NM WRRI Student Water Research Grant Final Report
Final Report due June 30, 2017

1. **Student Researcher:** Stephanie Richins, New Mexico State University
Faculty Advisor: Hongmei Luo, New Mexico State University

2. Project title: Oxidation of Arsenite by a Carbon Nitride Photocatalyst with Graphitized Polyacrylonitrile

3. Problem Statement and Objective:

Arsenic is prevalent in the well water and stream water in southern New Mexico. The Arsenic seeps into the water table from sites where copper mining has taken place. Copper mines use arsenic during the smelting process, and unfortunately many companies do not deal with the disposal of arsenic in an environmentally safe way. Arsenic in water is called 'arsenite'. This form of arsenic (Arsenic III) is toxic to humans. Arsenic III normally does not have any flavor or odor, so people are not aware of its presence if they are drinking water contaminated with it. Arsenic III is a carcinogen, meaning it can cause humans to develop cancer. If untreated this can lead to death. When exposed to light, Arsenic III along with a photocatalyst can transform into a higher oxidation state called Arsenic V; in water this is called "arsenate". Arsenic III is 60 times more toxic than Arsenic V [1]. Arsenic V is therefore much less toxic to humans, and relatively environmentally benign.

There are many ways people have been removing impurities from drinking water. Many involve boiling the water or running it through a filter. Arsenic does not boil out of water, and to target arsenic most people must buy a specific filter for arsenic. Modern arsenic filters often cost upward of $1000.

The research objectives for this project were to:

- Find a photocatalyst that was both environmentally safe, and able to facilitate in the transformation of Arsenic III into Arsenic V.
- Successfully synthesize the photocatalyst.
- Use the instrumental techniques of X-ray diffractometer (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), and SEM-Energy Dispersive X-ray analysis (EDX) to characterize the samples.
- Use a solar simulator ultraviolet light source alongside my phototcatalyst to oxidize the Arsenic III into Arsenic V.
- Use an inductively coupled plasma mass spectrometer (ICP-MS) to understand the ratio of Arsenic V to Arsenic III.

4. Methodology

- The photocatalyst, graphitic carbon nitride (g-C_3N_4), was synthesized:
    - Melamine was heated (650°C) for 3 hours under inert gas (Argon).

- The g-C_3N_4 was put through the following characterizations:
  - XRD - to identify the crystalline materials, and their phases
  - FTIR - to identify organic, and inorganic materials, and observe their properties
  - TEM - to image nanomaterials and to observe their morphology, particle distribution and size.
  - SEM - to better understand the samples' composition and topography.
  - SEM-EDX - used to identify elements, and the composition of the sample as well as the ratios the elements are in.

- 50mg of g-C_3N_4 was added to a mixture of 0.672g Citric Acid and 1.911g Sodium Citrate in 100 mL Deionized water. 1mg Sodium (Meta) Arsenite was added to 100 mL Deionized water. 1mL of the Sodium (Meta) Arsenite solution was added to the g-C_3N_4 mixture. The solution was stirred at 350 RPM for an hour.
- The solution was placed under a solar simulator, while continually being stirred. Periodically every 10 minutes, 10 mL solution was removed to test the effect of the time the sample was exposed to the intensified UV light.
- These samples were tested using an ICP-MS to see how much of the arsenic still remained and which state it was in.

5. **Description of results; include findings, conclusions, and recommendations for further research.**

Characterizations:

XRD

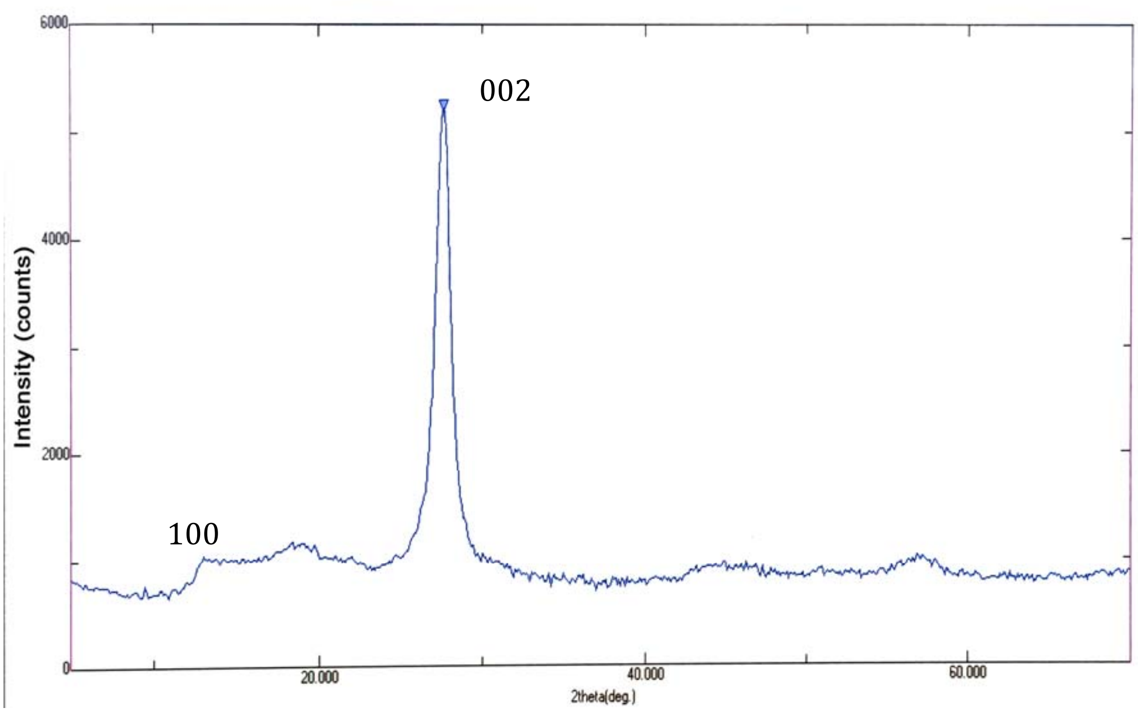


Figure1: shows the XRD data from g-C_3N_4.

FTIR

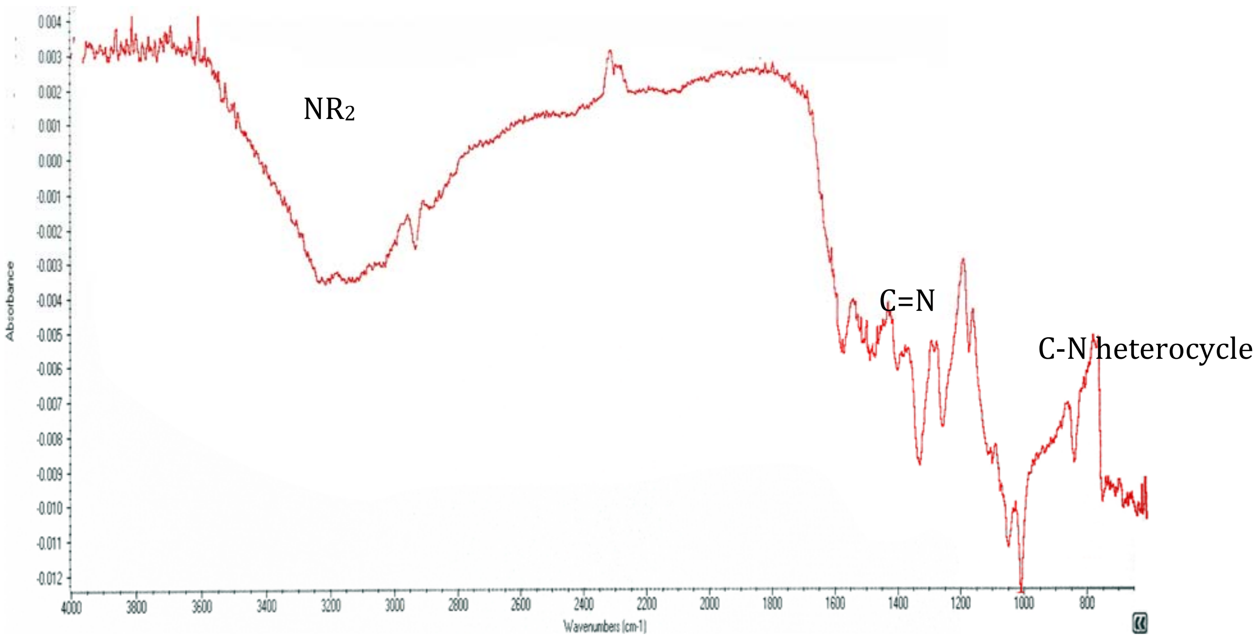


Figure 2: shows the FTIR of g-C_3N_4.

TEM

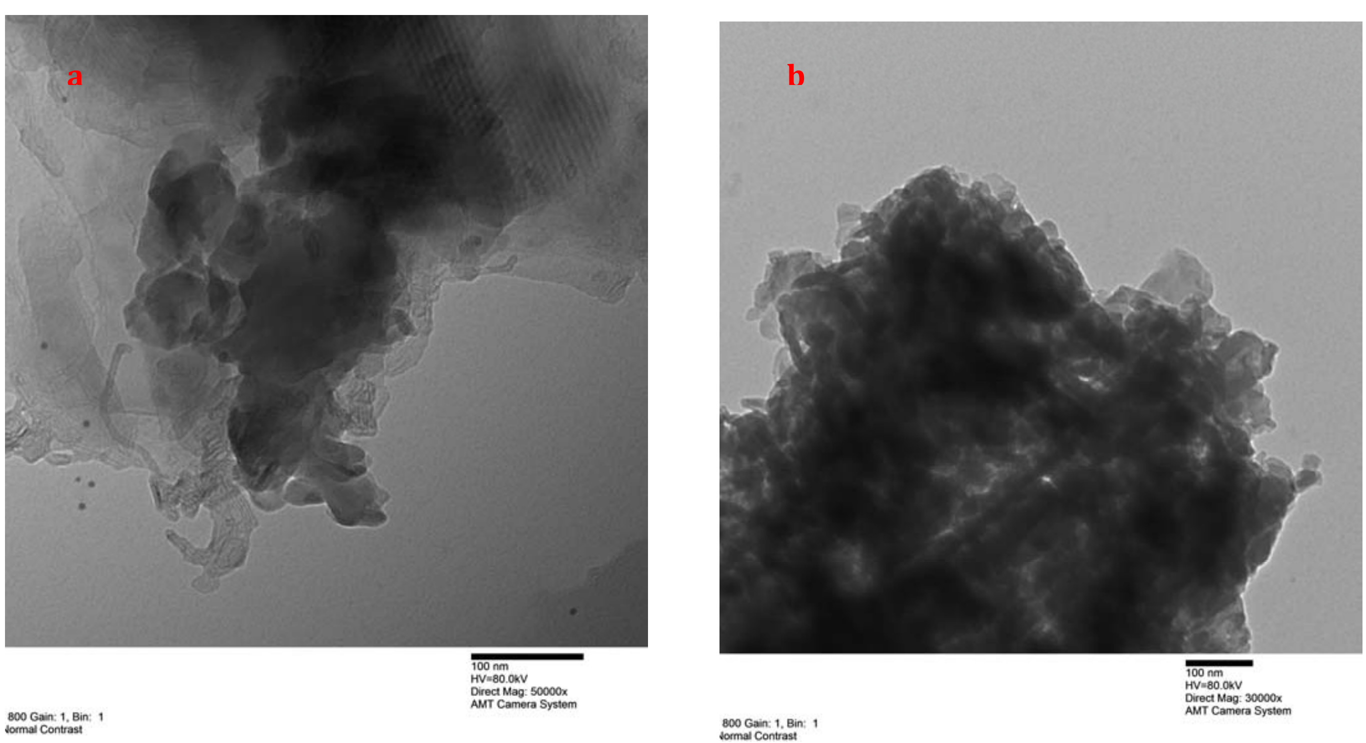


Figure 3:a,b shows TEM images of g-C_3N_4

SEM

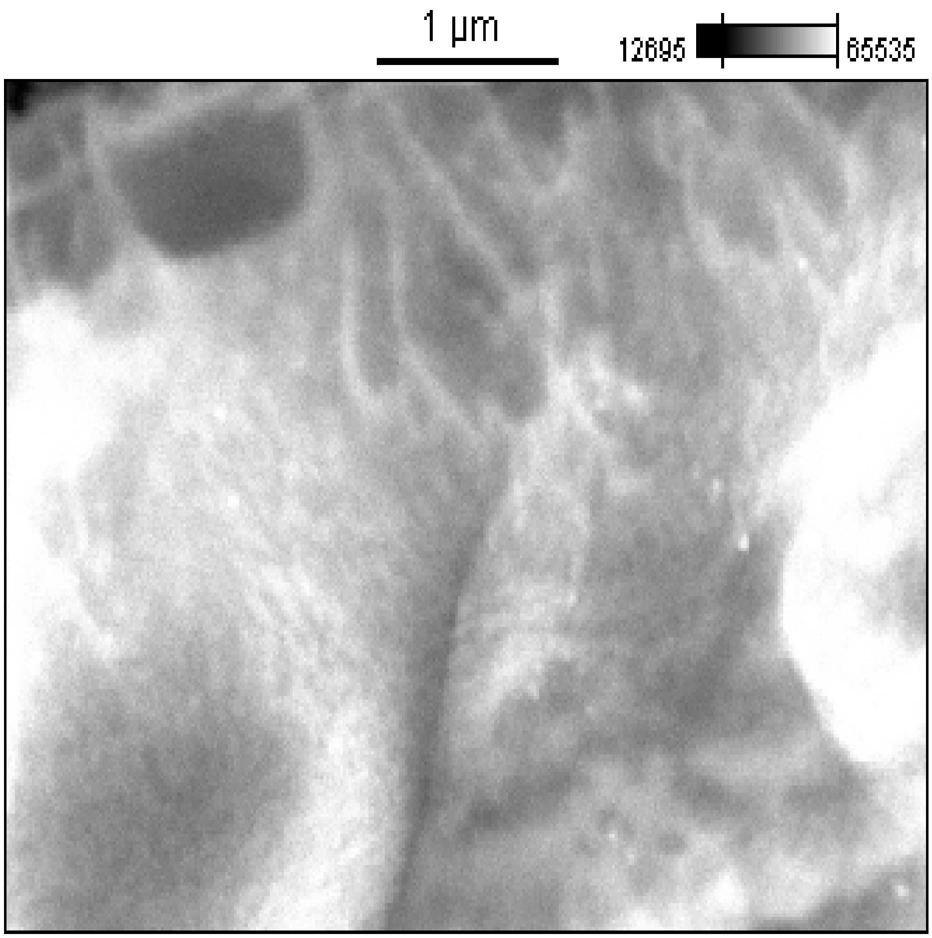
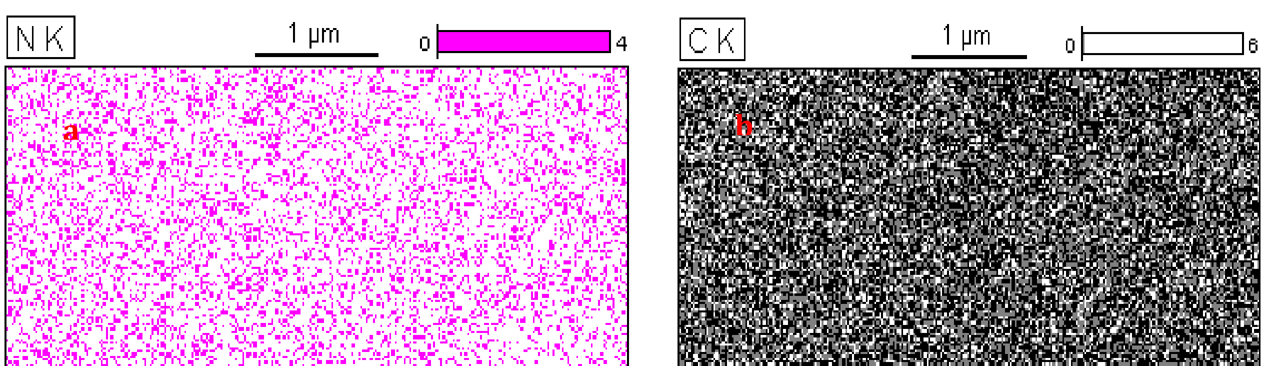


Figure 4: displays the SEM of g-C_3N_4

SEM-EDX



Element	Element	Wt.% Atom %	Atom %	c
Line Wt.%	Error	Error		
C K 43.07 ±	0.31	46.87	0.34	
N K 56.93 ±	1.38	53.13	1.28	
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Total 100.00	100.00			

Figure 5: shows the elemental distribution mapping for a) Carbon and b) Nitrogen and c) the Wt% of the composition of the g-C_3N_4.

Analysis:

The XRD displayed a graphite-like structure. The 002 peak occurs at a diffraction angle 2θ of 27.3° and the 100 peak occurs at 12.8°.

FTIR: the characterization peak around 3300 cm^{-1} indicates a NH_2 (H-N-H) bond. The peak around 1350 cm^{-1} shows a C=N bond. The 1000 cm^{-1} peak represents C-N, and the 900 cm^{-1} peak demonstrates a heterocycle.

The TEM displays a sheet- like structure. The sheet like structure has an increased surface area, which allows for more active sites. A greater number of active sites means the photocatalyst can react more completely with the arsenite.

The SEM shows that the g-C_3N_4 has no specific morphology. It is important to understand the morphology because different morphologies can impact the way particles will react with one another.

EDX provides elemental mapping. The mapping showed that the correct ratio (3:4) of carbon to nitrogen was achieved during the synthesis.

All of these characterizations help to better understand how the photocatalyst reacts with arsenite.

ICP-MS

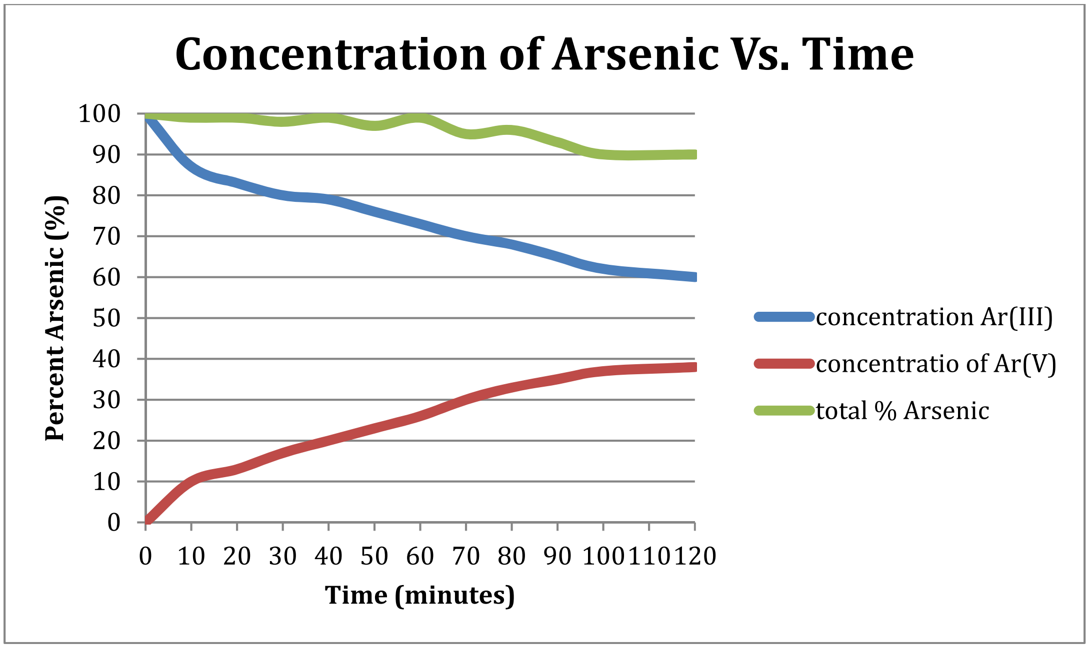

Figure 6: displays the concentration of Arsenic (V), Arsenic (III), and the total concentration of Arsenic.

The ICP-MS shows the transformation of toxic arsenite to relatively nontoxic arsenate. In just 2 hours almost half of the arsenic III was converted to arsenic V.

I would recommend that further research is done so that this problem can be better understood and dealt with.

6. Provide a paragraph on who will benefit from your research results. Include any water agency that could use your results.

The main Beneficiary for my research is anyone who has arsenic in water. Arsenic III is toxic and has been shown to lead to major health problems. Altering the toxicity can be helpful, because drinkable water is getting more scarce in New Mexico.

Water agencies who work on arsenic contamination would be able to utilize my research. The EPA has restrictions on the amount of arsenic in water;, this research could help them find a cost effective way to better control Arsenic.

7.Describe how you have spent your grant funds. Also, provide your budget balance and how you will use any remaining funds

There would be no left- over funds.

Salary- $6000
Travel-$277

8. List presentations you have made related to the project.

- Water Resources Research Institute Meeting, Socorro, New Mexico; August 2016
- American Physics Society Annual meeting, Las Cruces New Mexico; September 2016
- New Mexico Academy of Science Annual Meeting, Albuquerque, New Mexico; October 2016

9. List publications or reports, if any, that you are preparing. Remember to acknowledge the NM WRRI funding in any presentation or report that you prepare.

None

10. List any other students or faculty members who have assisted you with your project.

Lara Teich (high school student)

12. Provide information on degree completion and future career plans. Funding for student grants comes from the New Mexico Legislature and legislators are interested in whether recipients of these grants go on to complete academic degrees and work in a water-related field in New Mexico or elsewhere.

I have finished my freshman year at NMSU. I plan on graduating with my bachelors in chemical engineering in 2020. I hope to continue on to Graduate school.

References:

[1] Ratnaike, R N. "Acute and Chronic Arsenic Toxicity." *Postgraduate Medical Journal*, The Fellowship of Postgraduate Medicine, 1 July 2003, pmj.bmj.com/content/79/933/391.