Data Collection, Analysis, and Reporting

The following measurements are made and reported as a minimum for each test condition.

- Gas compositions and flows are reported as hourly averages for a 4- to 8-hour test period.
- Flow rates are reported in slpm, temperatures in °C, and pressures in kPa and psia.
- Analyses are described in detail sufficient to allow the reader to recalculate the results from experimental data.
- A comprehensive analysis of data is documented in quarterly reports to DOE.

Subtask 4.3 - Operation of the Skid and Data Collection

The Recipient will operate the modified bench skid for 300-500 hours, depending on field test host site availability. Parametric tests will be performed prior to steady-state testing at the optimal process conditions. The field test host site will provide the syngas from an oxygen-blown gasifier with the shifted gas composition (H_2:40-45%, CO: 2-3%, CO_2:40-42%, N_2:2-4%). The maximum syngas flow rate is 40 m3/hr. The syngas will be delivered to the modified bench skid at pressures up to about 30 bar. Permeate and retentate gas flow compositions will be monitored by the field test host site using a two-channel gas analyzer interfaced with process control hardware and software.

Field Testing

SRI team made three trips to the field test host site, accumulating over 800 hours of testing time and collecting data using both coal and natural gas-fed syngas. The skid was operated continuously without any loss in performance. Gas flow compositions were monitored using a two-channel gas analyzer and process control hardware and software. The results were analyzed to evaluate the fibers' performance in different conditions. Also, because of some issues with syngas feed (e.g., water in the line) that happened during our trips, the skid was modified to include water traps to prevent damage to the fibers during the process.

First Trip

During the first trip, the start-up procedures for the skid were successfully tested and the skid was operated for 1 week without any issues. However, the gasifier at the field test host site experienced some issues, and syngas was not available for the second week of testing. There were no problems with the skid itself during this gasifier down time.

The UKy gasifier is designed to be operated for 8 hours during the daytime and to be in standby mode overnight. However, we operated the skid 24/7 by shifting syngas to N_2 overnight and collecting data for assessing the longer-term temperature and pressure stability of the skid. During the first series of testing at the host-site (test campaign starting September 7, 2021), we obtained daytime data for ~14 hours with syngas, and we completed 100 hours of total test time including overnight testing without any loss in membrane performance at the selected test temperature and pressure. We obtained 32 data points: 26 data points using natural gas as the feed to the gasifier, and 6 data points using coal as the feed. The syngas feed compositions in this trip are given in **Table 12**.

Table 12. Gas composition using natural gas and coal as gasifier feed.

Natural Gas (Sept. 9th and 10th)

Gas	Mol%
H_2	30.37
N_2	12.94
CO	28.93
CO_2	26.15
H_2O	3.52
H_2S	0.00
COS	0.00

Coal (Sept. 10th)

Gas	Mol%
H_2	10.81 - 19.67
N_2	14.41 - 22.26
CO	9.00 - 14.40
CO_2	42.94 - 55.90
H_2O	3.62 - 4.11
H_2S	< 0.5%
COS	< 0.5%

Skid flooding with gasifier-produced water during 1st trip:

During the second week of testing, an unexpected issue with the gasifier caused the syngas feed line to become flooded with produced water that also entered the SRI skid and partially filled the membrane vessels (images are shown in **Figure 35**). The SRI team spent several days cleaning the affected section of the skid. The NOVA gas analyzer, located upstream of the skid, was protected by a water trap with a built-in safety mechanism that shut off the analyzer, preventing any damage to the sample cell. After the gas analyzer was cleaned, it was tested and found to be operating normally. The flooded sections of the skid were also cleaned and heated to around 190°C with continuous nitrogen flow to remove any remaining produced water. **Figure 36** shows the fibers after taken out from flooded vessels. All control valves and regulators were found to be operating normally. As a precautionary measure to prevent future gasifier failures from introducing unwanted materials into the skid, water traps were installed at various locations (shown in **Figure 37**).

Figure 35. Pictures of the inside of module Old-1 after it was flooded with gasification water. There is a black solid material coating on the walls and at the bottom of the module vessel.

Figure 36. Fibers after being flooded with gasification water. Some fibers are completely coated with a black tar-like substance.

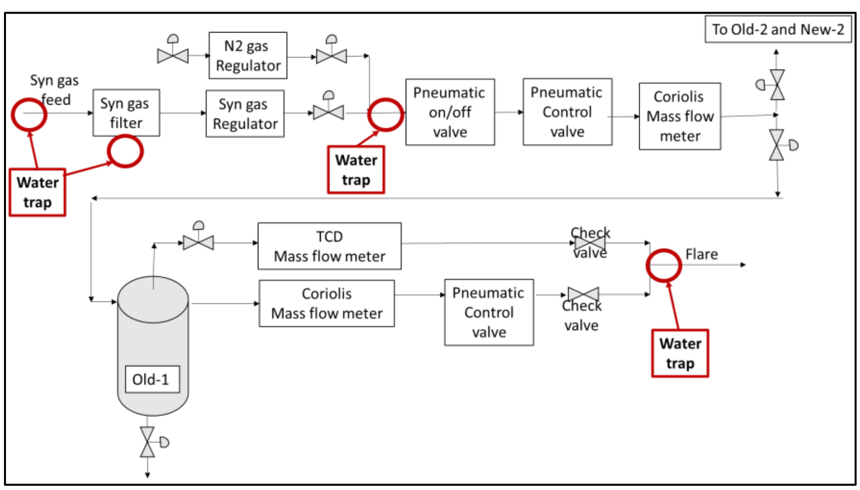


Figure 37. Location of water-trap installations.

Second Trip

During this trip, we collected 45 data points using a combination of coal and natural gas-fed syngas, and we obtained approximately 36 hours of data. The fiber skid operated continuously for over 200 hours without any loss in membrane performance, as shown in **Table 13**. The natural gas-fed syngas provided a more stable syngas feed composition for the fiber skid, but the syngas feed gas composition from coal fluctuated. To accommodate for this changing feed composition, we adjusted our data collection method and were able to capture three times more data, with 32 data points collected per day compared to only 12 using the old method. Unfortunately, we had to end our testing campaign prematurely due to extensive gasification problems at UKy-CAER.

Table 13. Summary of fiber skid operation time.

Feed to skid	1st Trip	2nd Trip	1st + 2nd Trip
Time on nitrogen	82	175	257
Time on syngas (natural gas)	14	18.5	32.5
Time on syngas (coal)	2	17.5	19.5
Total time syngas	16	36	52
Total time hot and pressurized	98	211	309

Our testing with synthetic gas at SRI showed the membrane cartridges were very stable during long-term operation as long as they did not experience large temperature fluctuations, which is typical for glassy polymers. Indeed, these types of polymers are prone to damage from extreme temperature changes. During the second week of the run campaign, the membrane cartridges were unexpectedly exposed to a temperature fluctuation from 150°C to below freezing due to a compressed air failure in the building, which caused an automatic power shutdown of the skid. This temperature fluctuation caused some of the cartridges to experience potting delamination. All of the cartridges were removed from the skid and shipped back to SRI for assessment of any damage to the fibers. The damaged cartridges were repotted and tested for the gas permeation rate at operating pressure.

Updating data collection

During the second trip, the data collection protocol was revised to collect data more frequently and account for the fluctuating syngas composition when coal was used as the feed for the gasifier. The coal-based syngas composition was not stable as shown in **Figure 38**. The original method involved using the NOVA gas analyzer of the skid to measure the composition of three streams—feed, retentate, and permeate—by using valve switching. While this method was accurate, the response time was slow because of the wait time for line flushing and pressure equilibration; these took more than 15 minutes per measurement. The approach also required a stable feed from the gasifier, which was not the case. In the revised protocol, the NOVA gas analyzer was only used to measure the permeate composition, while the feed composition data from UKy-CAER was used to determine the retentate composition. This allowed for a data point to be collected every 10 minutes. The skid operation procedure remained similar to that in the first trip, with the skid being heated and pressurized with nitrogen when not receiving syngas.

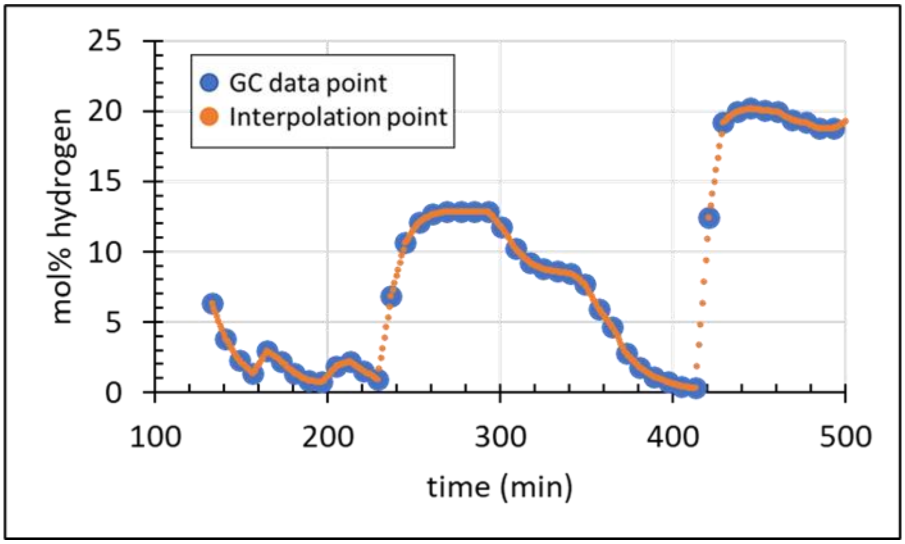


Figure 38. Instability of hydrogen composition in the syngas with coal feed.

PBI membrane module element stability

During the first testing campaign, the membrane module elements (cartridges) were able to maintain high temperatures and pressure for approximately 100 hours. In the second campaign, they were able to maintain these conditions for about 200 hours. When the membrane cartridges were heated, they maintained stability until they were cooled down. The PBI cartridges also demonstrated resilience to feed pressure drops. When syngas was not available, the skid was connected to UKy-CAER's house nitrogen feed, which experienced several instances of rapid pressure drops and recoveries. These events did not result in any leaks or decreases in fiber performance, as shown in **Figure 39**.

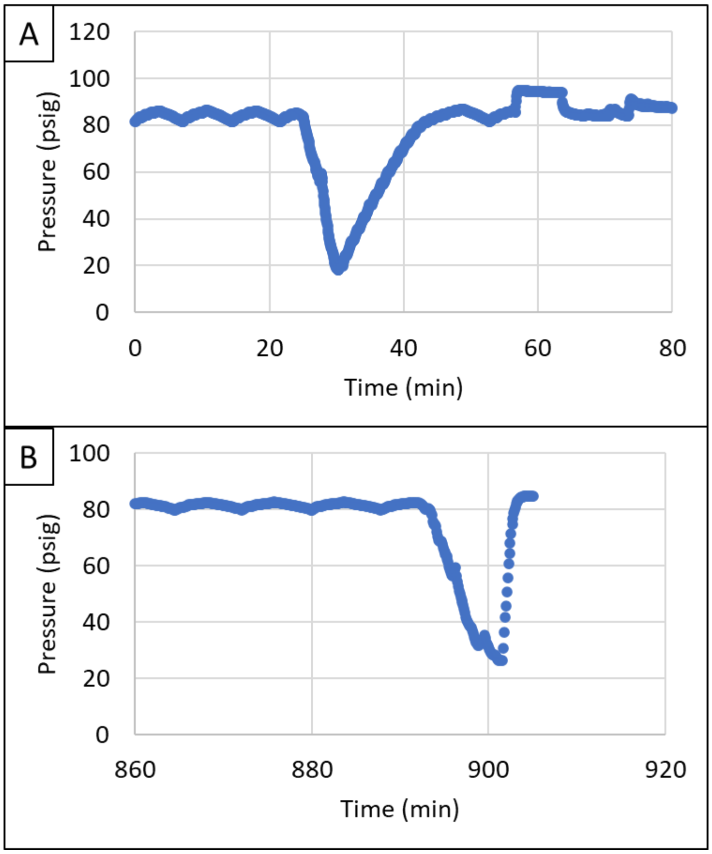


Figure 39. Two examples of the fiber skid experiencing a rapid drop in pressure and then a rapid rise in pressure. None of these pressure fluctuations affect the membrane or cartridge stability.

Third trip

<u>*Preparation of fiber module elements for skid testing.*</u>

We refurbished the fiber cartridges that were damaged in the second trip and confirmed the performance by re-testing them in a laboratory setup. The existing lab-scale system was updated to test both Old-Style and New-Style modules. To quickly evaluate the separation performance of refurbished fiber cartridges, the H_2 and CO_2 permeance of ~60 modules were measured at room temperature and 100 psi. The H_2 permeance of the modules varied from 1 to 100 gpu. Based on the H_2 permeance, modules were categorized into three separate groups with low permeance, high permeance, and leaky modules. The fibers of the leaky modules were removed from the metal sleeve and the sleeves were used to make new fiber module elements. **Table 14** summarizes the number of modules and permeance criteria to select each group.

Table 14. Module performance during screening.

	Number of Modules	H_2 permeance criteria (gpu)
High permeance	22	10 to 40
Low permeance	23	10<
Leaky modules	14	>40

Figure 40 is a histogram plot showing the number of new design modules in each permeance interval. Modules with hydrogen permeance greater than 3 gpu were categorized as useful. Modules that had permeances below 3 gpu required further refurbishing. The permeance measurements were made at room temperature with a 100-psi pressure difference.

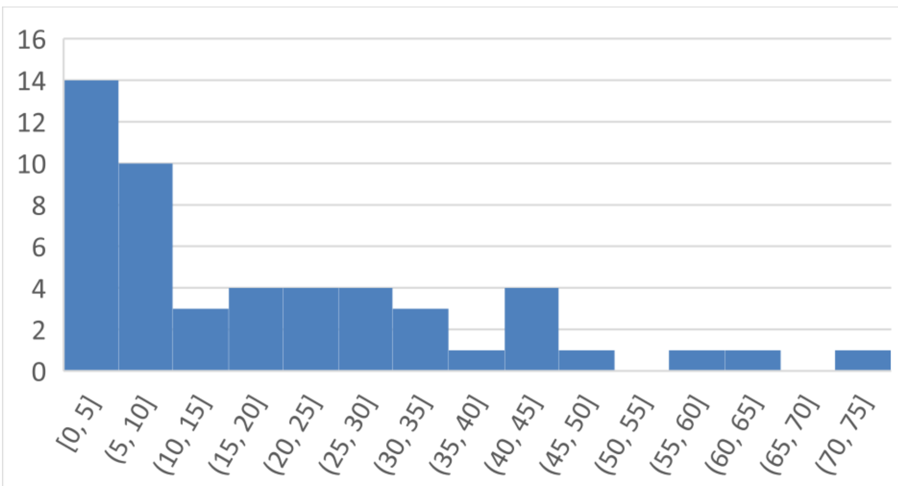


Figure 40. Histogram of H_2 permeance (gpu) of new design modules at 100 psi and room temperature. The x-axis is H_2 permeance (gpu), and the y-axis is the number of modules in each permeance interval.

Additionally, selected new design modules from high-flux and low-flux categories (e.g., Module numbers 84 and 101) were further tested at high temperature to confirm that H_2/CO_2 separation performance of each group was satisfactory for installation in the skid for field testing. The results are shown in **Table 15**.

Table 15. Performance comparison at room temperature and 150ºC.

No. of module and condition	H_2 permeance (gpu)	H_2/CO_2 Selectivity
Module no. 101, room Temperature	0.7	2.9
Module no. 101, 150ºC	17.1	21.1
Module no. 84, room Temperature	12.5	4.3
Module no. 84, 150ºC	126	14.9

We also completed performance testing for refurbished old-design module elements. The N_2 flux of 60 Old-Style modules at room temperature and 10 psi was measured to detect any leaky modules. All modules had reasonable N_2 flux <0.1 ml/min. The modules were packaged in cardboard tubes for shipping to the host-site (see **Figure 41**).

Figure 41. Photographs showing the old style and new style modules cartridges ready for shipping to the host site. Left: Old-Style cartridges; Right: New-Style cartridges.

Test details

During this trip, we collected data using a combination of coal and natural gas derived syngas. We obtained ~50 hours of data with syngas, and the skid operated for over 500 hours continuously without any loss in membrane performance. As in the 2nd trip, the natural gas derived syngas gave a stable gas feed composition to the fiber skid. Coal derived gasifier syngas feed composition fluctuated during the test period. Pressure and temperature profiles during the 500 hr. skid operation are shown in **Figure 42** and **Figure 43**, respectively. Figure **43**. Membrane module temperature profile during the 500-hr skid operation.

Table 16 shows the composition ranges for both natural gas and coal-based syngas streams. **Table 17** provides the cumulative skid operation time from all three trips. The skid has been operated for longer than 800 hours and received 105 hrs. of syngas.

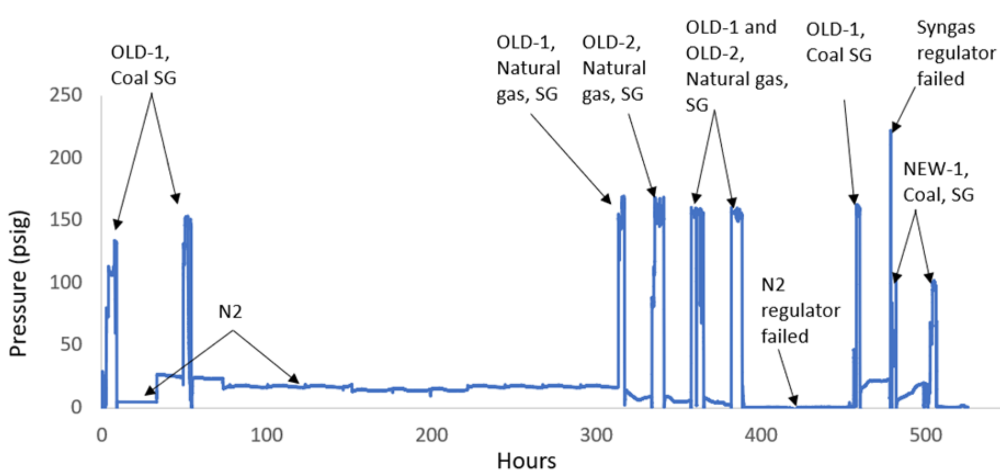


Figure 42. Membrane module pressure profile during the 500-hr skid operation.

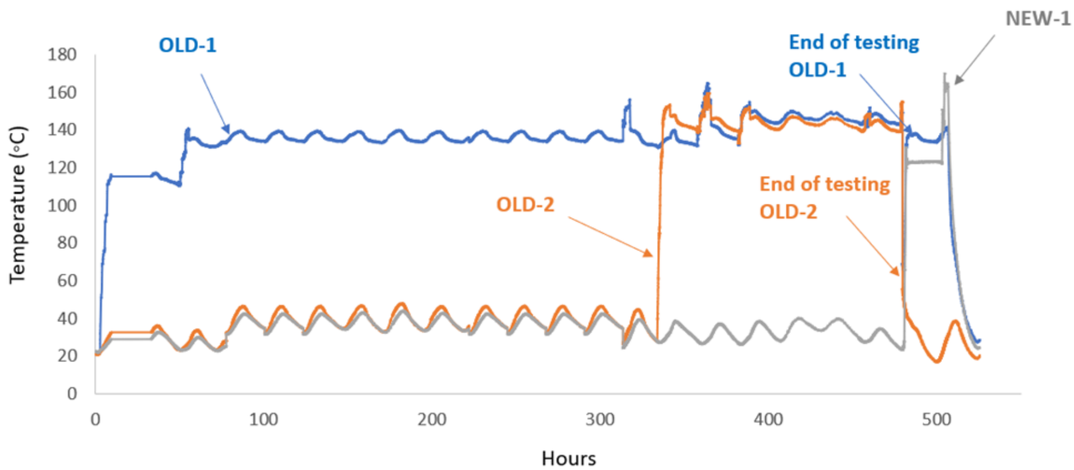


Figure 43. Membrane module temperature profile during the 500-hr skid operation.

Table 16. Syngas composition during 3rd trip.

Syngas type	H_2%	CO_2%	CO%	N_2%	H_2S%
Natural gas	20 - 22	20 - 21	26 - 27	20 - 22	0
Coal	22 - 30	30 - 40	21 - 31	11 - 17	0.3 - 0.4

Table 17. Summary of fiber skid operation time.

Feed to skid	1st trip	2nd trip	3rd trip	1st + 2nd +3rd trips
Time on nitrogen	82	175	447	694
Time on syngas (natural gas)	14	19	22	53
Time on syngas (coal)	2	18	31	51
Total time syngas	16	36	53	105
Total time hot and pressurized	98	211	500	809

Subtask 4.4 - Analysis of the Data from the Skid

Task Description: *The data collected from the parametric and steady-state operation of the modified bench skid with syngas from the oxygen-blown gasifier will be shared with team members for interpretation and analysis. Charts, plots, and tables will be prepared to present and interpret the data.*

Results from the 1st and 2nd Skid Demonstration Trips

During the 1st and 2nd trips to UKy, the skid testing was conducted with temperatures ranging from 90–145°C, pressures between 80–160 psig, and stage cuts between 0.10–0.37. All data were collected with ≈30% hydrogen and 30% CO_2 (see **Table 12** above for gas composition details). **Figure 44** shows the H_2 and CO_2 flow rates in various pressure and temperatures. As expected, the gas fluxes increased with increasing temperature and pressure. However, the increase in H_2 flux occurred much faster than that of CO_2 and this resulted in higher H_2/CO_2 selectivity at higher temperature and pressure.

The H_2 recovery improved with increasing stage cut because the feed retention time was longer in higher stage cuts (**Figure 45**), and CO_2 recovery decreased with increasing stage cut (***Figure 46***). However, the CO_2 recovery increased with temperature due to the increase in hydrogen selectivity at high temperature (**Figure 47**).

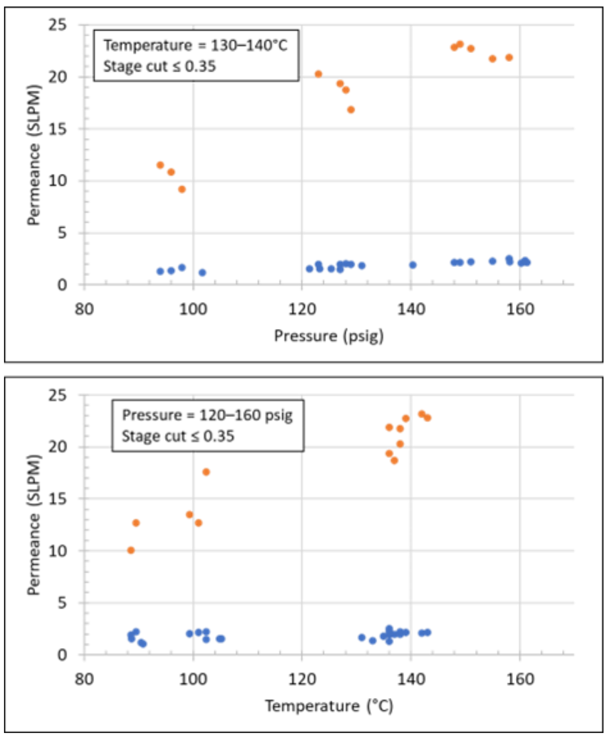


Figure 44. Hydrogen permeate greatly increases with temperature and pressure, CO_2 only slightly increases with increasing temperature and pressure.

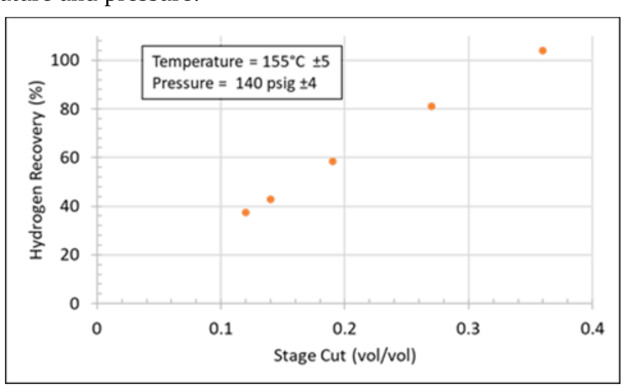


Figure 45. Hydrogen recovery as a function of stage cut.

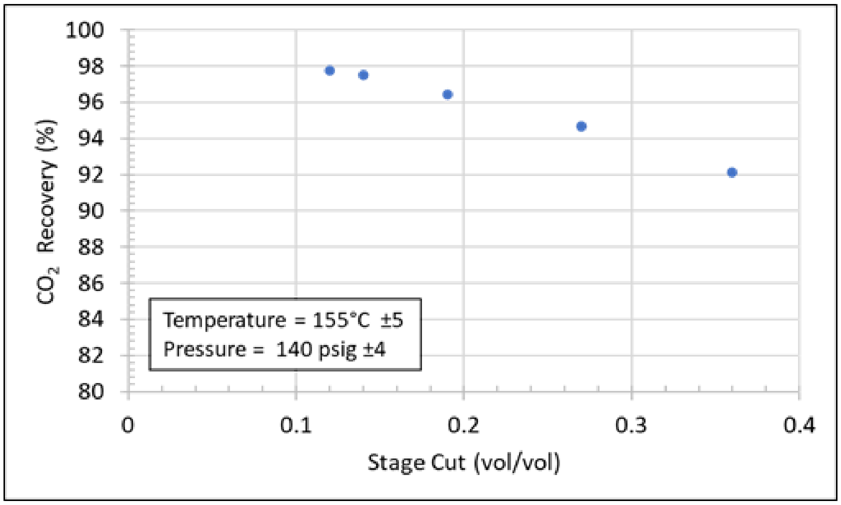


Figure 46. CO_2 recovery as a function of stage cut.

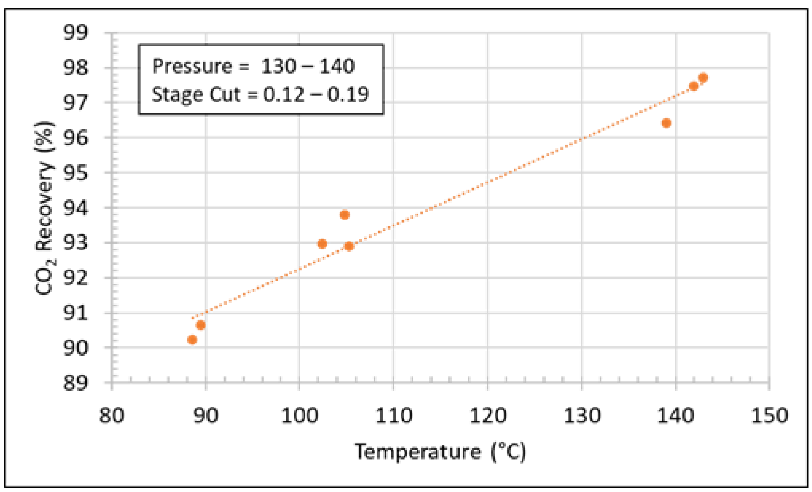


Figure 47. CO_2 recovery as a function of temperature.

Results from 3rd Trip

During the 3rd trip, the skid performance was measured at varying temperature, pressure, and feed flowrates. Temperature, pressure, and feed flow rate variations were 75–150° C, 100–163 psi and 10 - 47 lpm, respectively. The H_2/CO_2 selectivity values were estimated from the direct measurements of H_2 and CO_2 flowrates/composition in the retentate and permeate. **Table 18** shows the data from 6/27/2022. At 145°C, the estimated value for the H_2/CO_2 selectivity is in the range of 24 to 27 at about 145°C. The performance of the skid at 145°C observed with the field syngas from the UKy gasifier agrees well with the initial performance with synthetic gas at SRI.

Table 18. Measured selectivity with coal-derived syngas.

Date	Pressure (psig)	Temp. (°C)	Feed (lit/min)	Permeate (lit/min)	Stage cut (Per./feed)	H_2/CO_2 Selectivity
6/27/2022	162	142	9.48	2.72	0.3	22.7
6/27/2022	153	142	26.26	7.87	0.3	25.6
6/27/2022	161	143	16.97	4.52	0.27	23.5
6/27/2022	158	147	23.66	6.46	0.27	26.8

The average of H_2/CO_2 selectivity of the PBI HFMs in both bench lab tests and field demonstrations is displayed in **Figure 48.** The rate of increase in H_2/CO_2 selectivity in the field test was faster than that in the bench lab test. In the bench lab test that used pure gas for permeation testing, the H_2/CO_2 selectivity was 26 and, based on extrapolation, it was 36 for the field test at 200°C. The higher H_2/CO_2 selectivity in the field test can be attributed to the difference in H_2 and CO_2 permeance behavior of the fibers by increasing the temperature. **Figure** 49 and **Figure 50** show the H_2 permeance and CO_2 permeance of PBI HFMs, respectively, of both bench and field tests. In **Figure 49**, the H_2 permeance increases with temperature for both bench lab tests and field tests. However, CO_2 permeance decreases with temperature in the field test and increases with temperature in the bench lab test which is shown in **Figure 50**. This lead to a faster increase in H_2/CO_2 selectivity with temperature during field tests. CO_2 generally plasticizes polymers, resulting in higher gas permeance and a decrease in gas/CO_2 selectivity. The bench lab test utilized pure CO_2 to test the performance of PBI HFMs, but mixed gas was used for the field test. The use of mixed gas in the field test minimizes the plasticization effect, leading to an increase in H_2/CO_2 selectivity with temperature.

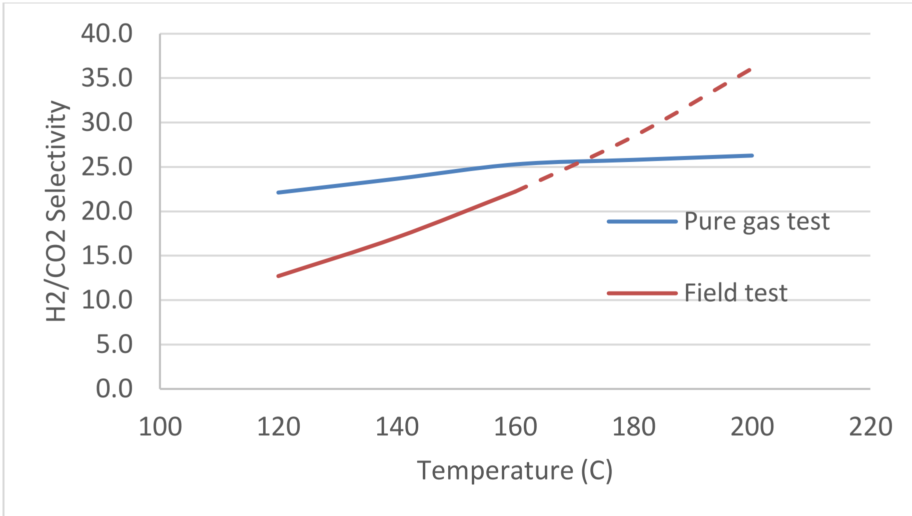


Figure 48. H_2/CO_2 selectivity as a function of temperature for bench lab system and field demonstration.

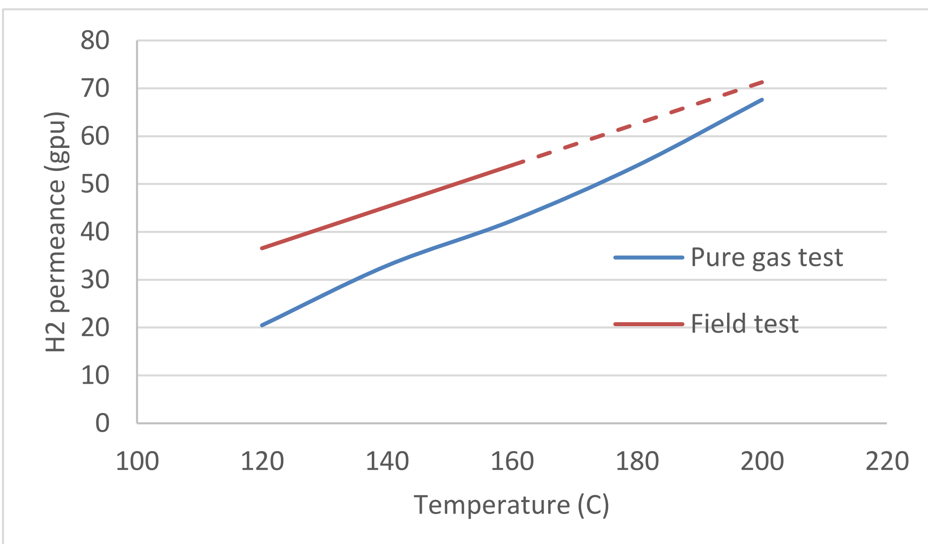


Figure 49. H_2 permeance as a function of temperature for bench lab system and field demonstration.

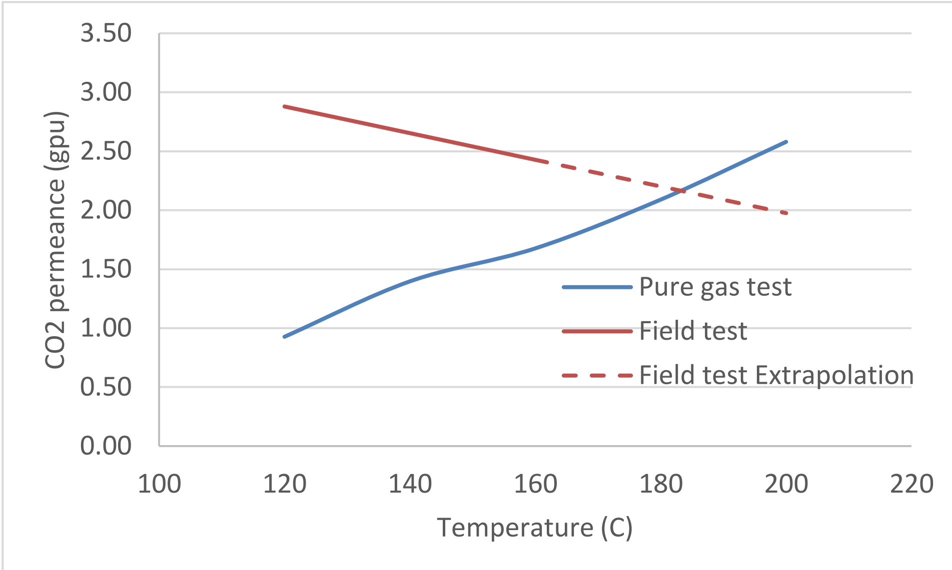


Figure 50. CO_2 permeance as a function of temperature for bench lab system and field demonstration.

TASK 5.0 - TECHNOLOGY ASSESSMENT

Task Description: *The Recipient will conduct a final technology assessment following bench-scale field testing with actual coal-derived syngas from an oxygen-blown gasifier. These results will be incorporated into the following deliverables.*

Summary of Accomplishments

Research activities in Task 5 were conducted under five subtasks that included the Techno-Economic Analysis (TEA), update of the state point data table, Technology Gap Analysis (TGA), Environmental Health and Safety Assessment (EH&S), and Technology Maturation Plan (TMP). These activities were completed, and the details are discussed below under respective subtasks.

Subtask 5.1 - Final Techno-Economic Analysis

Task Description: *The Recipient will update the Preliminary TEA based on results from bench-scale field testing. The Recipient will prepare and submit a Final TEA in accordance with SOPO Appendix A.*

Technology

The TEA was prepared as a standalone document and was submitted to DOE. A brief summary of the TEA is given here. An integrated gasification combined cycle (IGCC) power plant produces high-pressure shifted syngas, which is mostly a mixture of CO_2 and H_2. Capturing CO_2

from shifted syngas at high pressure improves the economics of an IGCC CO_2 capture power plant. Compressing captured CO_2 to a pipeline pressure of 2,210 psia for transport and storage is a significant energy demand that is reduced if the CO_2 is captured at high pressure. Separated H_2 is fed as fuel to the combined cycle power plant.

SRI International's PBI polymeric asymmetric HF membrane permeates H_2 from the shifted syngas feed and captures un-permeated CO_2 as high-pressure retentate. In addition to capturing CO_2 at elevated pressure, the PBI membrane is highly selective to permeate water thus drying the captured CO_2 to a low dew point. Water partial pressure in the permeate increases the PBI membrane's H_2 permeation driving force by lowering H_2 partial pressure in the permeate. **Table** 19 compares the performance and LCOE of an SRI PBI membrane plant with two Baseline plants, one without CO_2 capture and one with CO_2 capture.

Table 19. Technology comparisons.

Case Name	**IGCC-B1A**[1] **Baseline No Capture**	**IGCC-B5B** [1] **Baseline CO_2 Capture**	**SRI PBI Membrane CO_2 Capture**
CO_2 removal	No	Selexol	PBI membrane
CO_2 purification	No		Yes
Sulfur removal	Sulfinol	Selexol	
Performance and Economic Summary			
H_2/CO_2 Selectivity	n/a	n/a	40
H_2 GPU	n/a	n/a	215
CO_2 capture	n/a	95.18%	87.59%
CO_2 purity	n/a	99.08%	96.87%
H_2 recovery	n/a	99.46%	99.04%
HHV plant efficiency	43.00%	33.70%	32.77%
LHV plant efficiency	44.60%	35.00%	33.97%
LCOE w/o T&S ($/MWh)	$105.80	$144.20	$139.02
LCOE w/ T&S ($/MWh)	$105.80	$152.30	$147.12
% Increase in LCOE	0.00%	43.95%	39.05%
Total Plant Cost, 1,000 $	$2,447,360	$2,913,440	$1,750,036

The IGCC B1A Baseline plant without CO_2 capture removes sulfur with a Sulfinol H_2S removal unit. The power plant fuel in that case is unshifted syngas. The IGCC B5B Baseline plant with CO_2 capture uses a two-stage Selexol unit for CO_2 capture and H_2S removal. The SRI PBI membrane plant uses the SRI PBI membrane for CO_2 capture and a single-stage Selexol unit for

[1] Cost and Performance Baseline for Fossil Energy Plants Volume 1: Bituminous Coal and Natural Gas to Electricity, Rv4-1, September 24, 2019

H_2S removal. SRI has measured selectivity between 40 and 50 and uses a conservative performance in this report based on a selectivity of 40. The PBI membrane with 40 selectivity was modeled from bench scale and 4-inch module mass balance data. Future work will be directed to achieving about 46 selectivity in bulk PBI HF production.

SRI had a previous 2009 PBI membrane carbon capture cost estimate called Scenario 3[2]. Its process design was similar to NETL's baseline IGCC carbon capture case. Pertinent operating data of Scenario 3 PBI membrane with permeate N_2 sweep is presented in **Table 20**.

Table 20. Scenario 3-calculated PBI membrane recoveries.

	psia	scfm		mol frac	scfm	% rec.
Feed	777	565,764	CO_2	0.3113	176,122	n/a
Retentate	757	203,043	CO_2	0.7805	158,475	89.98%
Feed	777	565,764	H_2	0.4306	243,618	n/a
Permeate	250	630,320	H_2	0.3534	222,755	91.44%

From **Table 20**, the membrane feed to permeate pressure ratio in Scenario 3 was 3.11. From here onward, Scenario 3 will be called the 2009$ plant. The present SRI PBI membrane plant operates with a permeate pressure of 30 psia and therefore will have a higher membrane pressure ratio than the 2009$ plant's membrane pressure ratio. As a rule, it is desirable to operate a gas separation membrane at a higher-pressure ratio and it is a fundamental parameter to achieve a more effective operating separation.

Accordingly, the present SRI PBI membrane plant with the higher-pressure ratio and 40 selectivity recovers 99.04% of the membrane's feed H_2 in the permeate and captures 87.59% CO_2 in the retentate. In the 2009$ plant case, H_2 recovery was 91.44% with 89.98% CO_2 capture. Operating the 2009$ plant with 42.94 selectivity and 99.04% H_2 recovery with a pressure ratio of 3.11 gives a CO_2 capture rate of 83.33%. Pertinent operating data of the present SRI membrane without permeate N_2 sweep is presented in **Table 21** below.

2 G. Krishnan, I. Jayaweera, A. Sanjurjo, Fabrication and Scale-up of Polybenzimidazole (PBI) Membrane based System for Pre-combustion-Based Capture of Carbon Dioxide, SRI International, June 2012. Cooperative Agreement No. DE_FC26-07NT43090.

Table 21. SRI PBI membrane recoveries calculated from Appendix B, Table B-1

	psia	lb-mol/h		mol frac	lb-mol/h	% rec.
Feed	330	88,168	CO_2	0.3096	27,294	n/a
Retentate	328	26,400	CO_2	0.9055	23,906	87.59%
Feed	777	88,168	H_2	0.4375	38,578	n/a
Permeate	250	61,768	H_2	0.6186	38,207	99.04%

The present SRI PBI membrane plant's increase in LCOE with transport and storage is 11.14% less than the B5B increase. The total plant cost (TPC) of the SRI PBI membrane CO_2 capture power plant is 39.9% less than the TPC of the Baseline B5B CO2 capture plant. **Figure 51** presents the SRI PBI membrane capture plant by showing where the B5B Baseline CO_2 capture plant's diagram is modified to create the SRI PBI membrane plant. The less complex PBI membrane configuration of membrane modules, piping, and valves replaces the more complicated second-stage CO_2 Selexol absorber unit with its associated equipment, including refrigeration, power recovery turbines, compressors, heat exchangers, flash tanks, solvent dehydrator, and solvent pump. The single-stage H_2S Selexol unit is retained in the present SRI PBI membrane plant.

The SRI PBI membrane plant uses a catalytic oxidizer system to increase captured CO_2 purity to >95%. This system can achieve >95% CO_2 purity using the near stoichiometric amount of oxygen needed for combustion with small quantities of oxygen and carbon monoxide remaining after oxidation. The catalytic oxidizer is shown to achieve these limits for carbon monoxide at 34 ppmv and oxygen at 10 ppmv, well within the limits recommended range of 10 - 5,000 ppmv for CO, and 0.001 – 4 vol.% for O_2. A 205-kWe oxygen compressor with a motor efficiency of 95% and a compressor efficiency of 85% delivers compressed oxygen to the catalytic oxidizer.

The NETL impurity limit for water is 500 ppmv for the conceptual design and a limit range in the literature is at 20-650 ppmv. Water is the most permeable component in the SRI PBI membrane and is generally known to be more permeable than hydrogen. Water permeability in the PBI membrane has not been measured, so the model assumes it to be at least as high as hydrogen and it is usually set equal to the hydrogen permeability. The reason for this is that when H_2/CO_2 selectivity is relatively high, the simulation algorithm is overly sensitive to relatively small increases in water permeability relative to hydrogen permeability. The higher the H_2/CO_2 selectivity, the higher the sensitivity. Small increases in water permeability to relative hydrogen permeability cause the simulation algorithm to calculate negative water fractions in the retentate. Therefore, water permeability is generally made equal to hydrogen permeability to avoid negative retentate values.

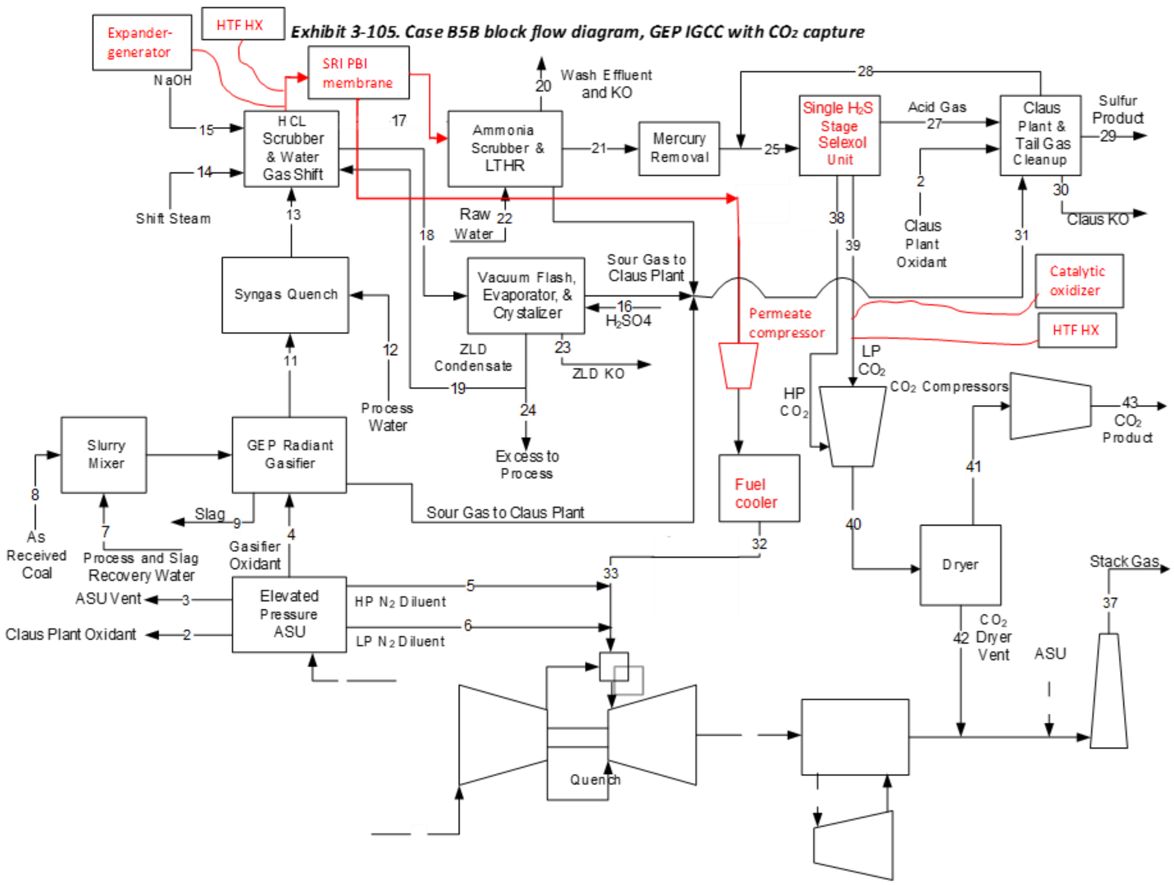


Figure 51. B5B Baseline capture plant modified as a PBI capture plant.

The PBI membrane, like gas separation membranes in general, is an excellent moisture removal or drying device. The water permeability in the GEN-1 membrane model in the preliminary SRI PBI TEA was set equal to the hydrogen permeability. In this report water permeability was increased to 1.015 times the hydrogen permeability. The simulation algorithm model calculated a water mole fraction of 0.0054 in the retentate stream. Consequently, the water mole fraction in retentate is now reduced to 198 ppmv. Therefore, the PBI membrane itself enables water impurity concentrations in the final CO_2 product to be less than the NETL upper limit of 500 ppmv.

For the TEA, SRI's updated PBI membrane feed gas pressure is 330 psia and the permeate pressure is 30 psia. Both are revised downward from 768.7 psia feed gas pressure and 60 psia permeate pressure in the preliminary TEA. Accordingly, the trans-membrane pressure difference is 300 psi revised downward from 708.7 psi, and the pressure ratio is 11.00 revised downward from 12.81. The reduction in the SRI PBI membrane feed pressure necessitated the inclusion of a pressure reducing expander-generator to lower the pressure from 768.7 psia and a heat transfer fluid heat exchanger (HTF HX) for reheating the feed gas after the expander-generator. The expander-generator is located after the low-temperature shift reactor and before the SRI PBI membrane. The heat transfer fluid is reheated in a catalytic oxidizer second HTF HX aftercooler.

A compressor increases the permeate pressure from 30 psia to 250 psia to meet the fuel pressure requirement at the gas turbine. The permeate compressor replaces an expander in the B5B Baseline case, which is needed to lower the fuel gas pressure from the Selexol CO_2 capture unit to meet the required gas turbine fuel pressure. An advantage of the PBI membrane plant is that the CO_2 retentate capture stream pressure at 320 psia is about 4.4 times higher than the B5B Baseline captured CO_2 stream pressure of about 75 psia coming off the Selexol unit's flash tanks. Accordingly, the auxiliary power and capital cost of the CO_2 product compressor for sequestration or utilization in the SRI PBI membrane plant will be less than the B5B baseline plant.

Subtask 5.2 - Update State-point Data Table

Task Description: *The Recipient will update the State-Point Data Table based on the test results from actual testing and the updated heat and mass balance data from modeling. The Recipient will prepare and submit an updated State-Point Data Table in accordance with SOPO Appendix C.*

The State-Point Data Table was updated and submitted to DOE as a standalone document. The updated State-Point Data Table is given below.

State-Point Data Table

Table 22. State-point data table for the PBI membrane system.

	Units	Measured/ Estimated Performance	Projected Performance
Materials Properties			
Materials of fabrication for selective layer		PBI	
Materials of fabrication for support layer		PBI	
Nominal thickness of selective layer	µm	0.3 to 1	0.3 to 0.5*
Membrane geometry		Asymmetric	Asymmetric
Max trans-membrane pressure	bar	15-25	20
Hours tested without significant degradation		1000	20,000-40,000#
Membrane Performance			

	Units	Measured/ Estimated Performance	Projected Performance
Temperature	°C	50 -225	225
Pressure normalized flux for permeate - H_2	GPU or equivalent	<50 - 125	125 (0.5μm)/215 (0.37 μm)
CO_2/H_2O selectivity	-	<0.05	0.02
CO_2/N_2 selectivity	-	Not applicable	Not applicable
CO_2/SO_2 selectivity	-	Not applicable	Not applicable
CO_2/H_2 selectivity	-	See below	See below
H_2/CO_2 selectivity	-	25-50	40 - 50
H_2/H_2O selectivity	-	>0.9	>0.9
H_2/H_2S selectivity	-	>200	>200
Type of measurement (ideal or mixed gas)	-	Pure and mixed gas	Mixed gas
Proposed Module Design			
Flow arrangement	-	Hollow fiber	
Packing density	m^2/m^3	~3000 (current), >4000 (final commercial product)	
Shell side feed	-	Retentate: CO_2 stream	

***Definitions for Table 1*:**

- *Membrane geometry* – hollow fiber.
- *Pressure normalized flux* – for materials that display a linear dependence of flux on partial pressure differential, this is equivalent to the membrane's permeance.
- *GPU* – Gas Permeation Unit, which is equivalent to 10^{-6} $cm^3/(cm^2 \cdot s \cdot cmHg)$
- *Type of measurement* – projected membrane performance is for mixed gas.
- *Flow arrangement* – hollow-fiber bundles with cross-flow arrangement
- *Packing density* – 40% (Ratio of the active surface area of the membrane to the volume of the module).
- *Shell side fluid* – retentate stream.

Subtask 5.3 - Technology Gap Analysis

Task Description: *The Recipient will prepare and submit a Technology Gap Analysis (TGA) in accordance with SOPO Appendix D. The TGA will include technical risks and risk mitigation strategies for advancement of the technology to the next scale of development.*

We have prepared a Technology Gap Analysis report and submitted it to DOE as a standalone document. A summary is given below.

Currently, PBI HFM technology for syngas separation is at TRL 5 after successful demonstration at CAER, University of Kentucky. The next step is an engineering-scale demonstration at 1 to 10 MW scale. Since the membrane systems can be easily designed as modular units, process scale up and commercialization is relatively straightforward. We do not anticipate any major engineering challenges for large-scale demonstrations for equipment and instrumentation. The challenges exist in large-scale high-quality fiber production, potting, and module manufacturing.

Large-scale fiber production: Production of large-scale high-quality HFMs requires a dedicated facility. The PBI HFM that we used for gas separation has a gradient microstructure with a thin dense layer outside. One of the challenges in the laboratory-scale production was obtaining a defect-free thin dense layer. A thin dense layer enables high gas permeance, but it is susceptible to defects. The thin layer is easily damaged by dust particles in the production lines, rough surfaces on spin wheels, and excessive manual handling during post treatment and potting. These issues can be addressed in a large-scale dedicated facility with high degree of automation. Manufacturing HFMs at large scale is a well-established process, and manufacturing facilities can be configured for PBI HFM production.

Module potting: Potting fibers to withstand temperature and pressure fluctuations requires a glue that has thermal properties similar to PBI and high temperature stability. We used commercially available high-temperature epoxy from Cotronics®. However, this epoxy showed shrinkage with time and tend to delaminate when subjected to temperature cycles. We identified the issue to be related to a slight thermal expansion mismatch of epoxy, PBI, and the stainless-steel sleeve of the fiber module. In addition, this epoxy did not flow freely to wet all fibers (too viscous) and hence it had to be manually forced to get complete surface coverage in the potting region of the fibers. This epoxy precludes the use of the standard industry fiber potting approach of dipping the module in a glue bath and trimming the end to expose fiber holes. Thermal and wetting properties of the epoxy must be resolved, and a better epoxy is needed for large-scale module production for an engineering-scale demonstration. Testing of variety of other high-temperature epoxies was not feasible during the current project. This must be investigated through a separate R&D program.

Fiber module design: PBI HFMs exhibit expansion and contraction depending on the temperature and the moisture content. This expansion/contraction is negligible in the diameter-wise direction; however, it is quite significant in the lengthwise direction. In the SRI fiber skid, we tested only a one-end-open design, and hence the fiber bundle was free to expand and contract inside the metal sleeve because it was held only at one end. It is preferable to have a both-end-open design as it allows the use of sweep gas, which improves the gas permeance. For a both-end-open design that allows sweep gas flow, the fiber bundle needs to be potted at both ends to the metal sleeve. Thus, the module element and metal sleeve must be designed to accommodate thermal expansion and ease of replacement. These are engineering design challenges that need to be addressed for large-scale demonstrations.

Fiber packing density: Increasing the fiber packing density in the module element will reduce the gas bypass. This will also reduce the overall size of the module assembly for a selected capacity and thus the skid footprint. Densely packed fiber module elements can be manufactured at a dedicated and automated production facility.

Subtask 5.4 - Environmental Health and Safety Assessment (EH&S)

Task Description: *The Recipient will prepare and submit an EH&S process risk assessment in accordance with SOPO Appendix E. The assessment will provide a full overview of the risks and hazards associated with the PBI-HFM CO_2 capture process and detail the risks and mitigations for the technology based on data and information collected throughout the project.*

We performed an Environmental, Health, and Safety (EH&S) risk assessment for the project DE-FE0031633, which involved the design, engineering, and testing of a 50-kW$_{th}$ pre-combustion CO_2 capture test skid using SRI's high-temperature PBI HFMs. We submitted this report as a standalone document to DOE and a brief summary is given below.

The SRI PBI membrane test skid was tested during September 1, 2021, to July 31, 2022, at University of Kentucky, Center for Applied Energy Research (UKy-CAER) in Lexington, KY. The project led by SRI, in collaboration with ENERFEX, Inc. (membrane modeling company); PBI Performance Products, Inc. (polymer blend supplier), and Kevin O'Brien (consultant) to evaluate a pre-combustion CO_2 capture system based on a high-temperature PBI polymer membrane separation system and to optimize the process for incorporation into an oxygen-blown IGCC plant.

As a part of the project, an EH&S risk assessment report on the operation of the SRI PBI membrane skid was prepared. Assessing the feasibility of the project, skid design, and skid operation in terms of the EH&S is required so that measures may be taken to mitigate any recognized risks. Boolean risk assessment methodology is introduced and applied to the data presented in this report. Syngas flammability and toxicity are the two risks associated with an accidental gas leakage scenario that necessitate a hazard risk identification. A literature survey indicates that syngas toxicity is a minor risk compared with its flammability. As the top possible eventuality, syngas flammability is discussed in detail and the Boolean risk analysis method is used to evaluate the frequency and probable outcomes of accidental syngas gas ignition in the SRI PBI membrane gas separation test skid. The analysis shows the probability of all the accidental syngas leak and ignition events is significantly low. Accordingly, the SRI PBI membrane gas separation module test skid (as designed) can be operated without additional safety measures.

Subtask 5.5 - Technology Maturation Plan (TMP)

Task Description: *The Recipient will update the TMP following bench-scale field testing. The Recipient will prepare and submit a Technology Maturation Plan (TMP) in accordance with SOPO Appendix B.*

Technology Maturation

SRI prepared a TMP and submitted it to DOE. As mentioned earlier, the separation process is based on HFMs made of high-temperature stable PBI polymer developed at SRI. The PBI polymer blend is available commercially (PBI Performance Products), and SRI developed the asymmetric HFMs using a PBI polymer blend for gas separation applications. **Figure 522** shows photographs of SRI-made PBI-HFMs and membrane modules prepared to install in the skid. In the PBI-based membrane separation, non-fuel gases will be retained at the high temperature (~225°C) and high pressure (>20 atm) of the feed syngas, while the fuel gas H_2 and steam permeates through compact, hollow, asymmetric PBI fibers for use in gas and steam turbines. The membrane-based system is coupled to an IGCC plant downstream of the water-gas shift (WGS) reactors. The feed syngas stream to the membrane unit is maintained above its dew point to avoid condensation of steam.

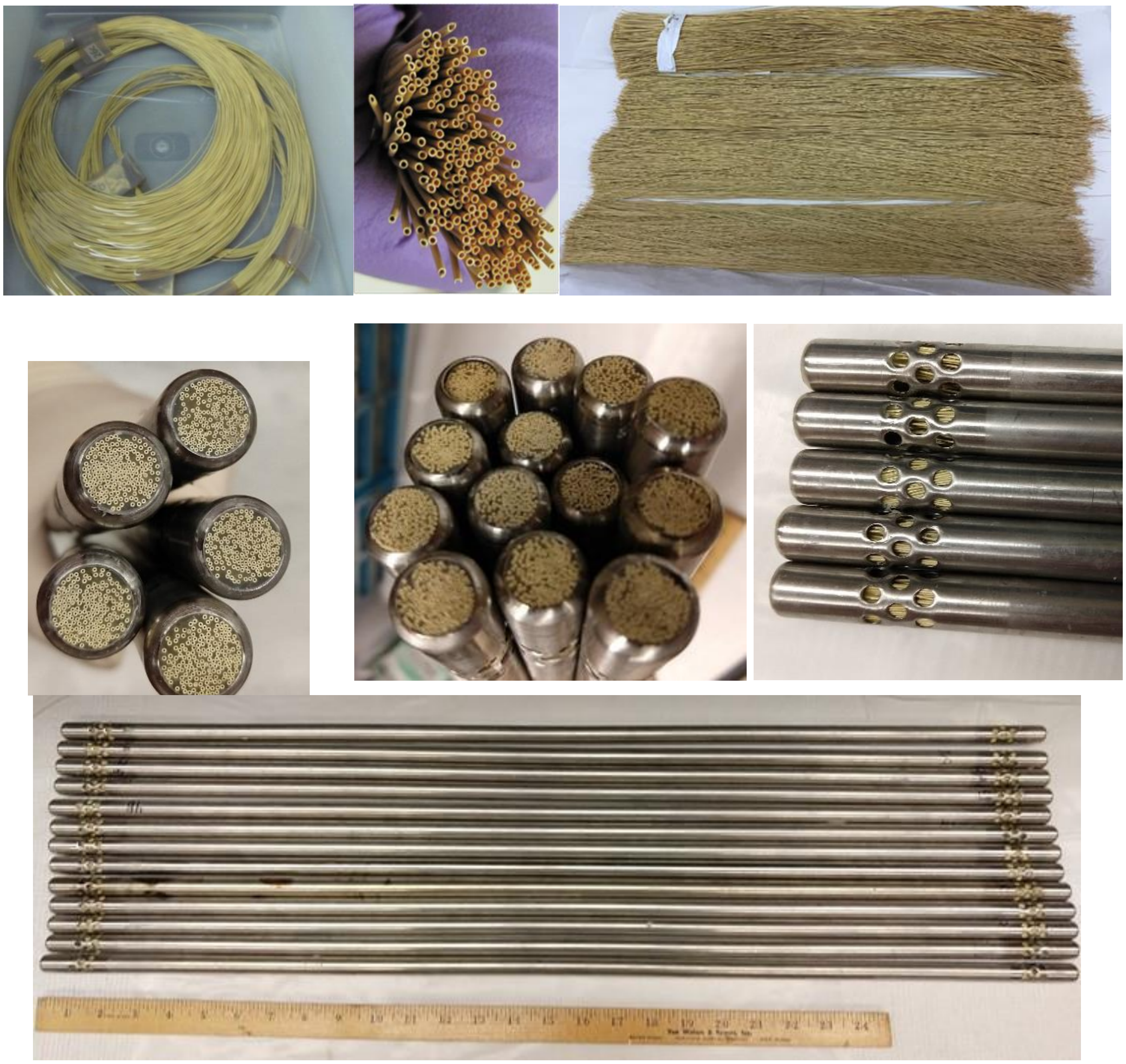


Figure 52. Photographs of PBI-based, asymmetric HFMs (top) and fiber module elements (bottom) made at SRI.

SRI tested the performance of PBI-HFM modules with syngas for CO_2 and H_2 separation at the National Carbon Capture Center (NCCC) under air-blown gasifier conditions (DE-FE0012965). SRI used fiber modules with 1st-generation (GEN-1) microstructure fibers that have H_2/CO_2 selectivity about 25. SRI's GEN-2 PBI-HFMs have a H_2/CO_2 selectivity of approximately 40 in lab tests with simulated syngas. Based on previous analyses, the enhanced H_2/CO_2 selectivity of the PBI fibers will significantly improve the process economics as explained above in the TEA. The second field testing was planned with an oxygen-blown gasifier at CAER, University of Kentucky.

Technology Readiness Level

DOE Technology Maturation Process

The technology maturation process involves the DOE Program Manager's and the subject matter experts' careful analysis of the technology being developed, identified risks and mitigation methods, and the path to commercial validation. The team used the TRL table that DOE-NETL has established shown in Appendix A of DE-FOA-0001830 to specify the current TRL of the proposed technology. Our analysis indicates that PBI-HFM gas separation technology is currently at TRL-5 as shown in **Table 23**.

Table 23. Technology readiness level.

TRL	Definition	Description
5	Basic technology components integrated and validated in a relevant environment	Technology Validated in a Relevant Environment. Basic technology component configurations have been validated in a relevant environment. Component integration is similar to the final application in many respects. Data sufficient to support planning and design of the next TRL test phase have been obtained. Performance attributes and requirements have been updated.

SRI successfully demonstrated the performance of PBI-HFM modules with syngas for CO_2 and H_2 separation at the NCCC in 2017 using GEN-1 based modules (H_2/CO_2 selectivity approximately 25) and syngas derived from an air-blown gasifier. In the current project, GEN-2 based modules (H_2/CO_2 selectivity 25-40) was tested with a syngas from an oxygen-blown gasifier at CAER, University of Kentucky in 2021 and 2022. The current field demonstration of the 50-MWth fiber test skid matured the technology because it evaluated: (1) GEN-2 fibers with an enhanced selectivity leading to expected improved process economics; and (2) performance testing in a more commonly used oxygen-blown gasifier environment.

Target Commercial Applications

The PBI-HFM is targeted for pre-combustion CO_2 capture applications. The polymer's inherent properties enable it to withstand the high temperatures and pressures typically seen downstream from the WGS reactor used in pre-combustion capture. The high selectivity (40) of GEN-2 provides a pathway to achieving DOE's pre-combustion capture goals (at least 95% CO_2 purity with no more than a 30% increase in COE).

Post Project Plans

Team members had preliminary discussions as to the next steps after successful completion of the current project. It was expected that nearly all of the team members would be involved in future technology maturation efforts, hence enabling continuity in know-how gained during the technology commercialization process.

The most critical phase in scaling-up to a small pilot and beyond is the production of consistent, high-quality, and reproducible HFMs based on PBI. PBI is typically supplied as a spinning “dope”, i.e., polymer in solvent at a given concentration. The handling of the dope requires special expertise. The spinning equipment used in the production of PBI-HFMs is typical of equipment used in producing HFMs produced using other polymers. The challenge and the business advantage is that there is a great deal of know-how involved in spinning, handling, and potting PBI-HFMs. This know-how and intellectual property (IP) have been developed by SRI, and SRI will license this capability to a company that can produce high volumes of commercial PBI HFMs.

An analysis was performed to determine when fiber spinning would need to be transferred from SRI to a commercial organization. This relationship is outlined in **Figure 53**. The current test size (0.016 MWe) can be produced using existing SRI facilities. The following assumptions were used for the SRI production analysis:

- Fiber production capacity analysis based on two (2) existing fiber production lines
- Run rates of 3 m/min
- Yield assumed to be 75%
- Maintain one shift.

Based on these assumptions, sufficient fiber could be produced for the 0.016 MWe (50 kWth) test by running 5 hr /day with 3 day/month production. The other points have been calculated using this same analysis. It is important to consider that SRI is NOT designed to be a facility to produce high volumes of materials in a commercial fashion. Based on the analysis shown in **Figure 53**, it is not likely that SRI will be able to meet fiber production goals in a timely fashion for tests in excess of 0.1 MWe. This indicates it will be critical to transfer the fiber spinning technology to another commercial organization to produce sufficient fiber for tests of >0.1 MWe. This volume of fiber production will also require significant capital investment in commercial fiber spinning equipment. This transition point is outlined in **Figure 54**. It is also important to note that as the test size exceeds 0.1 MWe, more engineering partners will need to be engaged to provide inside battery limit (ISBL) and outside battery limit (OSBL) capabilities. This is discussed further below. The considerations listed above will be factored in when selecting the next pilot size and host site.

Preliminary discussions have been focused on PBI Performance Products taking over the scale-up of the fiber production process. PBI Performance Products not only produces the polymer but has production facilities for solid PBI fibers for a variety of applications. For example, PBI fibers have been used in commercially produced fire-fighting protective apparel since the 1990s (https://pbipolymer.com/about/about-celazole-pbi/). PBI Performance's facilities in South Carolina could be adapted for production of GEN-2 HFMs. A major advantage of this route would be that the production of the polymer and the spinning of the HFMs could be conducted at one facility under the control of one entity.

Potting and construction of modules based on the GEN-2 HFM could be done at PBI Performance or by a separate organization. The future business decision that PBI Performance will determine is whether they desire to supply bundles of HFMs or whether they want to supply membrane modules. Regardless as to whether they supply fibers or modules, PBI Performance's production facility is sufficiently close to a large transportation hub (within approximately 1 hour of Charleston, SC) to provide global demands.

The final modules, whether supplied by PBI Performance or another organization, could then be assembled by any number of standard engineering, procurement, and construction (EPC) firms into a final membrane system with the necessary control systems and ancillary equipment to meet the needs of an individual power plant. This membrane system would be defined as the ISBL component for the capture system at the power plant. As this project develops further, the team expects to develop relationships with large EPC firms skilled in the managing ISBLs for capture systems. The selection of the EPC firm would be partially driven by the small pilot-scale (i.e., whether 0.1 or 1.0 MWe). The EPC selection will become critical as the test size exceeds 0.1 MWe. The team plans to devote efforts to seek funding opportunities, develop commercial interest, and demonstration sites.

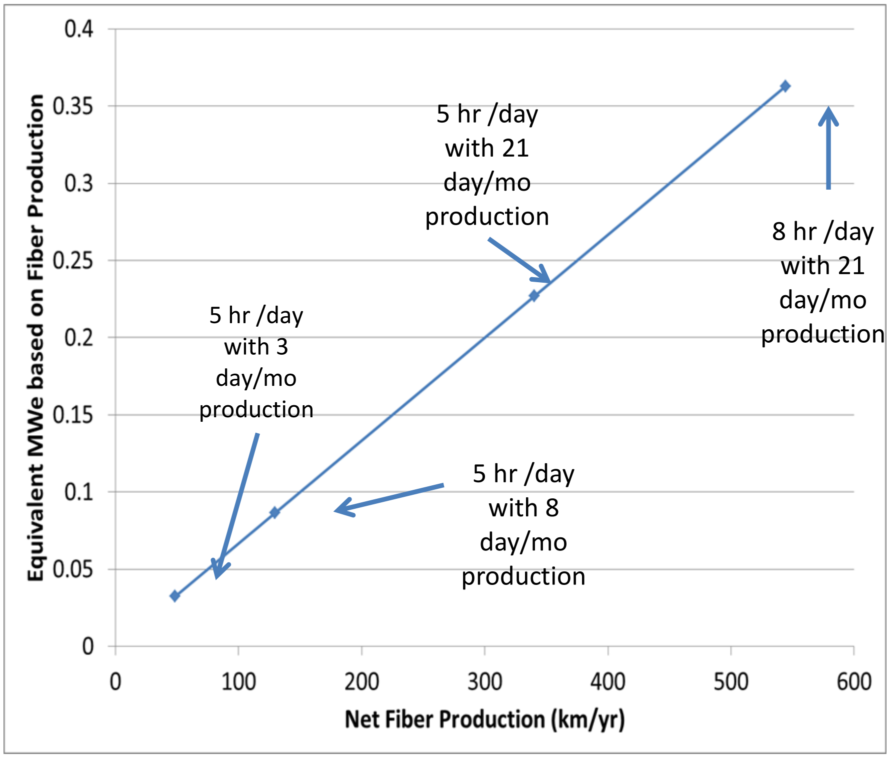


Figure 53. Relationship between fiber production and test size in Mwe.

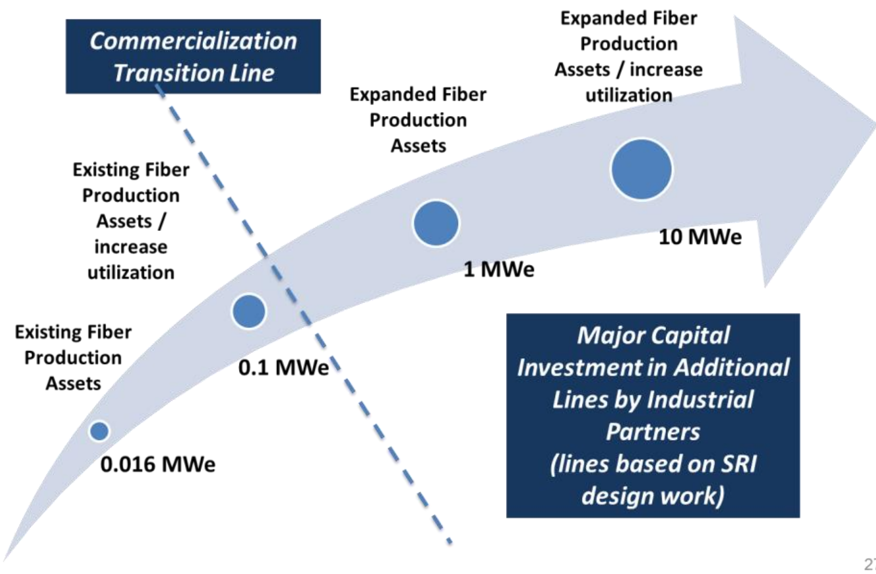


Figure 54. Analysis of when to transition fiber spinning.

TASK 6.0 - BENCH SKID DECOMMISSIONING, TRANSPORT AND STORAGE

Subtask 6.1 - Bench Skid Decommissioning and Transport

Task Description: *The Recipient will dismantle and remove the modified bench skid from the field test host site and transport to the Recipient's facilities. This task involves disconnecting the tie-ins to the syngas feed and other utility connections at the field test host site, as well as bench skid transport to the Recipient's facilities.*

We transported the PBI skid to Menlo Park in Dec 2022 for storage. **Figure 555** shows the arrival of the skid at Menlo Park.

Figure 55. Arrival of PBI skid at Menlo Park after field testing at UKy-CAER. (The skid is housed in the container on the truck.)

Subtask 6.2 - Bench Skid Post-Testing Analysis and Storage

Task Description: *The Recipient will prepare the modified bench skid for storage. This work involves the inspection the skid components, such as valves and pressure gauges and preparing them for storage. Special care will be taken to observe any deposits from the syngas. PBI membrane fiber elements will be removed from the reactors and inspected to identify any foreign deposits on the fiber surface as a result of their exposure to coal-derived syngas.*

We have conducted the post-testing of the testing modules on on-going basis after each run campaign as reported in detail under Subtask 4.3. The skid is securely stored at SRI facility for use in any future demonstration projects.

PRODUCTS

TECHNOLOGIES OR TECHNIQUES

SRI is pursuing the development of a novel and cost-effective technology that uses a high-temperature polymer membrane to separate CO_2 and other gases in a shifted syngas stream from a coal gasifier. The polymer is produced as asymmetric hollow fibers that are bundled into modules suitable for high-temperature, high-pressure operation. This technology was tested at SRI and at the NCCC in Wilsonville, AL. We are pursuing further study to advance the technology and provide scale-up demonstration opportunities in the future.

INVENTIONS, PATENT APPLICATIONS, AND/OR LICENSES

SRI's patent approval in Japan was granted during the period of performance of this project. US Patent 9,321,015 was issued on 26 April 2016 as follows: Title: Process for Fabricating Hollow Fiber Asymmetric Membranes for Gas Separation and Liquid Separation.

PARTICIPANTS & OTHER COLLABORATING ORGANIZATIONS

The following organizations are participating in the program:

- SRI International, Menlo Park, CA
- Enerfex, Inc., Isle La Motte, VT
- PBI Performance Products, Inc., Charlotte, NC
- Dr. Kevin O'Brien, Consultant, Energy Commercialization, IL
- The University of Kentucky Center for Applied Energy Research (CAER), Lexington, KY