UNIVERSITY OF NOTRE DAME NDnano CENTER FOR NANO SCIENCE AND TECHNOLOGY

ND*nano* Undergraduate Research Fellowship (NURF) 2015 Project Summary

1. Student name: Diego Rosas Villalva
2. Faculty mentor name: Dr. Kyle Doudrick
3. Project title: Nano-structured arrays for sustainable water treatment technologies.
4. Briefly describe any new skills you acquired during your summer research:

   In this project I strengthened my skills on thin films deposition and synthesis of nanostructures using electrochemical and thermal methods. I gained more knowledge on electrochemistry while using a potentiostat to perform and monitor photoelectrochemical reactions. I also got training on Dynamic Light Scattering, zeta potential and X-ray Fluorescence to characterize the synthesized materials.
5. Briefly share a practical application/end use of your research:

   Water is one of the most important resources for humanity however the industrial activity as well as human waste are making it less available. The photoactive semiconducting materials are a promising technology for water treatment and when processed into a nanostructured architecture their efficiency improves. These are capable of transforming sunlight into electric potential that will help us breaking harmful organic pollutants in water that threaten both wild life and human development. Since the sun provide us with a huge amount of light every day this is considered a sustainable technology. The resultant products are carbon dioxide and hydrogen gas. This last one could be stored and used to produce energy.
6. Begin two-paragraph project summary here (~ one type-written page) to describe problem and project goal and your activities / results:

   Photoactive semiconducting materials are typically used in the form of a noparticulated powder, this zero-dimensional materials have a great surface area which is the main reason why they achieve a high efficiency removing materials. However, because of their small size, separating them from the water may become a very hard task. One approach to solve this problem is supporting the material as a film on a substrate such as a fluorine doped tin oxide (FTO) glass, which will allow us to perform the removal in an electrolytic cell and separate hydrogen and carbon dioxide in the different electrodes. On the other hand this will cause a big lost in the amount of surface area thus decreasing dramatically the efficiency. Thus the goal of the project is to improve the efficiency of the films for water treatment.

   My activities included the preparation of various semiconducting materials in powder form, including BiOI, C_3N_4, BiOBr and $BiVO_4$. I compared how useful they were to remove methylene blue, a common dye used in the textile industry, by reading the change in absorbance with an UV-Vis spectrometer. The Bismuth Vanadate ($BiVO_4$) gave the most promising results so I started to prepare films of this material using electrochemical deposition followed by calcination with vanadium acetylacetonate, the films can be seen in figure 1. I used linear sweep voltammetry and chronoamperometry techniques to characterize the photoactivity of the film using a xenon arc lamp as the source of radiation. I also characterized the film using XRF to determine element content. The capacity of the films to remove methylene blue from water was also tested applying 0.5 V and using certain amount of sodium chloride NaCl to improve the current and charge separation in the electrolytic cell.

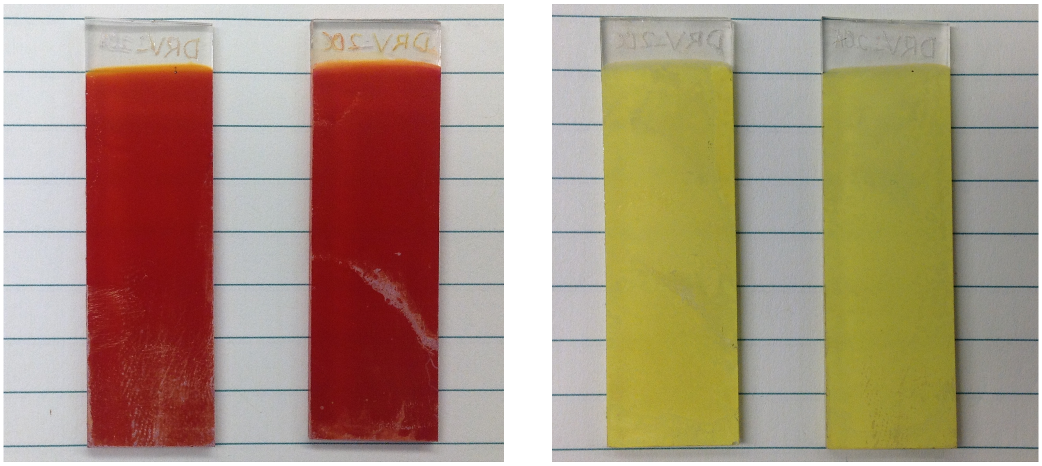

Figure 1. Films Prepared by electrodeposition prior (left) and after (right) calcination. The red material is BiOI which serves as a template for the yellow $BiVO_4$.

It is expected that higher currents will achieve a higher removal so different electrolytes were tested to see which one would achieve the biggest current, although sodium sulfite (Na_2SO_3) achieves the highest current among the tested salts, it reduces the methylene blue into a colorless compound, making it hard to track the reaction using UV-Vis spectrometry. Other electrolytes included sodium sulfate (Na_2SO_4), phosphate buffer, NaCl which resulted to be the best. Two concentrations of NaCl were tested, 1M and 0.1M, from which the last one achieved the highest removal.

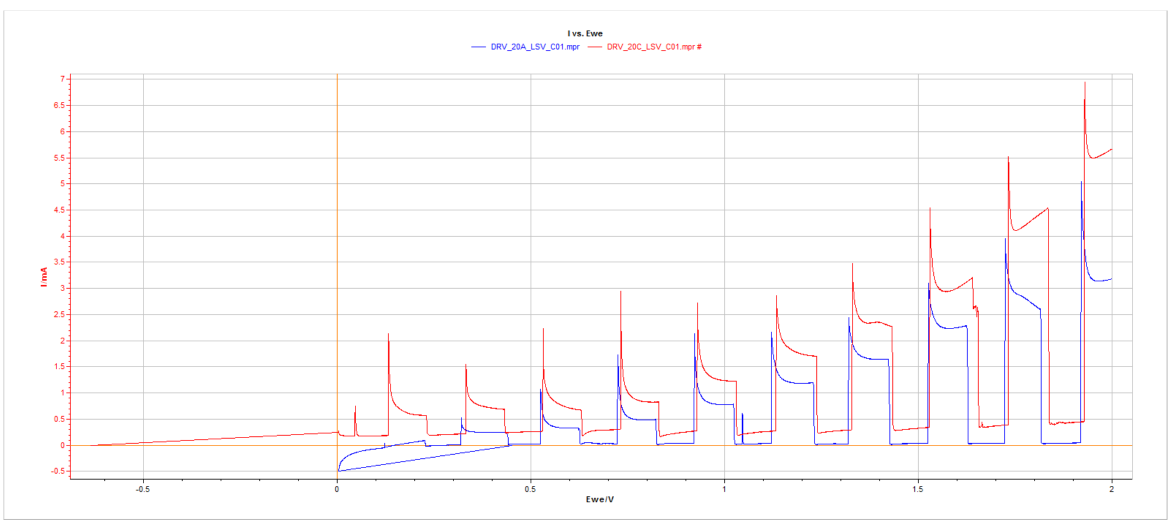

Figure 2. Linear sweep voltammetry of the two films in figure 1. The high bands are produced by illuminating the film the low bands are the result of interrupting the illumination.

One of the first drawbacks of the films is the small amount of mass from a semiconductor that you can put on a film. While the standard for the powder is typically 1 g of $BiVO_4$ per liter of solution, this films contain only from 1.6 to 1.8 mg in an area of 2.25 cm^2 of the material, meaning that to observe comparable results, solutions with very diluted concentrations of methylene blue should be used, otherwise a very small amount of solution. However if we compare the results obtained by the mass ratio of MB to $BiVO_4$, one gram of the powder would be able to remove about 9 mg of methylene blue while one gram of the film would be able to remove about 64 mg of the dye. Still we would need a film with an area about 4 times larger to achieve the same removal as the powder. Next steps in the research include a more extensive characterization such as XRD, SEM and DRIFTS as well as loading a cocatalyst to improve the efficiency of the film.