SLATT UNDERGRADUATE RESEARCH FELLOWSHIP
FINAL REPORT

SCHOLAR NAME:	Benjamin Speybroeck
FACULTY ADVISOR:	Dr. Ruilan Guo
PROJECT PERIOD:	May 16th – July 20th
PROJECT TITLE:	Microporous Polymeric Membranes for Energy-efficient Gas Separations
CONNECTION TO ONE OR MORE ENERGY-RELATED RESEARCH AREAS (CHECK ALL THAT APPLY):	**(X) Energy Conversion and Efficiency** **( ) Smart Storage and Distribution** **( ) Sustainable Bio/Fossil Fuels** **( ) Sustainable and Secure Nuclear** **( ) Transformation Solar** **( ) 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
Synthesize 6FDA to dicarboxylic acid	Reflux with commercial 6FDA, Acetic Acid, Glycine for six hours at elevated temperature. Purities were accounted for.	100%
Monomer Synthesis of diacid chloride	Reflux with Dicarboxylic Acid and Thionyl Chloride overnight at elevated temperature. Product was dried using rotavapor and vacuum oven.	100%
Polymerization of PHAI	Reflux with Diacid Chloride, commercial Terephthalic Dihydrazide, Pyridine, and NMP at decreased temperature for a short time then room temperature for 4 hours. Some challenges occured which prevented high molecular weight PHAI. Using fresh monomers prevented the issue	95%
Cyclodehydration of PHAI to poly oxadiazole	PHAI is converted using thermal, solution, or microwave radiation to poly 1,3,4-oxadiazole imide.	100%
Film Casting	The oxadiazole is dissolved in solution then cast onto a glass slide to form a thin film. The film was often very brittle.	50%

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	
EXTERNAL AWARDS RECEIVED	
JOURNAL ARTICLES IN PROCESS OR PUBLISHED	
BOOKS AND CHAPTERS RELATED TO YOUR RESEARCH	
PUBLIC PRESENTATIONS YOU MADE ABOUT YOUR RESEARCH	Notre Dame Undergraduate Research Symposium, Machine Learning to Predict poly (1,3,4-oxadiazole-imide) Membrane Gas Separation Performance, 7/20/22, Jordan Hall of Science
AWARDS OR RECOGNITIONS YOU RECEIVED FOR YOUR RESEARCH PROJECT	
INTERNAL COLLABORATIONS FOSTERED	
EXTERNAL COLLABORATIONS FOSTERED	
WEBSITE(S) FEATURING RESEARCH PROJECT	
OTHER PRODUCTS AND SERVICES (e.g., media reports, databases, software, models, curricula, instruments, education programs, outreach for ND Energy and other groups)	

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 really enjoyed my research experience. I expected to learn lab technique as well as research skills, and I gained a great understanding of both. My faculty advisor and mentor both did outstanding jobs at teaching me about the project and keeping involved in the research. I would highly recommend participating in summer research.

FINAL WRITTEN REPORT

Polymer membranes used for gas separation have become a promising method for energy-efficient separation of gases. Thousands of polymers exist that can potentially be used for gas separation, and scientists simply do not have the time explore every option. To solve this problem, machine learning algorithms were trained to predict the separation performance of polymers. In this collaborative project between the MONSTER and Guo labs, three of the predicted polymers were chosen based on outstanding performance. Although these algorithms predict the performance of the polymers, it does not consider their ease of synthesis, and there is need to experimentally validate these computational models. In this aspect of the project, poly 1,3,4-oxadiazole-imide (POXI) is being investigated, it has a rigid structure as well as its excellent chemical and thermo-oxidative stability, which makes it exceptional for gas separation. POXI is commonly prepared via a two-step reaction; the first step involves low temperature polycondensation between diacid chloride and dihydrazide to obtain poly hydrazide amide imide (PHAI) while the second step involves the cyclodehydration of the PHAI to POXI. The cyclodehydration reaction could be done either by heating in solid state, in solution (with strong dehydration or silylation reagents), or with the use of microwave radiation. The polymers were characterized mainly by ^{1}H NMR, FTIR, DSC and TGA to confirm desired products were achieved at each reaction step. To evaluate the gas separation performance, a flexible thin film is prepared from the final polymer and then inserted into the permeation cells, however, due to the brittle nature of the POXI, flexible films aren't obtained still, and the gas separation performance is yet to be evaluated. Therefore, the polycondensation and the cyclodehydration method is being further tuned to obtain high molecular weight and thin flexible films.

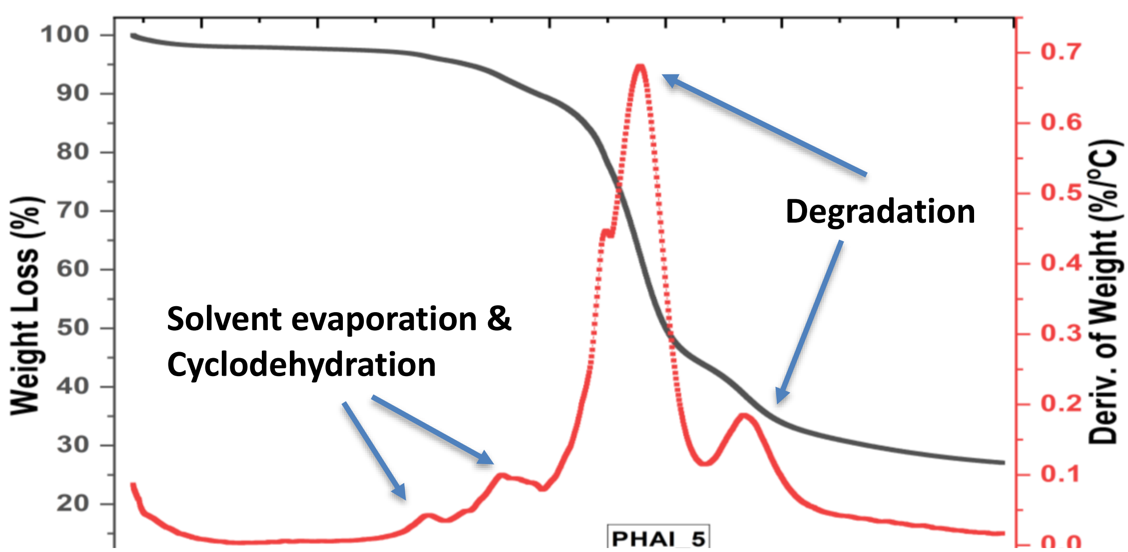

Figure 1. TGA of PHAI. Solvent Evaporation: 200-250C. Cyclodehydration: 250-350C. Two Stage decomposition: 375-650C

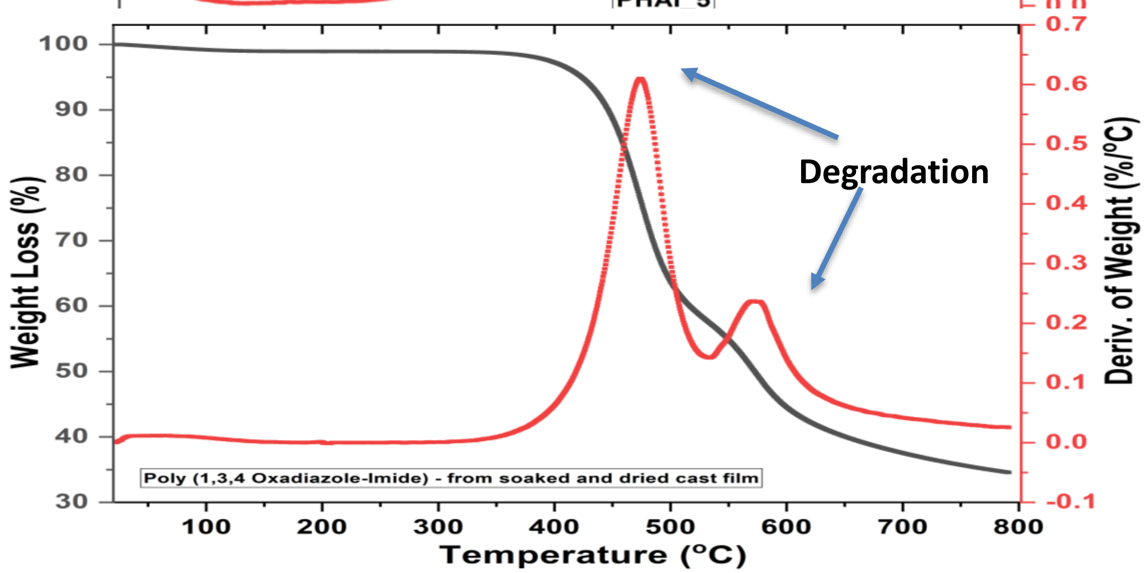

Figure 2. TGA of PHAI. Solvent Evaporation: 200-250C. Cyclodehydration: 250-350C. Two Stage decomposition: 375-650C


DMAc
DMF
DMSO