**Hydrothermal Synthesis of Ni$_x$S$_y$ using Microwave Irradiation**

Katherine Wright, H.M. Morales, and J.G. Parsons

UNIVERSITY OF TEXAS RIO GRANDE VALLEY

---

**ABSTRACT**

NiS$_2$ nanoparticles were synthesized via microwave irradiation, using sodium thiosulfate and nickel (II) nitrate hexahydrate in an aqueous media. The synthesis occurred at varying temperatures from 120°C to 200°C in 20° increments. Each synthesis was performed at temperature for 30 min. In addition, a second synthesis was performed at 200°C for a total time of 2 hours to determine if reaction time had effects on the products from the synthesis. The products were analyzed using XRD and SEM to determine the effects of temperature on nanoparticle size and product composition. Composition and morphology of the nanoparticles were examined based on heating time and temperature. In addition, the crystallite size of the particles was calculated from the XRD analysis. As well, the nanoparticles were observed to have a spherical morphology, determined from the SEM imaging.

**MATERIALS AND METHODS**

Approximately a 1:1 ratio of nickel nitrate hexahydrate and sodium thiosulfate were dissolved in 500mL of 18MΩ deionized water. The mixture was divided into equal portions and placed in a Teflon lined digestion bomb. Using microwave irradiation, the solution was heated to the desired temperature, cooled, filtered, and washed with portions of 18MΩ deionized water, methanol, and acetone. The solution ran at 6 different temperatures from 120°C to 200°C at 20° increments for 30 minutes each. In addition, the solution ran at 180°C for 2 hours.

---

**BACKGROUND**

NiS$_2$
- Crystallizes in pyrite structure with +4 oxidation state
- Sulfur removal through HDS reactions for oil refining
- MoS$_2$ promoter in HDS reactions
- Other techniques used to synthesize nanoparticles

Microwave Irradiation
- Applied to compounds other than NiS$_2$
- Increased reaction rate and low temperatures
- Small deviations in grain size

**PURPOSE AND HYPOTHESIS**

- Green chemistry
  This experiment performs the hydrothermal synthesis of NiS$_2$ nanoparticles which will be analyzed via XRD and SEM to determine average particle size, composition, and morphology.

**RESULTS**

- Intensity increase
- 2 phases present
- NiS$_2$ and Ni$_9$S$_8$
- Stabilization at higher temperatures
- Increase in particle size with temperature

**CONCLUSIONS**

- Begin hold time after temperature is met
- Increase hold time
- 2 phase to 1 phase
- Apply to Cobalt, Iron, and Lead

**ACKNOWLEDGEMENTS**

Thank you, Dr. Parsons, and Ms. Morales for the constant help, support, and assistance. Also, thank you to Danny Ramirez, and other graduate students for the laboratory assistance. Lastly, thank you to the UTRGV, the COS program for this research opportunity, and the Welch foundation.