Preparation of CuS nanoparticles by microwave irradiation

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Abstract: Copper sulfide nanoparticles were synthesized by microwave irradiation, by the decomposition of $[Cu(NH_2CSNH_2)_2]Cl_2$ complex, formed by the reaction of copper(II) acetate, thiourea (Tu) in the presence of surfactant Sodium dodecyl sulfate (SDS). X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transform infrared (FT-IR) used to characterize the product. The result nanoparticles have a diameter of about 60-70 nm.

Keywords: Microwave, Nanostructure, Copper sulfide, Characterization.

Introduction

Copper sulfide nanomaterials are widely used for different applications, such as p-type semiconductors, thermoelectric and photoelectric converters, high capacity cathode materials in lithium secondary batteries, solar radiation absorbers and nonlinear optical materials microwave shielding coatings[1-2,16-19]. Various kinds of structures of CuS have been synthesized recently. For example, copper sulfide hollow spheres by self assembly coupled with H_2S bubble templating [4,5], CuS hollow [9], wires [10], rods [11], tubes [12], platelets [13,14] flower-like structures [3,8] and nanoribbones[20]. Since 1986, microwave irradiation as a heating method has found a number of applications in chemistry. The microwave synthesis, which is generally quitefast, simple, and energy efficient, has been developed, and widely used for zeolites and ceramic materials, etc. Compared with conventional method, microwave synthesis has the advantages of very short time, small particle size, narrow particle size distribution, and high purity [15]. There are various methods for preparation of copper sulfide in nanoscale such as hydrothermal/solvothermal[8,20], template assisted growth[9], microwave irradiation[1], grinding[21] sonochemistry[6, 7] and so on. Herein, we report the synthesis of CuS nanparticles by using microwave irradiation.

Results and Discussion

There are no absorption bands in the FT-IR spectrum. This indicates that reaction was done completely. $[Cu(NH_2CSNH_2)_2]Cl_2$ complex, obtained by copper (II) and thiourea (NH_2CSNH_2) coordination, were decomposed by microwave heating. Subsequently, thiourea was hydrolyzed by H_2O to form H_2S, which further reacted with copper (II) ions to form copper sulfide compound.

$$[Cu(NH_2CSNH_2)_2]Cl_2 \longleftrightarrow Cu^{2+} + 2\ NH_2CSNH_2 + 2\ Cl^- \quad (1)$$

$$NH_2CSNH_2 + H_2O \longrightarrow NH_2CONH_2 + H_2S \quad (2)$$

$$Cu^{2+} + H_2S \longrightarrow CuS + 2H^+ \quad (3)$$

It was specified that nitrogen atoms of thiourea molecules were used to form Cu- thiourea complex caused by donating lone pair electrons on nitrogen atoms [7], to form coordination compound with the vacant d-orbital of Cu cations.

The XRD pattern of the product was indexed with the CuS structure (JCPDS: 24-0060). No other peaks for impurities were detected. This sample has covellite structure.

SEM image of CuS is presented in Fig. 1 and shows copper sulfide nanparticles in the the range of 60-70 nm in diameter.

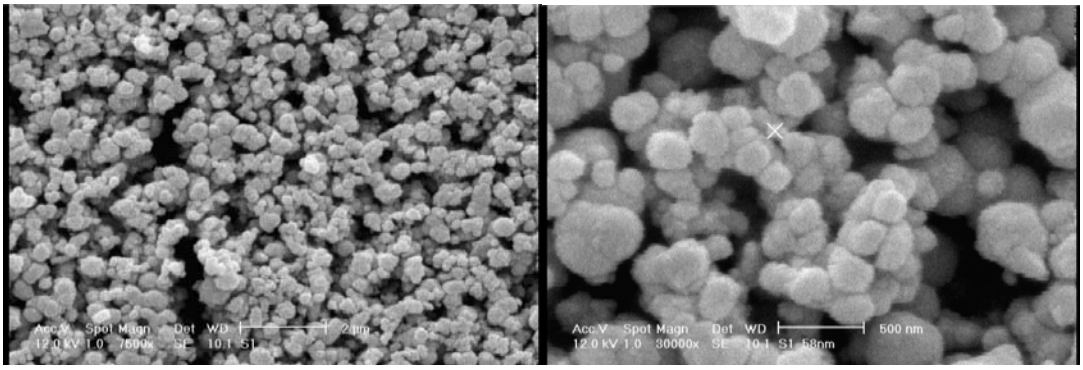

Figure 1. SEM images of CuS nanoparticles

Conclusion

CuS nanparticles were synthesized by reaction of $Cu(CH_3COO)_2.H_2O$ and thiourea in the presence of surfactant (SDS) under microwave irradiation. This method has the advantages of very short time, small particle size, narrow particle size distribution, and high purity and it is suitable for preparation of materials in nanoscale. Nanoparticles in the range of 60-70 nm in diameter were produced.

Experimental Procedure

All chemicals used in this work were analytical reagent grade from commercial market without further purification. 1.52 g (0.02 mol) Tu and 0.06 g SDS in 25 ml solution 0.5 M Cu(II) acetate were dissolved under vigorous stirring and heat to 333 K for 15 min. The result suspansion was put in a domestic microwave device for 3 min with power 450 W. After cooling to room temperature naturally, and centrifuged, the precipitate was washed with distilled water and ethanol and then dried at 80°C for 4 hours. The product was characterized by using FT-IR, XRD and SEM.

Acknowledgments

Authors are grateful to council of Iran University of Science and Technology and Iranian Nanotechnology initiative for providing financial support to undertake this work.

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