ND*nano* Undergraduate Research Fellowship (NURF)
2012 Project Summary

1) Student name: Brittany Hark
2) Faculty mentor name: Tao Wang and Abhijit Biswas
3) Project title: Synthesis of hybrid cellulose/metal nanocomposites

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

I have learned various synthesis procedures including how to use liquid Nitrogen to freeze-dry samples. I also learned how to use green manufacturing to produce nanocomposites with excellent mechanical properties and functionalities. Additionally, I was trained to use an Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) to analyze various concentrations of elements within a given sample solution, as well as an Ultra-violet visible spectrometer (UV-Vis) to detect the absorbance of different analytes. Finally, I gained excellent experience in researching, reviewing, systematic analysis and scientific writing through papers and reports.

5) Please briefly share a practical application/end use of your research:

By synthesizing cellulose/metal nanocomposites, specifically those with Silver nanoparticles throughout the composite's porous structure, we hope to continue to improve the mechanical properties and functionalities of the novel structure. This composite could be used in water filtration to remove mercury and other toxic elements due to the chemical reaction between silver and that metal. It can also be applied for future use as a semi-conductive material, battery electrode, and/or as an ion exchange source in various filtration devices.

Project summary:

Cellulose nanocomposites have the ability to be used as novel materials because of the excellent mechanical properties and functionalities that cellulose itself possesses. However, that being said, it is important to utilize and exploit these properties to the best of their abilities. To do so, the structure of a 3-dimensional cellulose microfibril aerogel needed to be investigated. A synthesis approach was needed in order to produce a composite with structural stability, ideal pore size, and a high surface to area ratio. Once this can be achieved, the task of producing a composite with silver nanoparticles throughout its matrix was addressed. Our goal was to create a nanocomposite with ordered metallization that reduced the amount of silver aggregation but increased the control of deposition. Characterization of the cellulose/silver nanocomposites could be used to help identify ways to optimize the synthesis process and to prepare for application of the nanocomposites. Once synthesized, our goal was to determine whether the composite was able to be used as an ion exchange source in water filtration, specifically in removing mercury from water.

Through systematic chemical analysis, I worked alongside Chunhua Yao, a Chinese exchange graduate student, to go about solving the aforementioned problems and working to achieve our goals. Starting with the structure of cellulose, we researched and designed a freeze-drying method to preserve its sheet-like structure and to produce a matrix with percolating pores. We then wanted to create our metal nanocomposite. Through oxidation and reduction reactions, we treated our cellulose samples with $NaIO_4$ and a silver nitrate/ammonia solution, respectively. This created the necessary functional groups to act as the locus of deposition to which silver would attach. Analysis of the cellulose/silver nanoparticle composites was done by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). One such image from the SEM is shown below with visible silver particles throughout the cross-section of the cellulose structure.

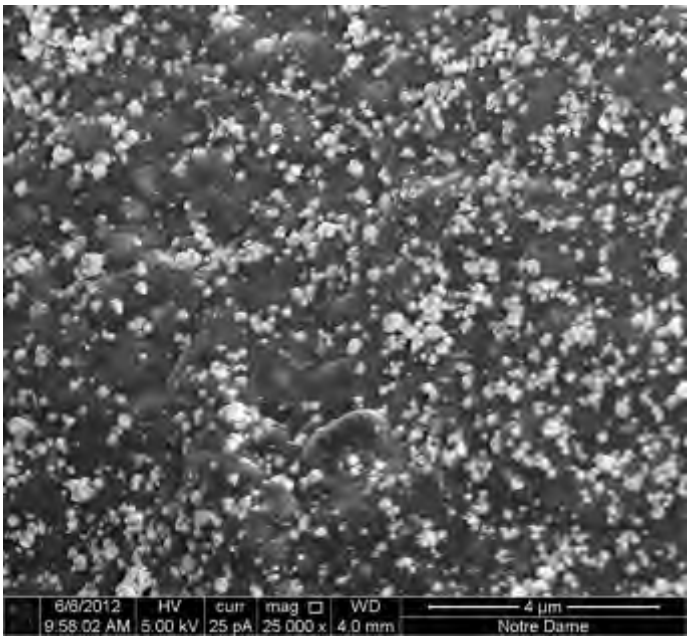

Figure 1. An SEM image of silver nanoparticles distributed on the cellulose matrix. The distribution of silver was controlled by the cellulose oxidation method and Na_2EDTA.

Optimization of this dispersion process was researched and implemented. Agglomeration was one area of improvement as we tried to reduce the size of the silver nanoparticles so as to improve their functionalities in future work. A complexing agent, Na_2EDTA, was added to the composite to slow down the reaction time between the silver ions and the –CHO functional groups on the cellulose. This not only decreased nanoparticle size but also improved uniformity of distribution throughout the cellulose matrix. The oxidation method used was also researched. We used two methods, that of electroless plating and of selective oxidation. These two methods showed us that the mode of silver deposition is greatly dependent on the form of the reducing agent, whether it was a dissolved species or a matrix immobilized species. The method of selective oxidation provided us with improved results of silver deposition as characterized by EDX, which estimated an average silver loading to carbon weight percent at 47.9%. Having achieved our initial goals, we continued to work on the optimization process of immersion times, pH and solution concentrations through further analysis by ultra-violet visible spectroscopy and pH analysis. An image of the experimental synthesis procedure steps is shown below indicating the changes that the cellulose hydrogel underwent.

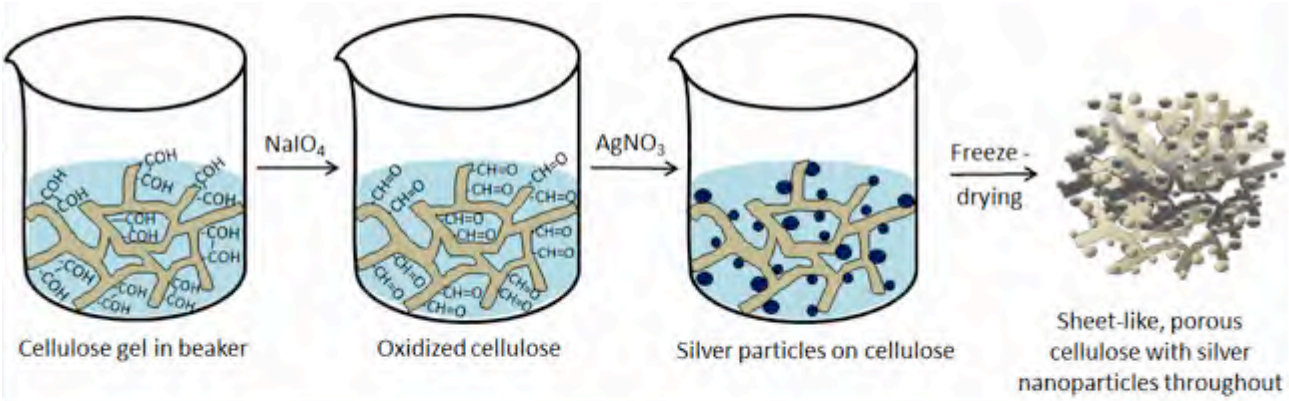

Figure 2. A schematic diagram showing the preparation process for the cellulose/silver nanoparticle composite.

Our research continued with the application of our novel cellulose/silver nanoparticle composite. We began by applying it to water filtration in hopes that it would remove the mercury from a water solution due to reaction between silver and mercury. We created a mercuric solution to determine the amount of mercury that reacted with the silver and thus was absorbed by the composite. We used two methods, that of filtration and immersion to determine its absorption qualities. Analysis was done with an Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES). Our composite was able to remove a given amount of mercury from the water without depositing a large amount silver back into the solution. Results are still pending but they are promising.

Publications (papers/posters/presentations):

Together, Chunhua and I co-authored a communication paper. It is currently in the publication process. Awaiting some final editing, our paper, "Design and synthesis of porous cellulose scaffold/silver nanoparticles composites" will hopefully be published in the *Journal of Materials Chemistry*, published by the Royal Society of Chemistry in the United Kingdom.

I also presented a poster at the 2012 Summer Undergraduate Poster Symposium on the work that I have done over the past ten weeks. (A copy of my poster can be found in the attached file below.)

NDnano Poster_BrittanyHark.pdf