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Synthesis and characterization of HgTe nanorods via solvothermal route

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ABSTRACT

HgTe nanoparticle were conveniently prepared via solvothermal decomposition of novel single source molecular precursor(SSMP), i.e. mercury (ii) complex of bis(isopropyltelluro) propane. Synthesized nanomaterials were well characterized by powder XRD, SEM, TEM. Surface morphology of synthesized nanomaterials was studied by Scanning electron microscope showed rod like crystalline nanomaterials. This was also confirmed by transmission electron micrographs. Average size of nanocrystallite was calculated from Debye Scherer equation. Nanorods with average diameter 179 nm were formed by stacking one dimension growth of individual crystallite.

Keywords: Solvothermal, Single source molecular precursor, PXRD, SEM, TEM, Debye Scherer.

INTRODUCTION

Semiconducting nanomaterials have attracted much attention in last two decades, because of underlying fundamental theoretical aspects, particularly quantum confinement and a vast variety of applications. because of strong quantum confinement, shows photoluminescence with high quantum efficiency and their emission peaks initiation of absorption is highly size dependent[1].These nanomaterials are useful for enhancing the property of light emitting diodes to obtain room temperature operated single source photon. This is useful for development of optically induced laser devices [2-4]. In addition to above these materials have also applications in various fields especially in vivo biological imaging and therapeutic [5–7].

Because of toxicity of mercuried only small number of attempts were taken to synthesize mercury chalcogenide nanomaterials. These problems were overcome by preparing air stable mercury complexes. This is useful as a single source molecular precursor and thermolysis of these precursors gives rise metalchalcogenide nanomaterials [8-9]. The precursor route was extensively studied and used by O'Brien and co-workers [10-11]. They investigated cadmium dithio- and diselenocarbamate complexes as precursors for the preparation of TOPO-capped II-VI semiconductor nanoparticles. This method involves the dispersion of the precursor in TOP followed by injection into hot TOPO. The formation of the nanoparticle is consistent with the La Mer mechanism for colloids [12]. There has been extensive work reported by Revaprasadu and O'Brien on ZnS[13], ZnSe[14], CuSe[15], InSe[16], InS[16], GaS[17], and PdS[18] nanoparticles synthesized in various coordinating solvents using the respective metal dithio- and diselenocarbamates. Green et al. [19] reported the use of single-molecule precursors such as metal diorganophosphides to synthesize InP and GaP. However use of single source molecular precursor to prepare telluride nanomaterials is in little extent much work done on selenides and sulfides[20,21]. Capping agents prevents the agglomeration of nanoparticles and explore the properties of nanomaterials, various capping agents were available to passivate the bare surfaces of nanomaterials [22, 23].

Herein, we reported convenient synthesis of HgTe nanorods, using solvotrhermal decomposition of single source molecular precursor. Individual nanorods are usually composed of nano crystallites stacking along the rod growth direction [24].

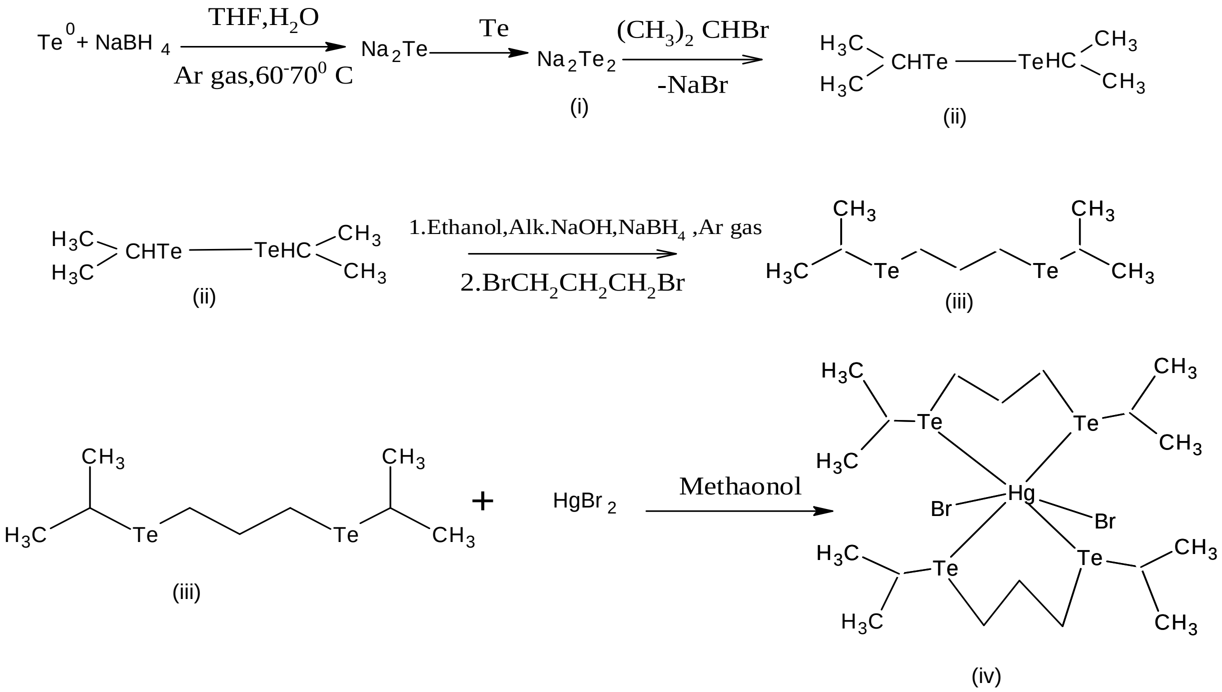
MATERIALS AND METHODS

2.1Materials

All chemicals and reagents were of analytical grade and used as such as without further purification, Tellurium powder, Sodium borohydride($NaBH_4$) were purchased from sigma Aldrich, Merck branded 1,3 dibromopropane, isopropyl bromide, Tetrahydrofuran were used. Methanol was purchased from Fischer scientific and purified by standard method before complexation. Mercury (ii) bromide was purchased from Kemie Labs.

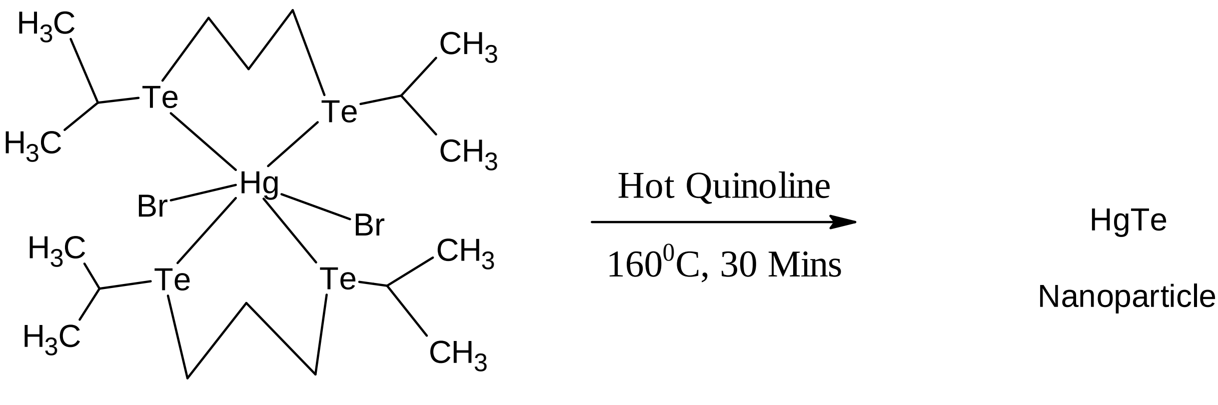
2.2Method

Mercury telluride nanomaterial was prepared by solvothermolysis of known single source molecular precursor, which was prepared by slight modification of reported method [25], quinoline was used as a capping agent, who control the particle growth and prevents agglomeration of nanoparticles. Bis(isopropyltelluro)propane(iii) was prepared, with the reaction of bis(isopropyl)ditelluride(ii) and 1,3dibromopropane in ethanol solvent. It was generated by reduction of elemental tellurium with alk.$NaBH_4$ under argon atmosphere followed by formation of Disodiumditellride(i) in situ addition of isopropyl bromide. Synthesized ligand (iii) was reacted with Hg(II)bromide in anhydrous methanol solvent. (Scheme1.)



Scheme 1. Synthesis of Hg (II) Complex of Bis(isopropyltelluro)propane

Hg(II)complex of bis(isopropyltelluro)propane (iv) was pyrolyzed in hot quinoline at 160°C for 30mins (Scheme2). Followed by addition of excess methanol, to get precipitate of flocculants blackish HgTe(v) nanomaterial. It was washed several times with methanol and hexane to remove co-ordinate solvent and impurities than kept in vacuum desiccators for dryness.



(v)

(iv)

Scheme2. Synthesis of HgTe nanoparticle

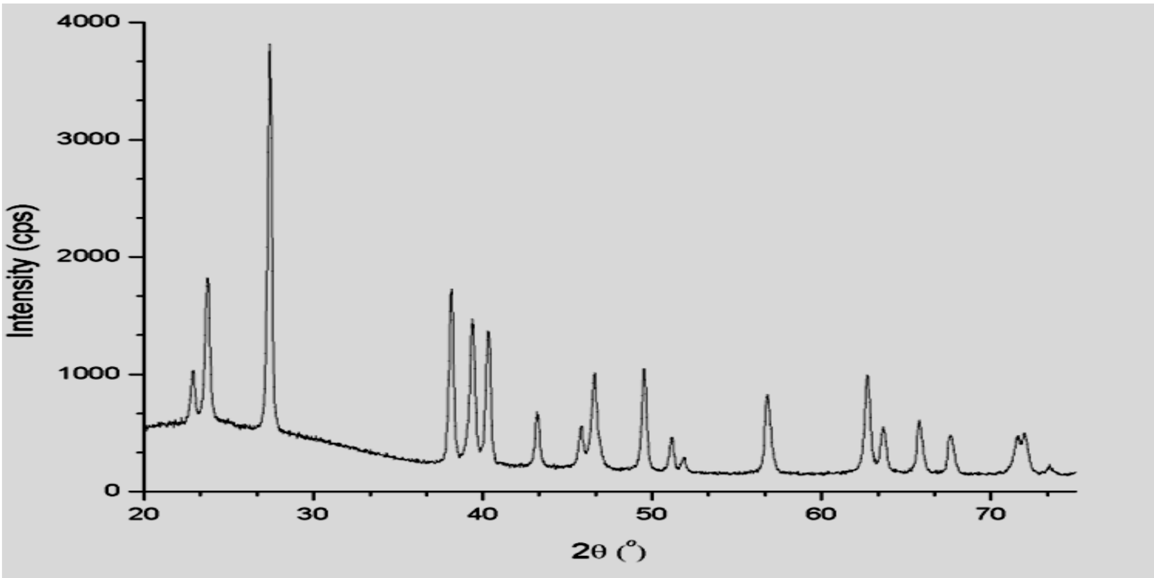


Figure1. Powder X-Ray diffraction pattern of HgTe nanomaterials

RESULTS AND DISCUSSION

The XRD measurements were carried out using Bruker D8 Advance X-ray diffract meter. The X-rays were produced using a sealed tube and the wavelength of X-ray was 0.154 nm (CuK-alpha). The X-rays were detected using a fast counting detector based on Silicon strip technology (BrukerLynx Eye detector). Average particle size was calculated by using debye scherrer equation.

$$\tau = \frac{k\lambda}{\beta cos\theta}$$

Where *K* is the shape factor, the dimensionless shape factor has typical value is abou0.9, λ is the X-ray wavelength was 0.154nm. , β is the line broadening at half the maximum intensity (FWHM) in radian, 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 size of crystallite was calculated 21.1 nm. Powder x-ray pattern (figure1) was also confirmed by matching JCPDS file (JCPDS No.88-2410). Revealed hexagonal primitive (a= 4.392 C = 10.03) Structured HgTe nanoparticles. Scanning electron photographs (figure2a) were taken JEOL JSM 5600 instrument. Surface morphology of HgTe nanoparticles indicates rod shaped crystalline nanoparticles (figure 2 a). This was further confirmed by TEM micrographs (figure2b). For TEM studies the sample powder was suspended in methanol using ultrasonification and small drop of it was put on the porous copper grid and allowed to dry. The TEM photographs were taken with Tecnai 20 G2 machine. Average diameter of rod shaped nanoparticles was found 179 nm (Figure 2 b).

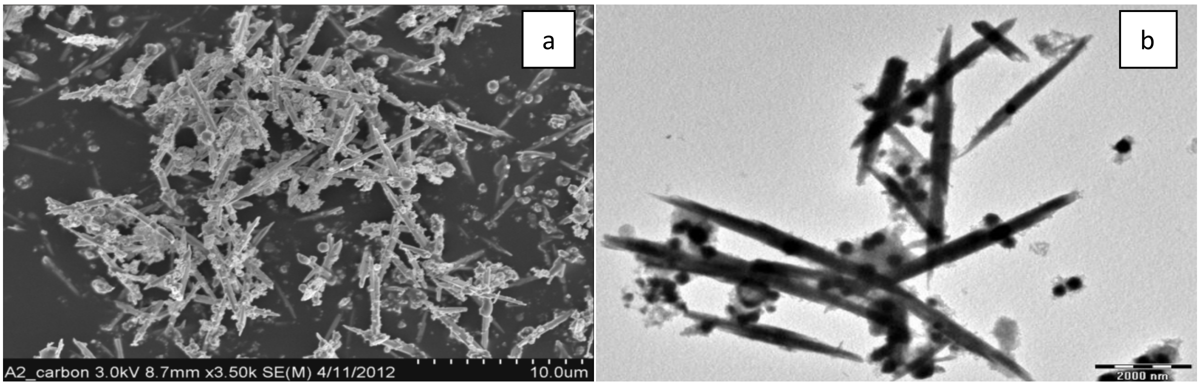


Figure. 2 (a) SEM photogtraph of HgTe **(b) TEM photograph of HgTe**

CONCLUSION

Single source molecular precursor Hg (II) Complex of Bis(isopropyltelluro) propane were prepared and their subsequent decomposition in HgTe nanomaterials. SSMP yielded HgTe nanorods with varying diameter. PXRD characterization of nanomaterials and matching with JCPDs file showed few extra peaks are obtained because of impurity, which can be removed by perfect washing of nanomaterials. Average size of crystallite was calculated 21.1 nm. SEM and TEM images revealed rod shaped growth of nanomaterial with an average diameter of 179 nm. In addition, each nanorods seems to become posed of a number of nano crystals stacking along the rod growth direction

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