Hydrothermal Synthesis of Ni_xS_y using Microwave Irradiation

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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 at 200°C was performed 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 using. The presence of 2 phases, $NiS_2$and Ni_9S_8, were determined in the products from the XRD analysis. As well, the nanoparticles were observed to have a spherical morphology, determined from the SEM imaging.

BACKGROUND

NiS2
- Crystallizes in pyrite structure with +4 oxidation state
- Sulfur removal through HDS reactions for oil refining
- MoS2 promoter in HDS reactions
- Other techniques used to synthesize nanoparticles

Microwave Irradiation
- Applied to compounds other than NiS2
- Increased reaction rate and low temperatures
- Small deviations in grain size

PURPOSE AND HYPOTHESIS

- Green chemistry

This experiment performs the hydrothermal synthesis of NiS2 nanoparticles which will be analyzed via XRD and SEM to determine average particle size, composition, and morphology.

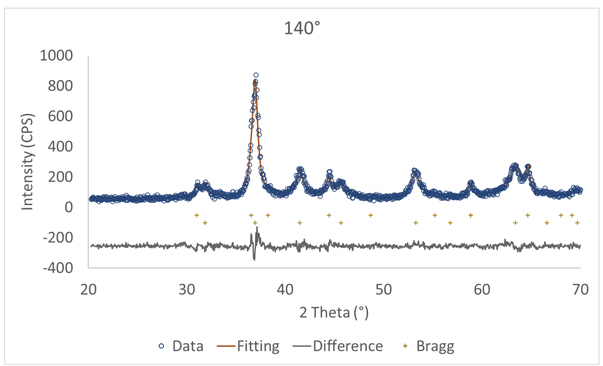


Figure 1: Fullprof fitting of Ni_xS_y synthesized under microwave irradiation at 140°C

MATERIALS AND METHODS

Approximately a 1:1 ratio of nickel nitrate hexahydrate and sodium thiosulfate were dissolved in 500mL on 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.

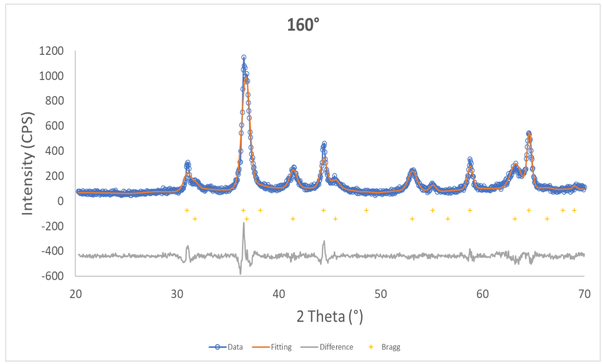


Figure 2: Fullprof fitting of Ni_xS_y synthesized under microwave irradiation at 160°C

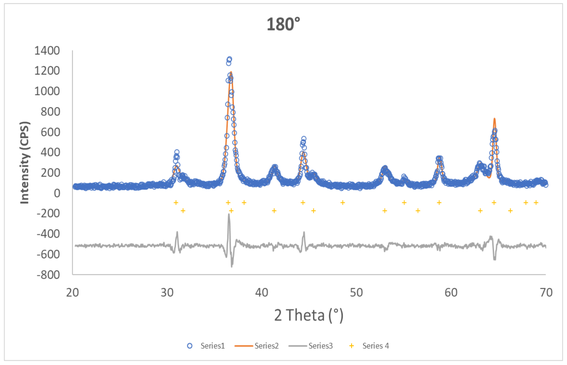


Figure 3: Fullprof fitting of Ni_xS_y synthesized under microwave irradiation at 180°C

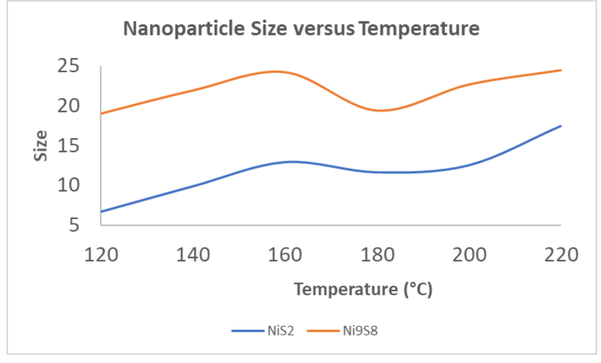


Figure 6: Effect of Temperature on particle Size for each Ni_xS_y Phase synthesized

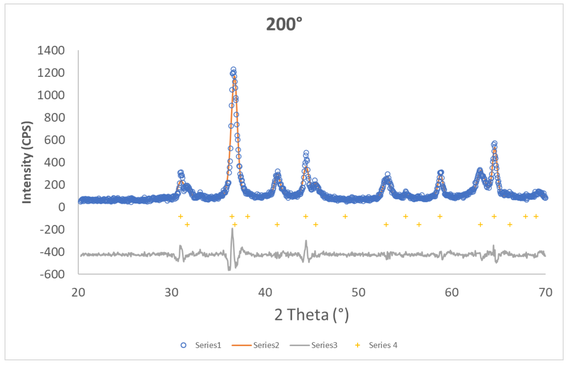


Figure 4: Fullprof fitting of Ni_xS_y synthesized under microwave irradiation at 200°C

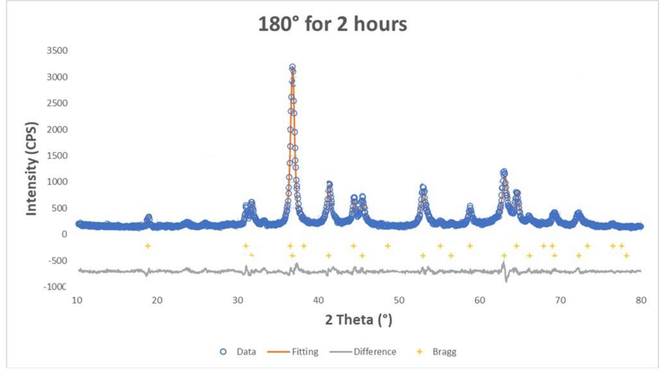
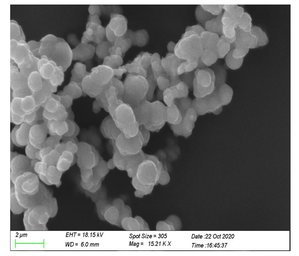
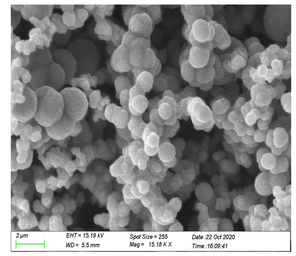


Figure 5: Fullprof fitting of Ni_xS_y synthesized under microwave irradiation at 180°C for 2 hours

RESULTS

- Intensity increase
- 2 phases present
- Ni9S8 and NiS2
- Stabilization at higher temperatures
- Increase in particle size with temperature





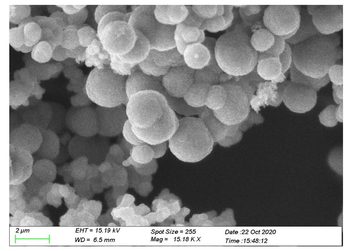


Figure 8: SEM of NixSy synthesized at 120°C (Top left) 160 °C (Top Right) and 200 (°C) bottom.

CONCLUSIONS

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

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