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# Facile synthesis of nickel sulfide hierarchical structures and its phase changes

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## 1. Introduction

Development of advanced inorganic materials has been attracting much attention in the recent years due to its tunable size, morphology and properties [1]. Among the family of metal sulfides, nickel sulfide (NiS) has its own importance, because of the variety in its phases [2] and diversity in applications. Synthesis of nickel sulfide hierarchical structures were previously discussed by several research groups, but there is no report on the structural changes with respect to the concentration on the formation of hierarchical morphologies by hydrothermal method. In the present work, monodispersed NiS hierarchy structures have been synthesized by a facile hydrothermal method using Ethelenediaminetetraacetic acid (EDTA) as a capping agent. The role of sulfur source, nickel source and capping agent concentration on the formation of nickel sulfide hierarchy structures was investigated. Formation mechanism of hierarchy structures with respect to the phase change was studied.

## 2. Experimental method

Synthesis of nickel sulfide hierarchy structures is as follows: 0.5 M concentration of $Ni(NO_3)_2.6H_2O$ was dissolved in 0.1 L of water. 1 M of thiourea sulfur source was added to the nickel nitrate solution. Concentrations of the source materials were fixed. Ethelenediaminetetraacetic acid (EDTA) was used as a capping agent. A mixture of the above source materials was stirred continuously for 60 min, then the solution was transferred to the Teflon-lined autoclave of 0.1 L capacity for hydrothermal reaction at 160 °C for 10 h. After the hydrothermal reaction, the precipitate of nickel sulfide was filtered, washed with distilled water three times and dried at 60 °C. To understand the formation mechanism of hierarchy structures, concentration of sulfur source, nickel source and capping agent was varied in different experiments. Along with that, lesser concentrations of source materials i.e., A/10, A/7.5, A/5, A/2.5 were performed at the same condition for the fixed concentrations of source materials A. Synthesized nickel sulfide materials were characterized by FESEM, XRD, TEM, FTIR and EDAX analysis.

## 3. Results and discussion

Fig 1 shows the XRD profiles of the samples with different EDTA concentrations. The EDTA concentration was varied from 0, 0.2 and 0.4 M (0.5 M nickel nitrate, 1.0 M of Thiourea). The synthesized material consisted of several phases of nickel sulfide ($NiS_2$, $Ni_3S_4$ and $Ni_{17}S_{18}$) for the uncapped experiment. For 0.2 M of EDTA, the number of the phases was reduced to $Ni_3S_4$ and $Ni_{17}S_{18}$. Further increase in the concentration to 0.4 M formed unreacted sulfur and $NiS_2$ phase. Fig 2 shows the FESEM images of different concentrations. For the uncapped experiment, hierarchical structures (1 - 8 μm) composed of different sizes of rods were synthesized. For the concentration of 0.2 M, sheets-composed hierarchical structures (6 - 8 μm) were formed as shown in fig 2 b. When the EDTA concentration was increased to 0.4, sphere-like hierarchical structure composed of cubic nanoparticles was synthesized. The addition of EDTA controlled the phases and acted as a shape modifier for the hierarchical morphologies. Similarly, concentrations of sulfur source and nickel source were varied. The results will be displayed at the conference.

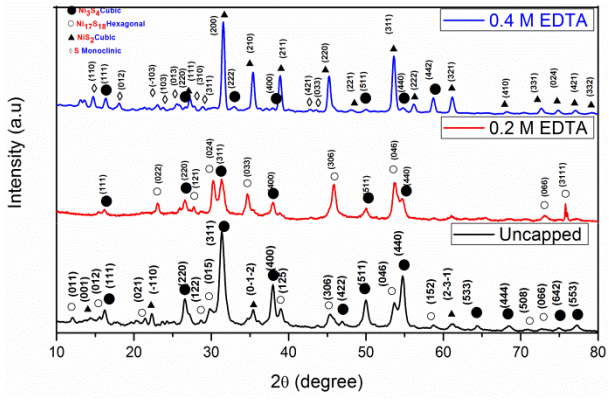
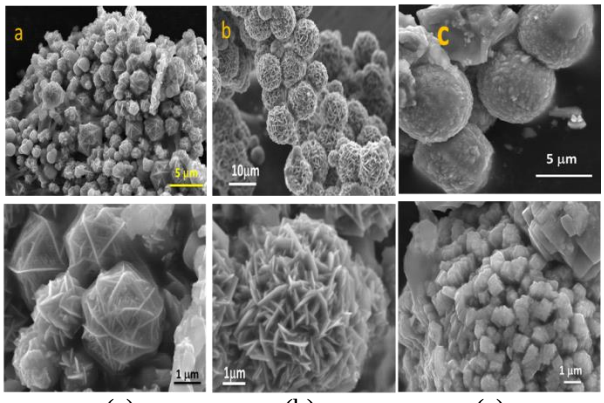



Fig 1: XRD profiles of the samples with different EDTA concentrations .




(a) (b) (c)

Fig 2: FESEM images a) Uncapped b) 0.2 M c) 0.4 M EDTA concentrations.

**[Reference]**

1. Yiming Chen et.al, J.of Nanomaterials, **6** (2012) 601736.
2. G. Kullerud and R. A. Yund, J. Petrol., **3** (1962) 126.