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Research Paper

Synthesis and physical characterization studies of nickel ions doped CdS nanoparticles prepared by combustion method

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Abstract

Microwave-assisted combustion method was developed to synthesize nickel ions doped CdS nanocrystals (NCs). The effects of nickel ions on the physical and magnetic properties of CdS NCs were investigated by XRD, FT-IR, HR-SEM, EDX, HR-TEM, SAED, UV-Visible spectra and VSM techniques. XRD results confirmed the formation of hexagonal Ni-CdS NCs. The formation of nickel ions doped CdS phase was also confirmed by FT-IR and EDX. The formation of spherical shaped cluster of nano-crystallites was confirmed by HR-SEM and HR-TEM. VSM results of the as-synthesized Ni-CdS NCs showed ferromagnetic behavior.

Keywords: Ni-CdS NCs; Combustion method; Magnetic Properties; Optical properties.

1. Introduction

Generally, nano-structured materials are of both fundamental and industrial attention for their interesting and potentially useful structural, physical, and chemical properties [1-5]. Nano-crystalline semiconducting materials are an important in technological and industrial applications, due to their unusual physicochemical properties than that of their bulk counterparts [6-10]. Since the properties of nanomaterials mainly depend on the morphological features, a variety of CdS nanostructures have been synthesized including nanowires, nanorods, nanotubes, hollow sphere, peanut and

nanocable by various physical and chemical methods [11-15]. Even though, it is inevitable to use template and high temperature or catalyst in the synthesis process, which seemed to be of less advantageous and low economically viable processes for the preparation of CdS nanostructures [16-20].

Microwave assisted combustion method is a novel method to produce nanocrystalline materials, since microwave heating is an in situ mode of energy conversion and is fundamentally different from conventional heating processes. The efficient way of localized heating has been reported [21-25] to lead to increase in reaction rates, yield and improvement of the product formation. Thus, low temperature and fast growth synthesis of II–VI semiconductor nano-crystalline materials is a major consideration of current research [26-30]. As we know, cubic CdS is in metastable phase, while hexagonal CdS is the thermodynamically stable semiconductor. Usually, it was believed that the phase transition process of CdS from cubic to hexagonal would be happened under high temperature [31-33]. Investigations on different scale nanostructures and respective morphologies of CdS are still warranted. To the best of our knowledge, the preparation of CdS nano-crystallites by microwave combustion method has rarely been reported so far. Among various conventional synthesis approaches of nanomaterials, microwave-assisted combustion method has been widely used to improve the crystallinity of nanostructures [34, 35]. Hence in the present study, we have employed a simple, rapid and efficient microwave assisted combustion technique to synthesize hexagonal CdS nano-crystallites. The whole process takes only a few minutes to yield CdS nano-crystallites. The structure, morphology, optical and magnetic properties of the as-prepared Ni-CdS NCs were investigated by XRD, FT-IR, HR-SEM, HR-TEM, EDX, and VSM analysis.

2. Experimental part

2.1 Materials and methods

All the reagents used were of analar grade obtained from Merck, India and were used as received without further purification. The samples were prepared with the addition of nickel ions to CdS. Stoichiometric amounts of cadmium nitrate, manganese nitrate and thiourea were dissolved in deionized water and poured into a silica crucible, which was placed in a domestic microwave-oven (2.45 GHz, 800 W). Initially, the

solution boils and undergoes dehydration followed by decomposition with the evolution of gases. After the solution reaches the point of spontaneous combustion, it vaporizes the solution instantly and becomes a solid. The obtained solid powders was washed well with alcohol and dried.

2.2 Characterization

The structural characterization of nickel ions doped CdS NCs was performed using a Philips X'pert X-ray diffractometer with CuKα radiation at λ = 1.540Å. The surface functional groups were analyzed by Perkin Elmer FT-IR spectrometer. Morphological studies of nickel ions doped nano-crystallites have been performed with a Jeol JSM6360 on scanning electron microscope. The transmission electron micrographs and energy dispersive X-ray analysis were carried out by Philips - TEM (CM20). The diffuse reflectance UV-visible spectra of nickel ions doped nano-crystallites were recorded using Cary100 UV-visible spectrophotometer to estimate their band gap energy. The optical properties were recorded using Varian Cary Eclipse Fluorescence Spectrophotometer. Magnetic measurements were carried out at room temperature using a PMC MicroMag 3900 model VSM equipped with 1 T magnet.

3. Results and discussion

3.1 XRD analysis

Structural phases of nickel ions doped CdS NCs were determined by XRD. Fig.1 shows the XRD pattern of nickel ions doped CdS NCs. The XRD diffraction peaks of nickel ions doped CdS NCs reveal that it has hexagonal phase (JCPDS No. 41-1049). Additionally, no peaks of impurities were detected, indicating the successful preparation of pure CdS by the simple microwave-assisted combustion method. The sharp peaks indicate that the nickel ions doped CdS NCs possess good crystallinity [36-40].

The average crystallite size of nickel ions doped CdS NCs was calculated using Scherrer formula given in Eq. (1)

$$L = \frac{0.89\lambda}{\beta \cos\theta} \qquad \text{---- (1)}$$

where *'L'* is the crystallite size, *'λ'*, the X-ray wavelength, *'θ'*, the Bragg diffraction angle and *'β'*, the peak width at half maximum (FWHM). The average crystallite size *'L'*

calculated from the major diffraction peaks of nickel ions doped CdS NCs was found to be in the range of 15.35 nm.

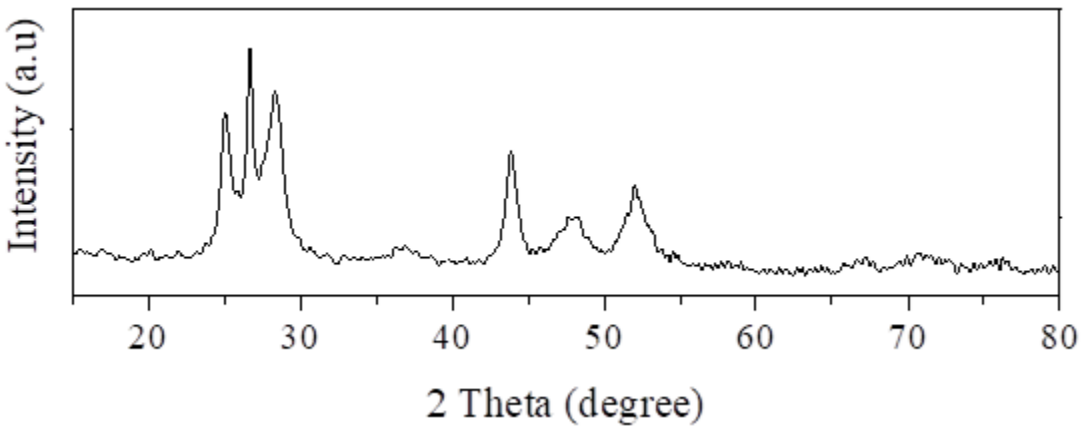

Fig. 1. XRD analysis of nickel ions doped CdS NCs.

3.2 FT-IR studies

FT-IR spectrum of nickel ions doped CdS NCs are shown in Fig. 2. The spectrum exhibit a common broad band near 3400 cm^{-1} due to the OH stretching vibrations of free and hydrogen-bonded hydroxyl groups, and a second typical absorption region at 1630 cm^{-1} is assigned to the deformative vibration of water molecules, which is most probably due to water adsorption during the compaction of the powder specimens with KBr [34, 35]. The formation of Cd-S phase is characterized by an intense and broad IR band with poor resolved shoulders at 625 and 1124 cm^{-1} [36, 37].

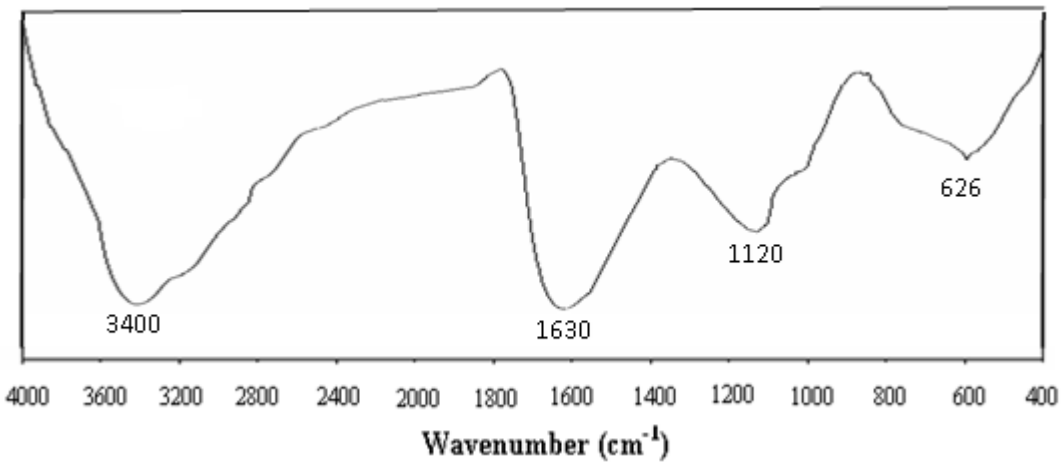

Fig. 2. FT-IR analysis of nickel ions doped CdS NCs.

3.3 SEM studies

The surface morphology of the as-prepared nickel ions doped CdS NCs were examined by HR-SEM studies and are shown in Fig. 3. The HR-SEM images show the presence of cluster of spherical shaped nanospheres. From the images it is found that the nanocrystallites are regular in shape and size. It is observed that the particle size of the as-prepared CdS crystallites is decreased with increasing the concentration of nickel ions content. Most of the nanocrystallites are found to be spherical and more evenly dispersed.

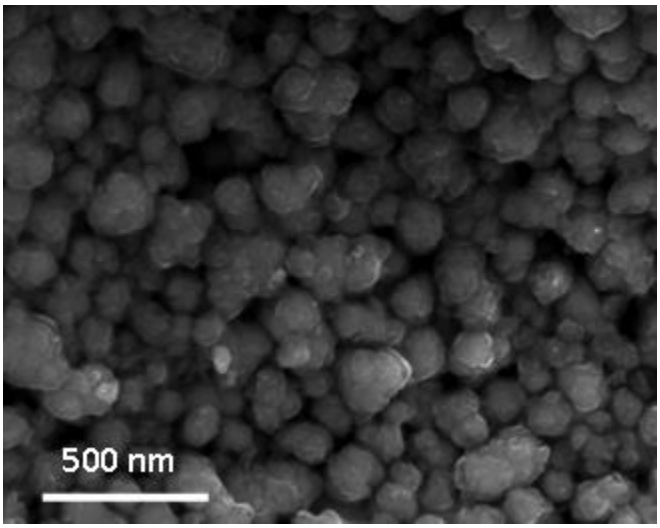

Fig. 3. SEM image of nickel ions doped CdS NCs.

3.4 TEM analysis

In order to investigate the formation of nickel ions doped CdS NCs like cluster structure, and to discern whether any secondary phase exist in the samples, a detailed structural characterization was performed by HR-TEM. These fundamental nano-particles may contain some defects, due to the microwave vibration frequency, internal stress and others. Morphology of the CdS powders obtained in H_2O with cadmium sulfate as precursor shows multiarmed nanorods which is composed by nanocrystallites while cadmium acetate as a precursor in same H_2O as a solvent using same microwave solvothermal route shows differently spherical shape morphology and are dispersed randomly. Fig. 4 display the corresponding selected area electron diffraction (SAED) pattern of an individual CdS nanosphere with bright concentric rings corresponding to the diffraction planes of the crystalline products.

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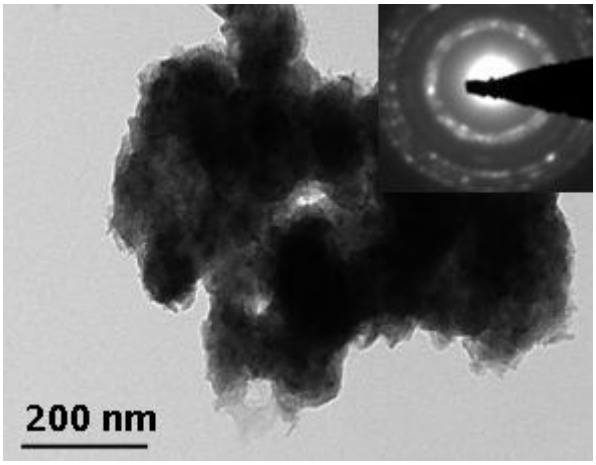

Fig. 4. TEM image of nickel ions doped CdS NCs.

3.5 EDX analysis

The chemical composition of obtained nickel ions doped CdS NCs was analyzed by EDX. As shown in Fig. 5, only Cd and S peaks are present in the spectrum. The results show that the prepared CdS nano-crystallites are elementally pure.

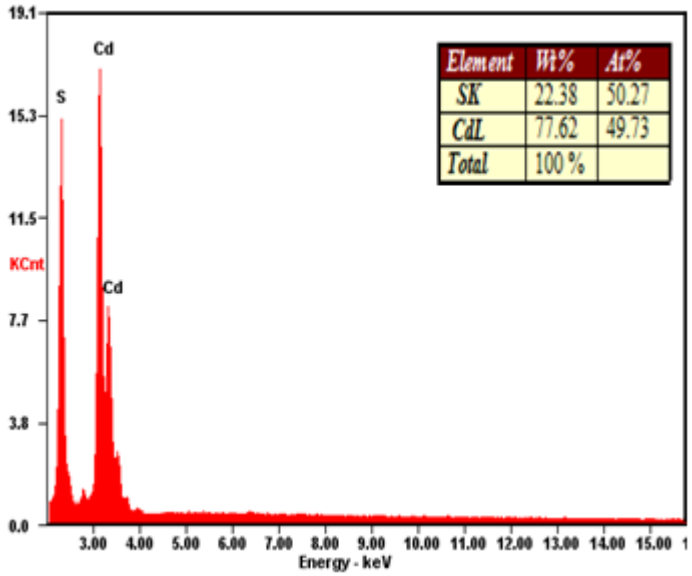

Fig. 5. EDX analysis of nickel ions doped CdS NCs.

3.6 Vibrating sample magnetometer (VSM)

Fig. 6 shows the VSM measurements of nickel ions doped CdS NCs. It denotes the effect of Ni concentrations on magnetic properties of CdS NCs. The shapes of magnetic hysteresis curves indicate that the diamagnetic signal for pure CdS nano-

crystallites and ferromagnetic for nickel ions doped CdS NCs [38-42]. The observation of ferromagnetic behavior indicates that the Ni^{2+} ions have been substituted the Cd^{2+} sites without changing the hexagonal structure of CdS NCs. The M-H curves show diamagnetic nature for CdS NCs, and ferromagnetic nature increases with increasing the Ni-doping at room temperature.

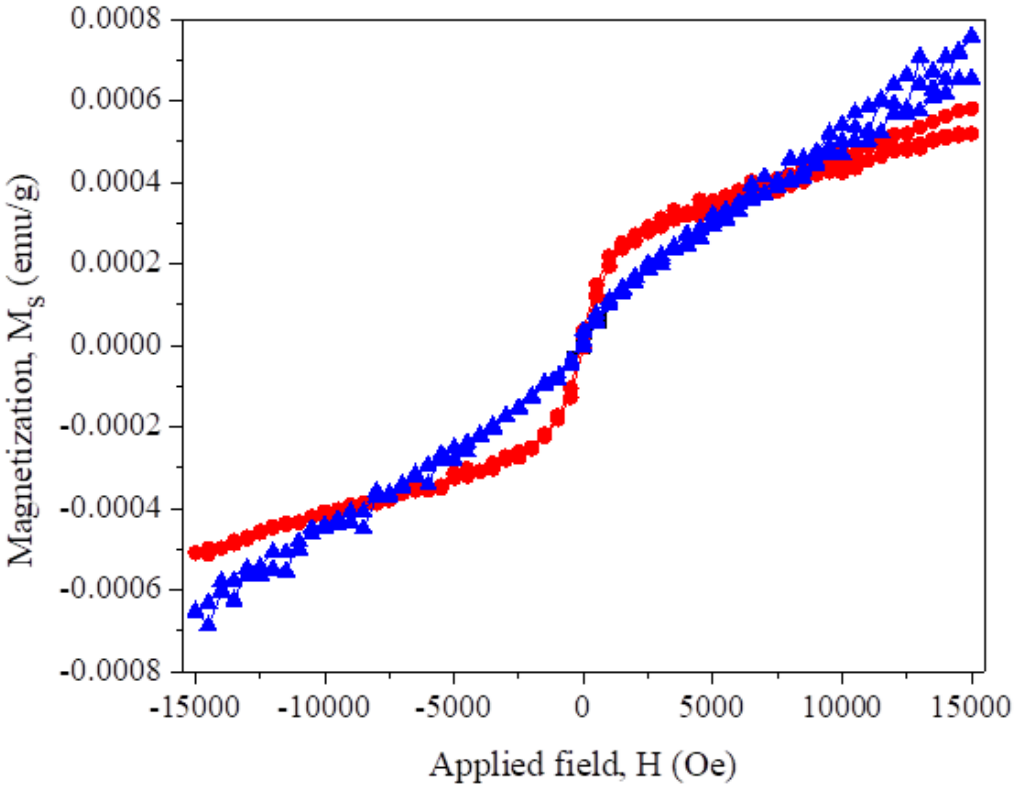

Fig. 6. VSM analysis of nickel ions doped CdS NCs.

4. Conclusions

CdS nano-crystallites have been successfully synthesized by a simple, rapid and efficient microwave-assisted combustion method. XRD and FT-IR confirmed the formation of CdS phase. It was found that the average size and morphology of CdS nano-crystallites in a sample are dependent on the microwave heating treatment and doping concentration. The formation of spherical shaped cluster of nano-crystallites is shown by SEM and TEM. The formation of pure CdS is confirmed by EDX. VSM results of the as-synthesized nickel ions doped CdS NCs showed ferromagnetic behavior. It is obvious and interesting to note that the microwave radiation is able to reduce time scales of the

reactions, and can rapidly lead to very high temperatures which have the influence of accelerating the reaction process, yield and improvement of the product formation.

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