

Protocol for synthesis of DADS-nFeS

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Method Article

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Abstract

This protocol describes the detailed procedure for preparation of DADS-nFeS. To get our target nanoparticle with stable property, we synthesised DADS-nFeS by solvothermal method with high temperatures and pressures.

Introduction

This protocol describes the detailed procedure for preparation of DADS-nFeS. We synthesized DADS-nFeS by solvothermal method under high temperature and pressure in a sealed autoclave reactor.

Reagents

ethylene glycol, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, sodium acetate (NaOAc), diallyl sulfide (DADS)

Equipment

magnetic stirring bar, oven, Teflon-lined stainless steel autoclave

Procedure

1. 0.82 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ was dissolved uniformly in 40 mL ethylene glycol in a beaker. 2. Keep stirring the mixture with a stir bar till it turned orange without distinct precipitation. CAUTION If the reactants added into the ethylene glycol cannot be dispersed easily, ultrasonic treatment on the mixture for 10 min or even longer would help. 3. 3.6 g Sodium acetate was added into the mixture until the iron source was totally dissolved and the suspension became dark brown. 4. Appropriate amount of diallyl sulfide was added into the mixture during the mixing process respectively. CAUTION Owing to the pungent smell of DADS, the whole process was performed in the fume cupboard and the beaker dissolved with mixed solution need to be covered by plastic wrap. 5. The mixture was transferred into a 50 mL Teflon-lined stainless steel autoclave in the fume cupboard after being sufficiently stirred and sonicated. CAUTION The screws of the stainless steel autoclave should be tightened for guaranteed pressure and safety. 6. The reaction was conducted at 200°C in an oven for 12 h. 7. After the reaction was finished, the Teflon-lined stainless steel autoclave was cooled to room temperature. 8. The product in the Teflon-lined stainless steel autoclave was poured into a 50 ml centrifuge tube with the supernatant discarded. 9. The product was dissolved in ethanol and washed for several times through centrifugal separation at 5000 g for 5 min until the washings became clear. 10. The product of DADS-nFeS was obtained after drying at 60°C for 4 h and preserved at 4°C

Timing

14 h or longer (including the mixing process and the reaction time under high temperature and high pressure)

Anticipated Results

The new formed products are black \ (or brown if the drying process is much more sufficient) powder which consist of hexagonal sheet-like nanostructures and sphere nanoparticles under the electron microscope.

References

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Figures

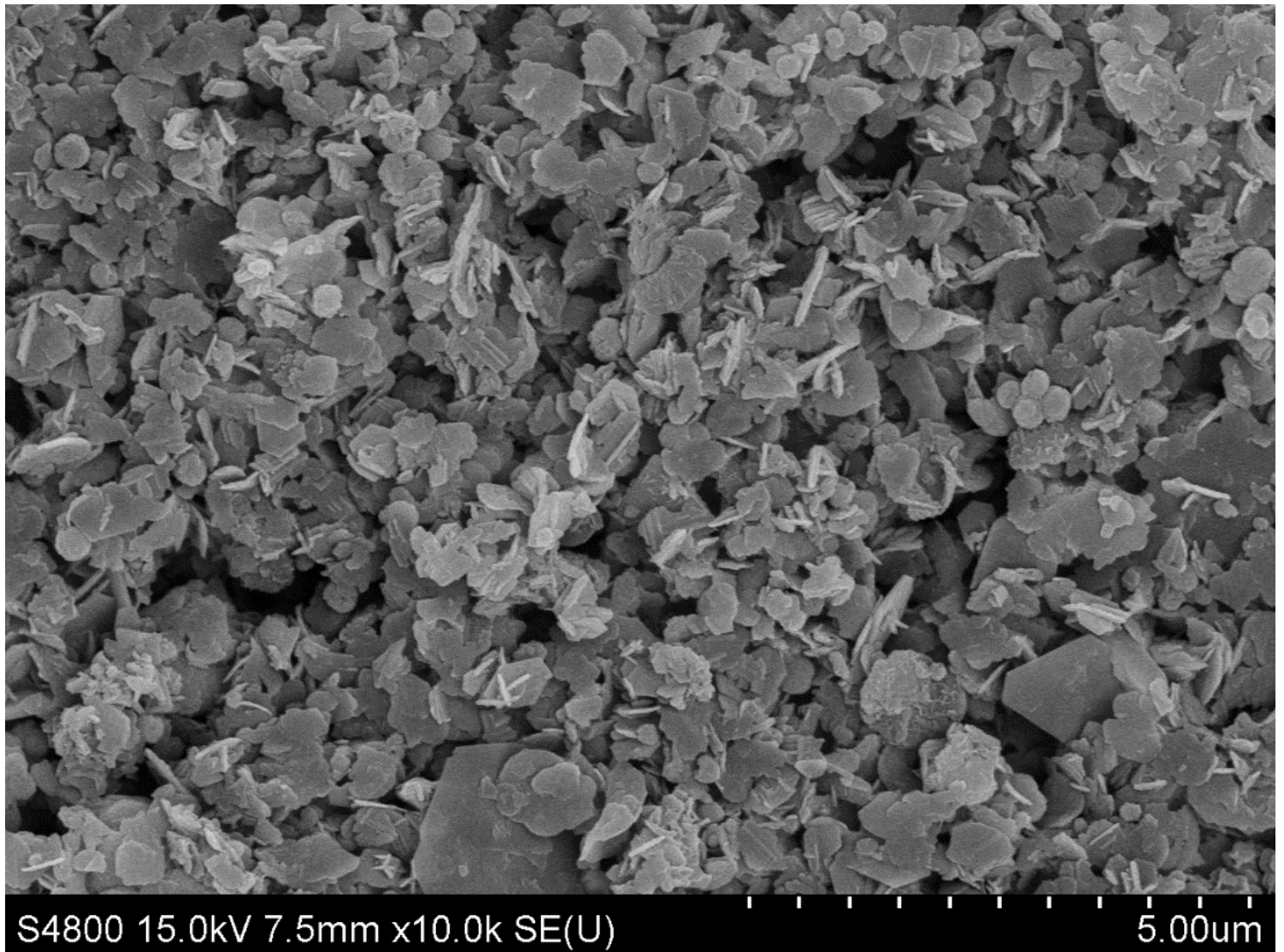


Figure 1

SEM image of DADS-nFeS SEM image of DADS~1.0~-nFeS(the amount of inputDADS in solvothermal synthesis is 1.0 g