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Synthesis and structural studies of hexadecylamine capped lead sulfide nanoparticles from dithiocarbamate complexes single source precursors

Thobani Chintso, Peter A. Ajibade *

Department of Chemistry, University of Fort Hare, Private Bag X1314, Alice 5700, South Africa

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ABSTRACT

We report the synthesis of lead sulfide (PbS) nanoparticles from Pb(II) alkyldithiocarbamate at 200 °C. The absorption spectra of the PbS nanoparticles are blueshifted and they also showed broad emission. The powder XRD of the PbS nanoparticles was indexed to the cubic rock phase with estimated crystallite sizes of 14–19 nm. The TEM images of the nanoparticles showed particles with spherical and rectangular shapes with average crystallite sizes of 5–16 nm.

1. Introduction

The research into synthesis and manipulation of nanomaterials has gained attention in recent times due to their dimension dependent properties that cannot beobtained from bulk materials [1–5]. Lead sulfide semiconductor nanoparticles have received considerable attention as semiconductor materials because of its various applications in photonic materials, electroluminescent [6] and lead ion selective sensors [7,8], due to its special small direct band gap of 0.41 eV [9–12]. PbS nanocrystals ranging from nanometer to micrometer sized cubes, stars, rods, wires and hexapods have been synthesized through thermal or solvothermal decomposition of single or multiple source precursors in the presence of different surfactants [13]. The use of single-source molecular precursors in which a metal chalcogenide bond is available has proven to be a very efficient route to high-quality nanoparticles [14,15]. Since the introduction of single source precursor as an alternative and most convenient approach to the synthesis of semiconductor nanoparticles, the compounds that have found the greatest dissemination as precursors for II–VI semiconductors are the dithiocarbamate complexes [16]. In this work, we report the synthesis and structural studies of PbS nanoparticles from Pb(II) complexes of N,N-diethyldithiocarbamate and N-hexyldithiocarbamate single source precursors.

2. Experimental

Chemicals and instrumentation: All solvents and reagents were of analytical grade purchased from Aldrich and used without further purification. The optical properties of the nanoparticles were measured on a Perkin-Elmer Lambda 25 UV–vis spectrophotometer and Perkin-Elmer LS 45 Fluorimeter. The scanning electron microscopy (SEM) images were obtained in a Joel JSM 6390 LV. Energy dispersive X-ray spectra were processed using energy dispersive X-ray analysis (EDX) attached to a Joel JSM. The transmission electron microscopy (TEM) images were obtained using a ZEISS Libra 120 electron microscope operated at 120 kV. Powder X-ray diffraction patterns were recorded on a Bruker-D8 ADVANCE powder X-ray diffractometer instrument operating at a voltage of 40 kV and a current of 30 mA with CuKα radiation.

Synthesis—Preparation of ligands: The ligands ammonium N,N-diethyldithiocarbamate (L^1) and N-hexyldithiocarbamate (L^2) were prepared by the modified procedure given in the literature [17,18].

Preparation of complexes: A solution of $Pb(NO_3)_2$ salt (1.25 mmol) was dissolved in 25 mL of water or methanol and added to 2.5 mmol of ammonium N-alkyldithiocarbamate. For PbL^1L^2, a solution of 1.25 mmol of L^1 and L^2 were poured simultaneously in a solution of $Pb(NO_3)_2$. In all the complexes, cream white precipitates formed immediately and the reaction mixture was stirred for 1 h at room temperature. The products were filtered and washed several times with water and methanol mixture (1:3) and finally with methanol. The resulting white Pb(II) dithiocarbamate complexes were dried at room temperature.

* Corresponding author. Tel.: +27 406 022 055; fax: +27 865 182 225.
E-mail address: pajibade@ufh.ac.za (P.A. Ajibade).

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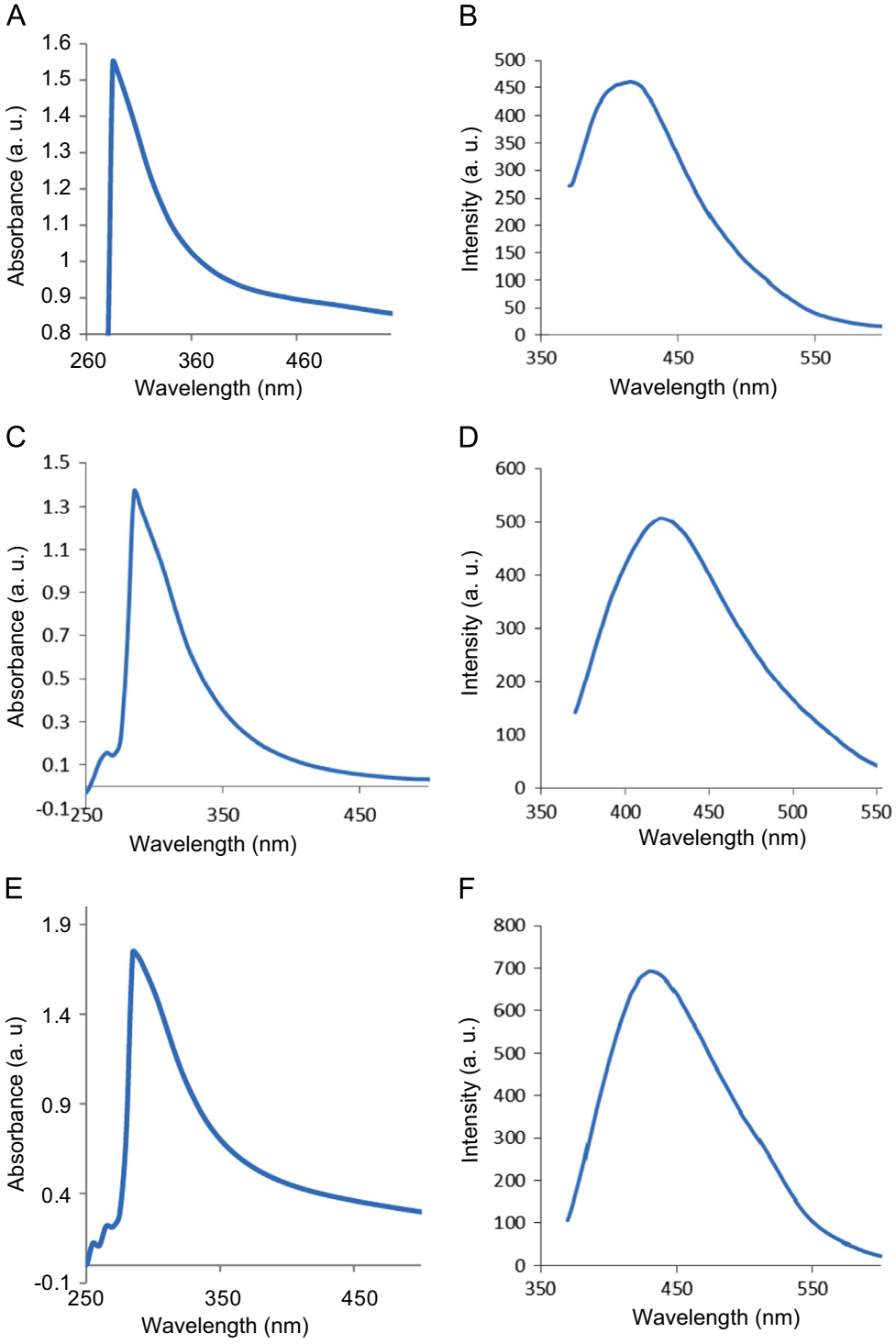

Fig. 1. UV–vis and photoluminescence of PbS1 (A, B), PbS2 (C, D), and PbS3 (E, F).

Pb(L^1)$_2$: Yield: 0.7837 g (84%). Selected IR, (cm^{-1}): ν(C–N) 1484, ν(N–H), ν(C–S) 993.

Pb(L^2)$_2$: Yield: 0.6652 g (71%). Selected IR, (cm^{-1}): ν(C–N) 1510, ν(N–H) 3229, ν(C–S) 997.

PbL^1L^2: Yield: 08260 g (88%). Selected IR, (cm^{-1}): ν(C–N) 1496, ν(N–H) 3211, ν(C–S) 998.

Synthesis of HDA-capped nanoparticles: In a typical synthesis, the lead complexes (0.10 g) were dissolved in 2 mL tri-n-octylphosphine (TOP) and injected into 1.5 g hot hexadecylamine (HDA) at 70 °C; then the temperature was increased to 200 °C. The solution was stabilized and the reaction was continued for 1 h at 200 °C. After this, the mixture was allowed to cool to 70 °C and methanol was added to