Synthesis and Characterization of CdTe Nanomaterial Using Single Source Molecular Precursor

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Abstract
In this paper, we have reported convenient way to access CdTe nanorods by solvothermal decomposition using single source molecular precursor cadmium (II) bis-(isopropyl telluro) propane (SSMP) and quinoline as solvent at relatively low temperature (200°C).As synthesized nanomaterials are structurally characterized by XRD, SEM and UV- Visible spectroscopy. SEM micrographs revealed formation of rod shaped structures. The average crystallite size estimated from XRD data are 28.7 nm. The average diameter of nanorods was estimated 0.9 µm from SEM micrographs. These are much larger than the average crystallite size estimated from XRD data. This is attributed to the agglomeration of nanocrystallites as quinoline is not a good capping agent. Absorption results were investigated by UV-Visible spectroscopy.

Keywords
Single Source Molecular Precursor, XRD, SEM, UV-Visible Spectra

I. Introduction

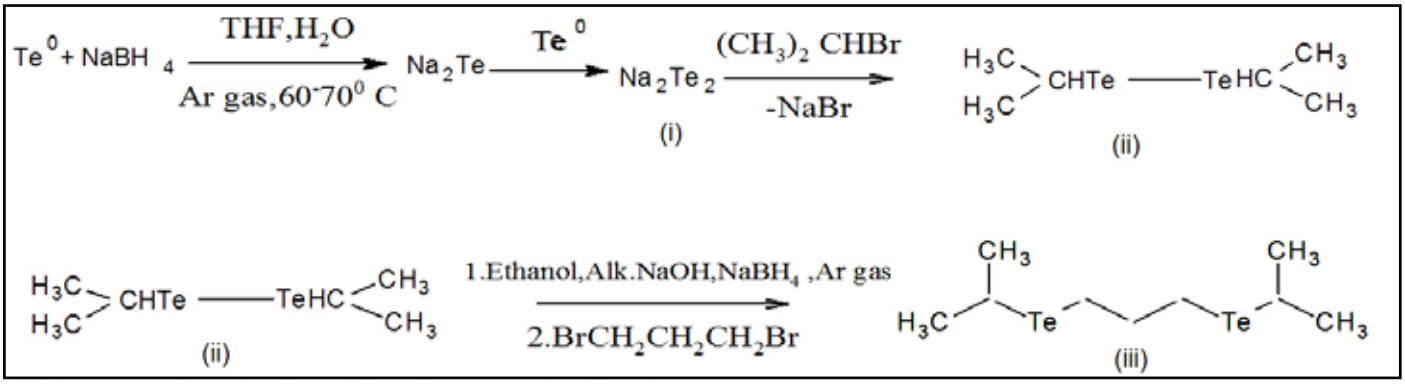
Fabrication of Semiconductor metal chalcogenide nanomaterial such as nanowires, quantum dots, nanorods, nanotubes or nanofilms have received great interest in past few years [1]. One dimensional semiconductor Nanocrystals having unique structural, optical and electrical properties have been considered as structural units for modern electronic devices, sensors, photonics materials, therefore several synthetic routes such as template directed method , vapor phase approach, vapor liquid solid growth, sol-gel technique, solvothermal synthesis, solution phase growth based on capping agents ,sonochemical, radiolytic method for synthesis of nanorods and nanowires. Each of these method have own merits and limitations [2-6]. Therefore need to develop different methodologies for synthesis of nanomaterials is inevitable in synthetic chemistry. Among the II-VI semiconducting nanomaterials, CdS and CdSe are widely synthesized, because of their high crystallites' size dependent features of photoluminescence and absorption [7] and most of the attempt has been done to synthesize CdS and CdSe nanocrystals. Because of tellurium anions susceptibility to aerial oxidation and that it also requires higher temperature for the reduction of elemental tellurium during the synthesis organotelluride ligands, very few work done on synthesis of CdTe nanomaterial. [8-9]. Multi armed CdTe nanorods were prepared by Nie et. al. [10] using myristic acid as complexing agent. Wang et. al [11] prepared CdTe nanorods with diameter of 150 nm and 1 µm length using thermal chemistry method. An attractive route for the preparation of nanostructures has been the decomposition of single source of molecular precursor, using non-aqueous high boiling co-coordinating solvent such as trioctylphosphine and its oxides [12-13]. The use of single source molecular precursor (SSMP) having metal chalcogen bond is very efficient route for generation of metal chalcogenide nanomaterial [14-15]. In this paper, we report the synthesis of CdTe nanorods using single source molecular precursors and their subsequent decomposition in CdTe nanorods.

II. Materials and Methods

All chemicals used were of analytical grade and were of highest purity, Tellurium powder (99.99% 200mesh), NaBH4 (AR), Tetrahydrofuran (THF), 1,3dibromopropane, isopropyl bromide were purchased from Merck brand, 3-Chloroethylamine hydrochloride (sigma Aldrich) Methanol purchased from Fischer scientific.

A. Synthesis of Bis (Isopropyl Telluro) Propane

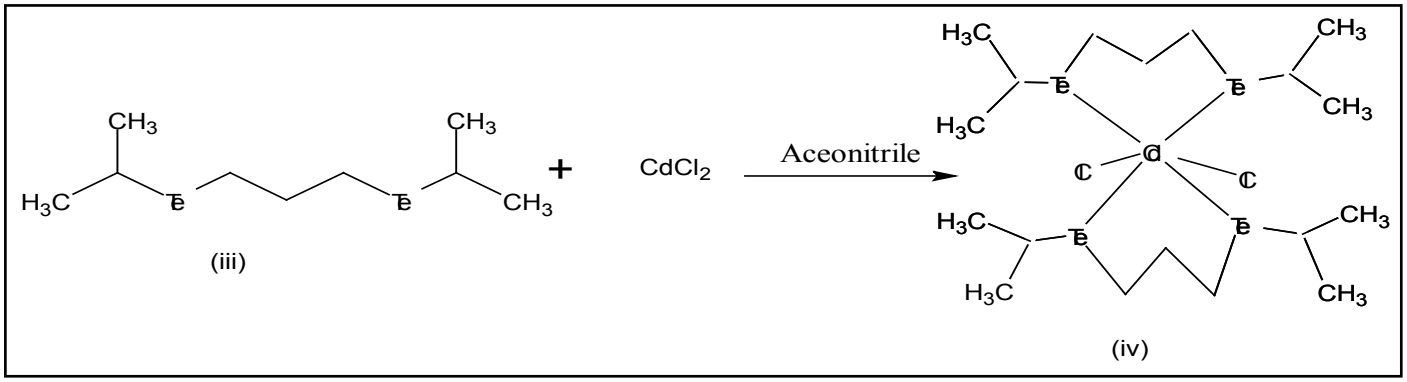
Bis (isopropyl telluro) propane were prepared by reported method [16] with slight modification in synthetic methodology.1 g of Te (0.0078 mol) was reduced by alkaline sodium borohydride ($NaBH_4$) (0.74 mol), under argon atmosphere at 60-70°C. After complete reduction 1 g of tellurium (0.0078 mol) was added to form disodium ditelluride(i). Further it was reacted to isopropyl bromide(0.7 ml) to form bis (isopropyl) ditelluride. It was extracted with Dichloromethane (DCM) from and again reduced by sodium borohydride($NaBH_4$) in ethanolic solution after complete reduction colorless solution is obtained. It was reacted with 1,3dibromopropane (0.8ml), stirred for 12 hrs. Synthesized organo- chalcogen ligand (iii) was extracted by DCM and dried over Na_2SO_4 in desecrator (Scheme 1).



Scheme1. Synthesis of bis (isopropyltelluro) propane.

B. Complexation of Bis (isopropyl telluro) propane with Cd(II)chloride

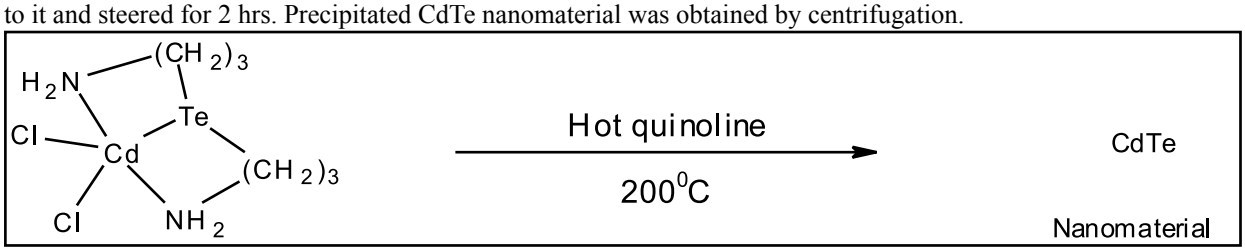
0.32g (0.083 mol) of Bis (isopropyl telluro) propane reacted with stirring suspension of $CdCl_2$ (0.152g) in dry acetonitrile. After completion of reaction, white precipitate of Cd (II) complex of bis (isopropyl telluro) propane was formed (iv), which was used as a single source molecular precursor for synthesis of CdTe nanomaterial (Scheme2).



Scheme 2. Synthesis of Cd(II) Complex of Bis (Isopropyl Telluro) Propane

C. Synthesis of CdTe Nanomaterials

Semiconducting cadmium telluride nanomaterial was synthesized by solvothermal method(Scheme3). 0.5 g of single source precursor Cd(II) complex of Bis (isopropyl telluro) propane were stirred in quinoline (20ml) for 2 hrs ,then injected in to hot quinoline(50ml) by syringe. Steered for 30 mins and cooled down above room temperature. To get precipitate of flocculent excess methanol added to it and steered for 2 hrs. Precipitated CdTe nanomaterial was obtained by centrifugation.



Scheme3. Synthesis of CdTe Nanomaterial

State color: Grey powder; yield: 33.64 %, Solubility-insoluble in aqueous or non aqueous solvent.

III. Results and discussion

X ray diffraction pattern of grey powder of CdTe nanoparticles obtained by thermolysis of single source molecular precursor [Cadmium (II) complex of bis (isopropyl telluro) propane (SSMP)] is shown in figure 1. The observed XRD pattern matches closely with JCPDS file no. 75-2083 confirming face centered cubic structure of crystallites with lattice parameter (a =6.410 Å0).

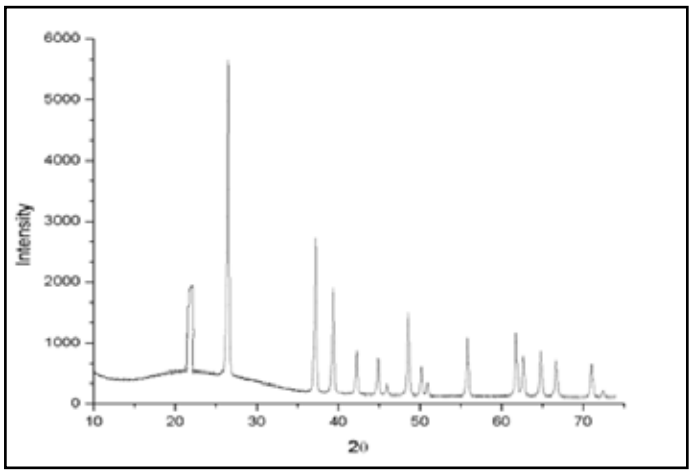


Fig. 1: Powder X-Ray Diffraction Pattern of CdTe (v) Nanoparticles Obtained by Thermolysis of Single Source Molecular Precursor Cadmium (ii) Complex of Bis(Isopropyltelluro) Propane.

Average particle size of crystallites is calculated by Debye Scherer equation

$$\tau = \frac{k\lambda}{\beta cos\theta} \quad (1)$$

Where K is the shape factor, the dimensionless shape factor has typical value 0.9; λ is the X-ray wavelength 0.154nm. β is the line broadening at half the maximum intensity (FWHM) in radians , and θ is the Bragg angle; τ is the mean size of the ordered (crystalline) domains, which may be smaller or equal to the grain size. Average sizes of crystallite calculated from Debye Scherer equation is 28.4 nm.

Micro structural Characterization is made by Scanning electron microscope (SEM).

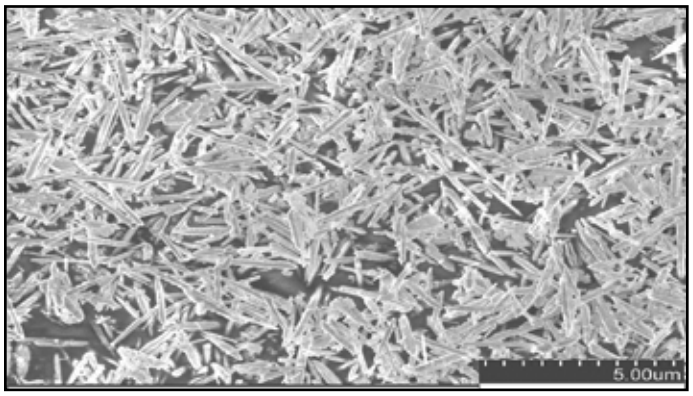


Fig. 2: SEM Images of CdTe Nanorods Obtained From Cadmium (II) Complex of Bis(Isopropyltelluro) Propane.

SEM micrograph of CdTe powder obtained from SSMP exhibits surface morphology with homogeneously distributed nanorods, diameter of rods are 900 nm and average length in submicron range.

The UV-Visible spectrum of CdTe nanomaterial in toluene shows absorption edge at 533 nm wavelength (2.32 eV) as shown in fig. 3.

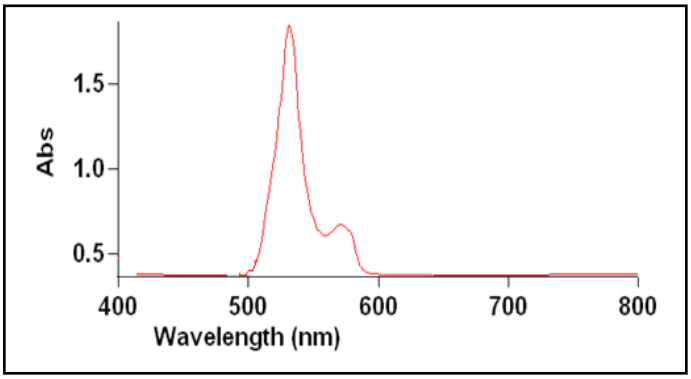


Fig. 3: UV- Visible Absorption Spectra of CdTe Nanomaterial

IV. Conclusion

Single source molecular precursor Cd (II) complex of bis(isopropyltelluro) propane were prepared and their subsequent decomposition in CdTe nanomaterials. SSMP yielded CdTe rods gave uniform nanorods with smaller diameter in nano dimensions. XRD characterization of nanomaterials matched With JCPDs file, the nanorods are oriented randomly with an average diameter of 900 nm and observed band gap 2.32ev calculated from UV- visible spectra. In addition, it was observed that each nanorod made up of number of Nanocrystals.

V. Acknowledgement

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