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Center for Nano Science and Technology

ND*nano* Summer Undergraduate Research
2017 Project Summary

1. Student name & university:

Mauricio A. Segovia
University of Notre Dame

2. ND faculty name & department:

David Go (AME)
William Phillip (CBE)

3. Project title: Project:

High-throughput spray coating of membranes for applications in desalination and waste chemical recovery.

4. Briefly describe new skills you acquired during your summer research:

I learned how to machine my own experimental setups using the resources in the AME machine shop and my background in machine elements. I have learned to be fully independent and systematic when it came to diagnosing and solving problems in a research setting. I was exposed to the underlying chemistry used to produce polymers for membranes and the underlying physics of the techniques used to spray the polymeric material. In terms of chemical analysis, I have used the following techniques:

- UV-Vis spectroscopy to measure the concentration of ions in a given solution.
- FT-IR spectroscopy to analyze a given chemical for certain functional groups.

5. Briefly share a practical application/end use of your research:

The techniques used in this research to produce the membranes could be scaled to manufacture many membranes. Production time of a membrane using one of the techniques was seen to be faster (seconds) when compared to other conventional techniques (minutes/hour). Implementation to a water filtration system could lead to an increase of clean water for places that would need it the most. In particular, given the size of the membranes produced in this research, implementation into a faucet would be relatively straight forward.

6. 50- to 75-word abstract of your project:

Membranes are an important aspect of many water filtration systems. However, many membranes produced from conventional methods have one of two functions: filtration of contaminates based on their size or filtration of contaminates based on their chemistry. In order to produce a membrane with both of these function, nanoporous membranes based on self-assembled polymers have been shown to be a promising class of membranes. The research done this summer was to build two spraying techniques of

thin-film deposition that utilized electric fields and to optimize the membrane performance based off of the spraying parameters.

7. References for papers, posters, or presentations of your research:

Summary

[1] Zhang, Yizhou, Jessica L. Sargent, Bryan W. Boudouris, and William A. Phillip. "Nanoporous Membranes Generated from Self-assembled Block Polymer Precursors:Quo Vadis?" Journal of Applied Polymer Science 132.21 (2014): n. pag. Web.

[2] Ramshani, Zeinab, Michael J. Johnson, Massood Z. Atashbar, and David B. Go. "Nature Index." A Broad Area Electrospray Generated by a Piezoelectric Transformer | Nature Index. Applied Physics Letters, 29 July 2016. Web. 22 July 2017.

PowerPoint

[3] Bain, Tom Slaymaker and Robert. "Access to Drinking Water around the World – in Five Infographics." The Guardian. Guardian News and Media, 17 Mar. 2017. Web. 22 July 2017.

[4] Water Treatment Solutions." Lenntech. N.p., n.d. Web. 22 July 2017.

One-page project summary that describes problem, project goal and your activities / results:

Water is one of the most important substances in the world. In particular, water free from biological and chemical hazardous materials are important to the health of any individual. Many of the current methods of water filtration depend on the use of membranes. Many membrane produced from conventional methods have one of two functions: filtration of contaminates based on their size or filtration of contaminates based on their chemistry. Advances in polymer chemistry and material science have produced nanoporous membranes based on self-assembled block polymer precursors[1]. These membrane can be made to have pores with well-defined sizes that can be changed based off of the method of deposition[1]. Additionally, functional groups that can trap contaminates based off of their chemistry can be integrated into the membrane after fabrication[1]. Having said this, these membranes can be readily implemented into current water filtration system in order to improve the quality of water being filtered.

The research performed this summer focused on the characterization and optimization of membrane performance based off of the spraying parameters used for the deposition. Two techniques were utilized and their apparatuses built: electrospray (E-spray) and a novel method known as piezoelectric transformer spray[2] (PT-spray). The technique of E-spray worked by pumping a conductive solution with polymeric material through a small diameter capillary tube. A high voltage was applied to the capillary tube which produced a Taylor Cone and a jet. The droplets ejected from the tip of the Taylor Cone reached the Rayleigh limit and Columbic fission occurred; this process can make nanometric droplets. The material was then deposited to a substrate in order to produce a membrane. The technique of PT-spray was essentially an electrospray. However, an alternating voltage was applied to it. The properties of the crystal are such that if a voltage is applied, mechanical stress is induced. Moreover, if the crystal experiences a mechanical stress, a surface voltage can be produced. The application of a relativity small input voltage resulted in a high voltage at the edge of the crystal. Conductive, polymeric solution was then deposited on this edge of the crystal and an electrospray was formed.

The solution used in each spray consisted of the following constant parameters: 70% dimethyl sulfoxide, 30% methyl ethyl ketone, and 2% wt. of the polymer. The variable parameter of the solution was the conductivity that was dictated by the concentration of sodium nitrate. The polymer consisted of three subgroups: a hydrophilic group which consisted of a chain of ether groups, a hydrophobic group which consisted of trifluoromethyl, and a functional group which consisted of an epoxide group. Once this solution was sprayed on the substrate by any of the techniques, the membrane was placed in an isopropyl (IPA) bath for 15 minutes in order to capture the nanostructure; if the membrane were to come in to contact with water for an extended period of time, the epoxide ring would have been opened and functionalization would not have been possible. After the IPA bath, the membrane was placed in a 5mL bath of 1.0M 1,6 diaminohexane. The diamine reacted with the membrane by opening up the epoxide ring and attaching the diamine to the last carbon group. The purpose of this functionalization was to have the nitrogen lone pairs from amine group form a coordinating bond with a heavy metal ion (i.e. copper 2+). To ensure that the membrane had indeed been functionalized, FT-IR was performed on samples before and after functionalization (see next page).

The two main performance parameter tested was the hydraulic permeability of the membrane,. For the E-spray, the spray parameter that was changed was the syringe pump flow rate. For the PT-spray, the main parameter that was changed was the time of deposition onto the substrate. The permeability results are shown on the next page.

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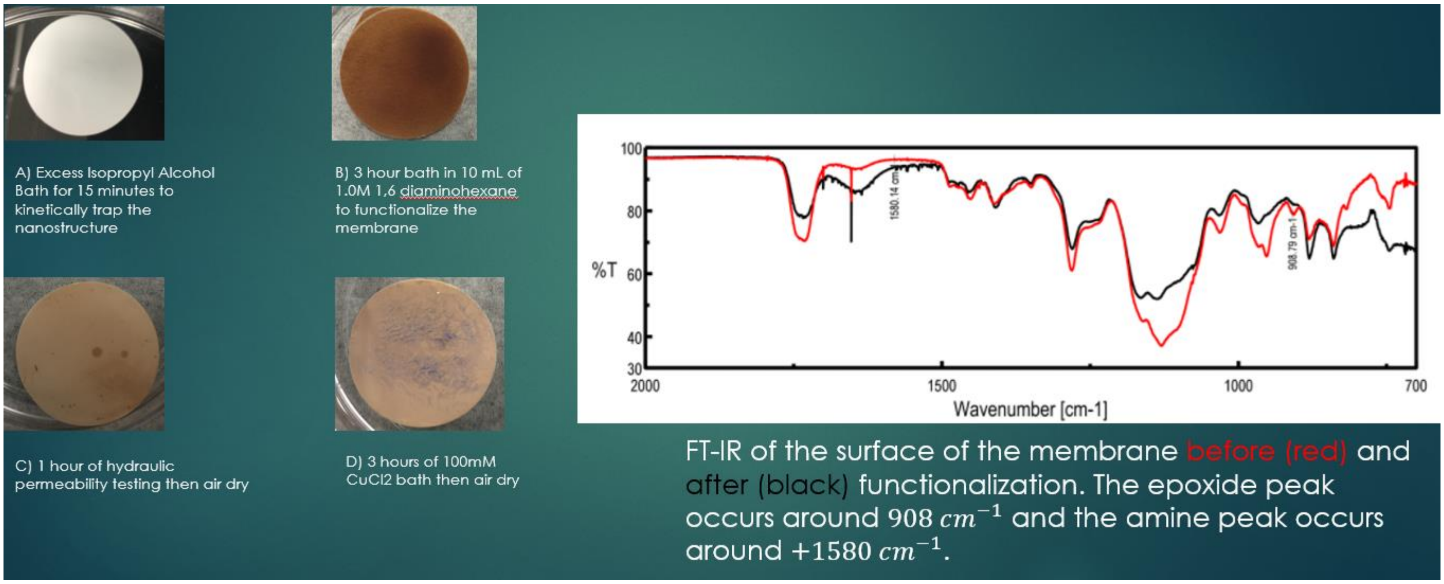

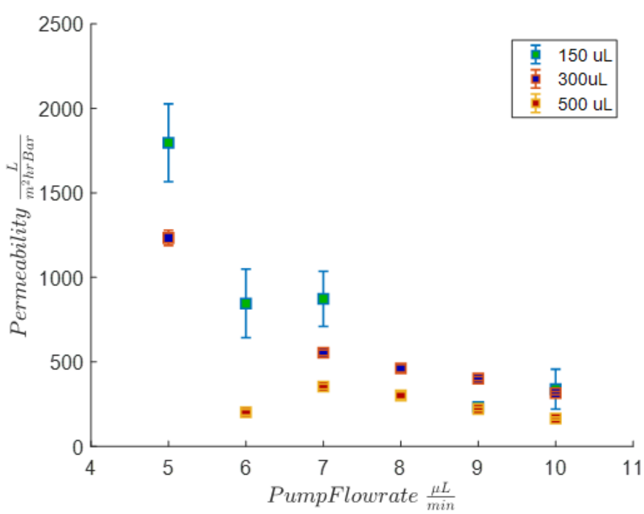

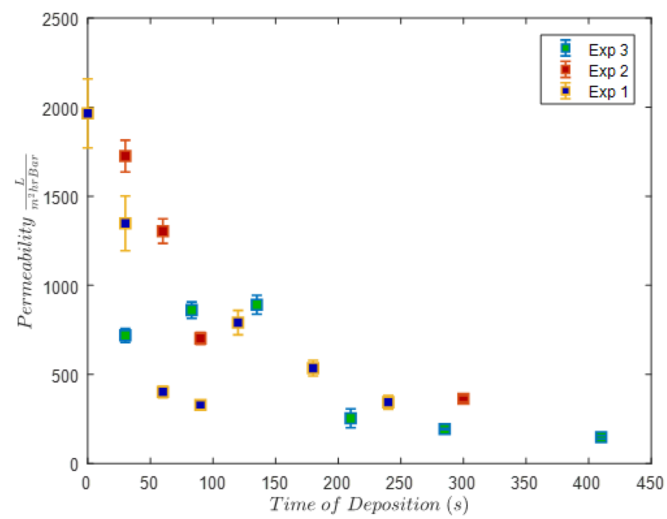

The figure to the left shows the permeability of a given membrane as a function of the syringe pump flow rate for the E-spray. This shows that as the flow rate increases, the permeability decreases for the same volume of solution deposited. Then as the volume increases for a given flow rate, the permeability tends to decrease.
The figure on the right shows the permeability as a function of the time of deposition for the PT-spray. This shows that as the time of deposition increases, the permeability tends to decrease. Permeability tests were done using a stir cell and nitrogen gas at pressures from 1-5 psi.