PREPARATION AND CHARACTERIZATION OF NANO CuS

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ABSTRACT

In this research paper,author has been study the preparation and characterization of copper sulfide Nano crystals from copper dithiocarbamate single molecule precursors. The precursors were thermalized in hexadecylam. The structural studies were carried out using powder X-ray diffraction (XRD). The XRD patterns indicates that the CuS nanocrystals are in hexagonal covellite crystalline phases.

Keywords: CuS; dithiocarbamate; nanoparticles; XRD

1. INTRODUCTION

The preparation and studies of the optical and structural properties of nanomaterials especially metal chalcogenides have received considerable attention in the last decades due to quantum confinement effects associated with their small crystallites sizes [1-6] that give them novel properties that make them useful in light-emitting diodes , solar cells , fuel cells, drug delivery [7-11].Especially ZnS and CdS nanoparticles have been widely studied but their toxicity limits any possible applications. As of result of the inherent toxicity of group 12 metal chalcogenides, copper sulfide nanocrystals are being explored for different applications CuS nanoparticle are also attractive because they exist in different stoichiometric compositions with varying crystalline phases.Various methods have been used to prepare the metal sulfide nanoparticles, including solvothermal synthesis, microwave], ultrasonic irradiation, and thermolysis of single-source precursors in high boiling point solvents that act as surface passivating. For the preparation of CuS nanocrystals, different synthetic techniques have also been used to produce nanoparticles with varying morphologies such as nanotubes, nanowires, and nanoplatelets, among others. Among nanocrystal synthetic methods, the single-source precursor technique produces nanocrystals with reasonable monodispersity, and studies have indicated that the sizes and shapes of the resulting nanocrystals are influenced by the precursor concentration, reaction time, and temperature. Resulted of nanocrystals' unique size-dependent physical and chemical properties, the synthesis of monodisperse nanocrystals continue to attract much research attention. In this paper, we report the use of three copper (II) dithiocarbamate complexes as efficient single-source precursors for the preparation of hexadecylamine (HDA)-capped copper sulfides nanoparticles. HDA was used as capping agent to passivate the sulface of the nanoparticles and prevent the particles from forming clump to larger particles. The structural properties of the nanoparticles were studied X-ray diffraction.

II. MATERIALS METHODS

2.1. Materials

All chemicals and reagents were used as received without further purifications. Hexadecylamine (HDA), trioctylphosphine (TOP), toluene, and methanol are analytical-grade reagents used as obtained from Sigma-Aldric. The ligands, sodium salt of *N*-phenyldithiocarbamate, *N*-ethylphenyldithiocarbamate and morpholinedithiocarbamate were prepared using literature procedures [12-14]. Powder X-ray diffraction patterns were obtained with a proportional counter using Cu Kα radiation (λ = 1.5405 Å, nickel filter).

2.2. preparation of Copper (II) Dithiocarbamate Complexes

In a typicalpreparation, a solution of $CuCl_2$ (0.625 mmol) was dissolved in 25 mL of water or methanol and added to 1.250 mmol of the sodium salt of *N*-phenyldithiocarbamate. Greenish brown 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. The resulting copper (II)-*N*-phenyl dithiocarbamate complex [Cu(phendtc)$_2$] was dried at room temperature. A similar procedure was used for the synthesis of copper (II) complexes of *N,N'*-ethylphenyldithiocarbamate [Cu(ephendtc)$_2$] and morpholinedithiocarbamate [Cu(morpdtc)$_2$].

2.3. preparation of HDA-Capped CuS Nanoparticles

The metal sulfide nanoparticles were prepared by dissolving 0.30 g of each metal complex in 5 mL of TOP and injected into 4 g of hot HDA at 180 °C. An initial decrease of about 18–35 °C in temperature was observed. The solution was stabilized at 180 °C and the reaction continued for 2 h. After completion, the reaction mixture was allowed to cool to 80 °C, and methanol was added to precipitate the nanoparticles. The solid was separated by centrifugation and washed three times with methanol. The resulting solid precipitates of HDA-capped copper sulfide nanoparticles were dispersed in toluene for further analysis. Synthesized CuS nanoparticles from copper (II) *N*-phenyldithiocarbamate complex is labeled **CuS**; from copper (II) *N*,*N*-ethyl phenyldithiocarbamate complex is labeled **CuS$_2$**, and from copper (II) morpholinedithiocarbamate complex is labeled **CuS$_3$**.

III. RESULTS AND DISCUSSION

XRD patterns for the nanocrystals prepared using different precursors are shown in figure 1. The diffraction patterns showed four broad peaks that could be indexed to the hexagonal covellite crystalline phase of CuS with characteristic (101), (102), (103), and (006), and in good agreement with the standard data for CuS (JCPDS Card No. 06-0464). The average crystallite size of the nanoparticles, as estimated using Scherrer equation are 18 nm for **CuS**, 17 nm for **CuS$_2$**, and 19 nm for **CuS$_3$**, respectively.

Another peaks that seems to be common for all XRD spectra are phosphorus, nitrogen, and oxygen due to CAP that was used for dispersing the precursor and the HDA that was used a capping agent.

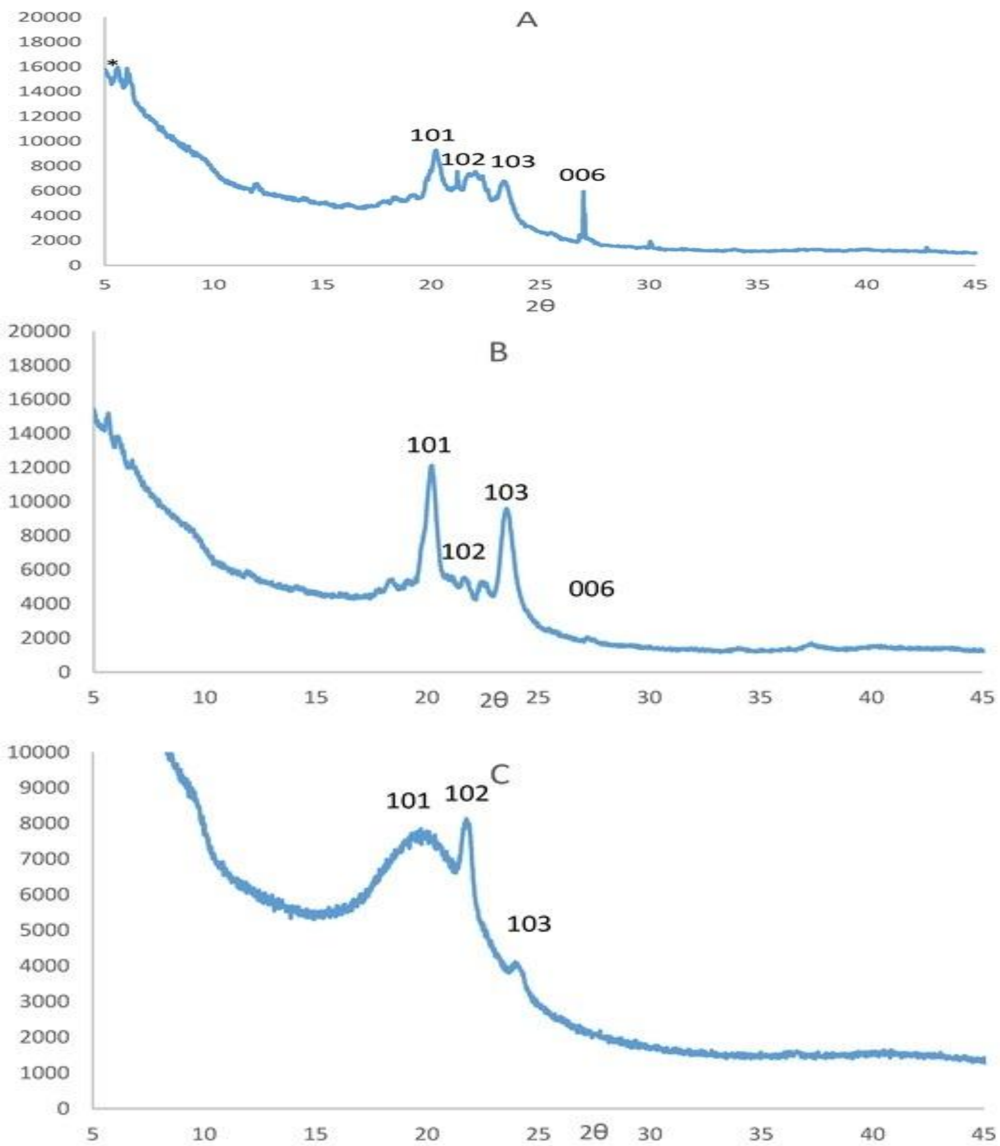

Figure 1. Powder X-ray diffraction patterns of **CuS** (**A**), **CuS_2** (**B**), and **CuS_3** (**C**) nanoparticles

IV. CONCLUSIONS

dithiocarbamate Copper (II) complexes were used as single-source precursors to prepareHDA-capped CuS nanoparticles. The X-ray diffraction patterns were indicated the hexagonal CuS nanocrystals with estimated particle sizes of 18–19 nm.

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