Journal of Industrial and Engineering Chemistry 20 (2014) 3141–3149

Contents lists available at ScienceDirect

Journal of Industrial and Engineering Chemistry

journal homepage: www.elsevier.com/locate/jiec

A facile and reliable route to prepare of flower shaped lead sulfide nanostructures from a new sulfur source

Maryam Shakouri-Arani [a], Masoud Salavati-Niasari [b,*]

[a] Department of Inorganic Chemistry, Faculty of Chemistry, University of Kashan, Kashan, P.O. Box 87317-51167, Iran
[b] Institute of Nano Science and Nano Technology, University of Kashan, Kashan, P.O. Box 87317-51167, Iran

ARTICLE INFO

Article history:
Received 22 October 2013
Accepted 27 November 2013
Available online 4 December 2013

Keywords:
Semiconductors
Nanostructures
Electron microscopy
Optical properties
PbS

ABSTRACT

Flower shaped PbS nanostructures were prepared by the solvothermal method using propylene glycol and a new sulfuring agent. Novelty of this work is application of a new thio Schiff-base as complexing and sulfuring agent for synthesizing of PbS nanostructures. SEM and TEM was used to examine the surface morphology of the grown samples; also the products were characterized by XRD, SEAD, UV–vis and FT-IR spectra. The results of this paper indicate that the shape and size of lead sulfide nanocrystals can be controlled systematically by setting certain reaction parameters.

1. Introduction

Nanoscale materials have attracted significant scientific and industrial interest during the last decade [1–7]. These materials are novel compounds of theoretical interest currently, but have huge potential to develop as important classes of materials for the electronics and Photonics industries in the current century. The functional materials for information storage have stimulated great interest in recent years, with metal sulfide semiconductor nanomaterials being an area of intense activity [8–10].

Semiconductor nanoparticles are materials which fall in an intermediate position between the metals and insulators, exhibiting both physical and chemical properties within an intermediate state of matter, between molecular and bulk.

Metal chalcogenide nanocrystals have been the most studied among the various nanocrystals due to their enhanced quantum confinement effects and photoemission characteristics. These nanocrystals have been applied to many different technological areas including biological labeling and diagnostics, light emitting diodes, electroluminescent devices, photovoltaic devices, lasers and single electron transistors [11–13]. Lead sulfide, among binary compound semiconductors, has an extended utilization in many fields because of small bulk band gap of 0.41 eV which makes it suitable for use in near of IR such as optical communication [13].

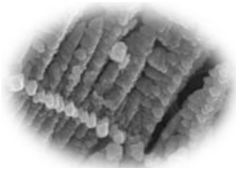
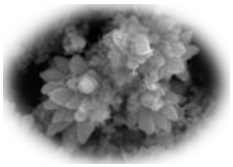
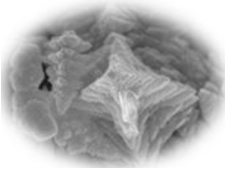
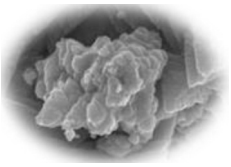
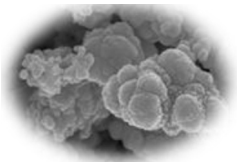
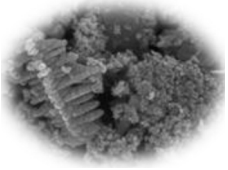
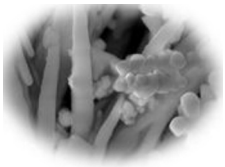
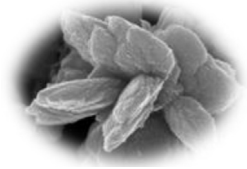
PbS can be prepared in various morphology such as nanocrystals [14], nanorods [15,16], nanotubes [15], nanocube [17], stare shapes [18] dendrites [17,19] and flower-like crystal [20] and so on [19,21–26]. Diversity of morphology is due variety of precursors and method of synthesis. Among method of synthesis, the hydrothermal route and thermal decomposition have been developed to prepare lead sulfide nanostructures due to its simplicity, low energy consuming and high potential and efficiency for large scale production [27]. Recently PbS stare-shaped nanocrystals have been prepared successfully by a hydrothermal method using lead(II) chloride and thiosemicarbazide [28].

Salavati et al. investigated about preparation of metal, metal sulfides and metal oxides in recent years. They used new inorganic precursor and tools of organometallic chemistry in this regard [29–35]. At present the fabrication and study of organometalic and organic compound for synthesis of nanoparticles is special interest [36–40]. Herein, we focus on the controlled synthesis and size evolution of high-quality flower shaped PbS nanocrystals, which are prepared by a solvothermal in the presence of thio Schiff-base ($C_{13}H_{11}NS$) and propylene glycol as solvent at 140–200 °C in different time ranges. The flower shaped PbS nanocrystals with almost uniform size and shape in high yields are synthesized by this method, without complex apparatus, reagents, and techniques. Furthermore, we discussed in detail systematic variations of the size and shape of PbS nanocrystals with the adjusting of reaction parameters, such as solvents, reaction time and temperature, and concentration of sulfuring agent.

* Corresponding author. Tel.: +98 361 591 2383; fax: +98 361 555 29 30.
E-mail address: Salavati@kashanu.ac.ir (M. Salavati-Niasari).

1226-086X/$ – see front matter © 2013 The Korean Society of Industrial and Engineering Chemistry. Published by Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.jiec.2013.11.057

Table 1
Products synthesized under different hydrothermal conditions.

Sample no.	Temperature (°C)	Reaction time (h)	Solvent type	Molar ratio Pb:S	PbS (mol)	Sulfur source (mol)	Morphologies of the products	SEM images
1	160	12	PG	1:1	0.003	0.003	Dendrite	
2	160	12	PG	1:2	0.003	0.006	Flower shaped	
3	160	12	PG	1:4	0.003	0.012	Flower shaped	
4	160	12	PG	1:6	0.003	0.018	Flower shaped	
5	140	12	PG	1:2	0.003	0.006	Microsphere	
6	180	12	PG	1:2	0.003	0.006	Dendrite	
7	200	12	PG	1:2	0.003	0.006	Rod like	
8	160	12	1-butanol	1:2	0.003	0.006	Flower shaped	
9	160	12	H_2O	1:2	0.003	0.006	Amorphous