SLATT UNDERGRADUATE RESEARCH FELLOWSHIP
FINAL REPORT

SCHOLAR NAME:	Austin Booth
FACULTY ADVISOR:	Dr. Casey O'Brien
PROJECT PERIOD:	August 2020 - January 2021
PROJECT TITLE:	Development of an Operando Spectroscopic Tool for Studying the Structure and Dynamics of Membranes in Complex Environments
CONNECTION TO ONE OR MORE ENERGY-RELATED RESEARCH AREAS (CHECK ALL THAT APPLY):	**(X) Energy Conversion and Efficiency** **( ) Sustainable and Secure Nuclear** **( ) Smart Storage and Distribution** **( ) Transformation Solar** **( ) Sustainable Bio/Fossil Fuels** **( ) Transformative Wind**

MAJOR GOALS AND ACCOMPLISHMENTS

Summarize your research goals and provide a brief statement of your accomplishments (no more than 1-2 sentences). Indicate whether you were able to accomplish your goals by estimating the percentage completed for each one. Use the next page for your written report.

RESEARCH GOALS	ACTUAL PERFORMANCE AND ACCOMPLISHMENTS	% OF GOAL COMPLETED
Construct a spectroscopic permeation cell for the observation of membranes	The spectroscopic permeation cell was constructed this summer by a graduate student in the O'Brien lab. I was not able to come in over the summer due to the pandemic, so this part of the project was completed before I arrived.	100%
Construct a temperature control system for the permeation cell	Independently researched, ordered, and assembled temperature control equipment for the permeation cell. Designed and constructed stainless steel panels to house the temperature control equipment.	95%
Prepare composite polyvinylamine (PVAm)-based membranes, typically used for CO_2 transport	During the semester, PVAm membranes for this project were prepared by our lab's graduate student. I am now working to synthesize a new type of PVAm membranes that incorporate copper silicate nanotubes to enhance gas permeation rates and CO_2 selectivity.	60%
Analyze PVAm membranes within the permeation cell and detect CO_2 transport intermediates	This part of the project has been moved to our graduate student's research area. PVAm membranes prepared by the lab's graduate student were analyzed during the semester; the new PVAm membranes I am synthesizing have not yet been analyzed.	20%

RESEARCH OUTPUT

Please provide any output that may have resulted from your research project. You may leave any and all categories blank or check with your faculty advisor if you are unsure how to respond.

CATEGORY	INFORMATION
EXTERNAL PROPOSALS SUBMITTED	(Sponsor, Project Title, PIs, Submission Date, Proposal Amount)
EXTERNAL AWARDS RECEIVED	(Sponsor, Project Title, PIs, Award Date, Award Amount)
JOURNAL ARTICLES IN PROCESS OR PUBLISHED	(Journal Name, Title, Authors, Submission Date, Publication Date, Volume #, Page #s)
BOOKS AND CHAPTERS RELATED TO YOUR RESEARCH	(Book Title, Chapter Title, Authors, Submission Date, Publication Date, Volume #, Page #s)
PUBLIC PRESENTATIONS YOU MADE ABOUT YOUR RESEARCH	(Event, Presentation Title, Presentation Date, Location)
AWARDS OR RECOGNITIONS YOU RECEIVED FOR YOUR RESEARCH PROJECT	(Purpose, Title, Date Received)
INTERNAL COLLABORATIONS FOSTERED	(Name, Organization, Purpose of Affiliation, and Frequency of Interactions)

EXTERNAL COLLABORATIONS FOSTERED	(Name, Organization, Purpose of Affiliation, and Frequency of Interactions)
WEBSITE(S) FEATURING RESEARCH PROJECT	(URL)
OTHER PRODUCTS AND SERVICES (e.g., media reports, databases, software, models, curricula, instruments, education programs, outreach for ND Energy and other groups)	(Please describe each item in detail)

RESEARCH EXPERIENCE

Please let us know what you thought of your research experience: Did this experience meet your expectations? Were lab personnel helpful and responsive to your needs? What else could have been done to improve your experience or achieve additional results?

I greatly enjoyed this research experience; it fully met my expectations. I had worked with the O'Brien lab before, and as a result I already knew most of the lab personnel. Everyone in the lab was always helpful, friendly, and responsive to my needs. Unfortunately, the University's research labs were closed for the summer, but I am happy that I was at least able to continue this program during the fall and winter semesters. Due to the schedule shift, I was only able to work on the project part-time during the fall semester, and a good amount of the work I was originally supposed to be involved in was completed ahead of time by graduate students. However, I was still able to contribute meaningfully to a large chunk of the project, specifically the temperature control portion. Conducting full-time research during the winter session is helping me achieve additional results as well. Overall, my research experience was very engaging and relevant to my studies. The project's implications for carbon capture and energy generation made it applicable to my interests and Energy Studies minor, and it will be valuable experience for my future career.

FINAL WRITTEN REPORT

(Please use the space below to describe your research project and objectives, any findings and results you can share, and graphs, charts, and other visuals to help us understand what you achieved as a result of this research experience.)

The objective of this research project is to develop an operando spectroscopic tool for the analysis of membranes in complex environments. Society depends on chemical separations for many necessities including clean water, pure chemicals, fuels, and food. Currently, most industrial chemical separations are performed using thermal techniques, such as distillation. However, these techniques use enormous amounts of energy, accounting for approximately 10-15% of total global energy consumption. Advanced membrane technologies that can efficiently separate chemicals could drastically reduce the energy consumption of chemical separations and have substantial global impacts. However, development of high-performance membranes is hindered by our limited understanding of the fundamental molecular- and nano-scale processes that determine membrane performance and degradation. To address this knowledge gap, this research project aims to design, develop, and test a new operando spectroscopic tool that will probe the chemical structure and dynamics of membranes in complex environments.

The spectroscopic tool the O'Brien group created is a permeation cell constructed from stainless steel, with stainless steel tubes connected at each end to allow gas (or liquid) to flow into and out of the cell. The main chamber contains a quartz viewport to allow access for Raman spectroscopic analysis of the membrane under realistic operating conditions. A schematic of the cell is shown below. In principle, this cell can be used to analyze membranes including metal, ceramic, and polymer membranes for many different applications and with many different types of spectroscopy. Performing operando spectroscopy on membranes in use will allow us to determine the various reactions that occur within these membranes, both those that facilitate transport and those that contribute to degradation. The permeation cell was already constructed by one of our lab's graduate students, Hui Xu, during summer 2020. When I arrived at the lab, I was tasked with developing a temperature control system for the cell.

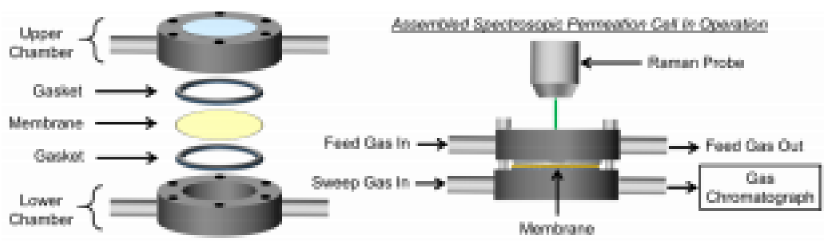

Figure 1. Schematic of operando spectroscopic permeation cell

My contribution to the research project during fall 2020 was independent planning and construction of the cell's temperature control system. I researched and ordered cost-effective and compatible temperature control equipment online, including a proportional-integral-derivative (PID) controller, solid state relay (SSR), fuses, and thermocouples to match the cartridge heaters already in stock in our lab. With assistance from graduate students, I then assembled the temperature control circuit and connected it to the reaction cell. The next step was to construct panels to permanently house this equipment; to accomplish this, I ordered stainless steel plates from a commercial provider. I then had these plates modified at the Notre Dame Machine Shop so that each piece of equipment could be permanently mounted on them. I constructed the panels by attaching supports to these plates and inserting all equipment and wiring. In total, I constructed three of these setups; one will be used to control temperature for the reaction cell, and two will be used for the lab's future projects. Images of the temperature control panels are shown below.

Figures 2 and 3. Front view of temperature control panel containing PID controller interface; back view of panel containing PID controller, SSR, fuse, and wiring.

The next step of the project was synthesis of membranes to be analyzed using the spectroscopic tool. As a first trial, Dr. O'Brien decided to test the tool on polyvinylamine (PVAm) membranes, which are commonly used for CO_2 separations in applications like carbon capture. Initially, the lab's graduate students prepared basic PVAm membranes to be analyzed. Since the start of the winter session, I have been working on synthesis of a new type of PVAm membrane that will also be applicable to future, related research projects. Under our postdoc's direction, I have been synthesizing PVAm membranes impregnated with copper silicate nanotubes. The introduction of these nanotubes is predicted to increase both gas separation and CO_2 retention due to nanotube interactions with amine groups in the membranes. For this section of the project, I first purified PVAm using dialysis tubing. Then, I synthesized the copper silicate nanotubes by reacting copper nitrate and sodium silicate in aqueous solution under high temperature and pressure. Finally, I synthesized the membranes using dip coating of the liquid PVAm onto a porous polysulfone support. As this project continues, we will insert the new PVAm membranes into the spectroscopic tool and observe differences in performance between the nanotube-impregnated and basic membranes.