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Microwave- assisted route for synthesis of nanosized metal sulphides

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Microwave-assisted route for the synthesis of nanomaterials has gained importance in the field of synthetic technology, because of its faster, cleaner and cost effectiveness than the other conventional and wet chemical methods for the preparation of metal sulphide nanoparticles. In the present work, synthesis of metal sulphide nanoparticles viz., PbS, CdS were carried out by microwave-assisted route without connecting any refluxing system and through the thermal decomposition of their respective metal acetate precursors employing sodium hydroxide as a fuel. The metal sulphide nanoparticles are then characterized for their size by employing X-ray diffraction(XRD) pattern. The morphology of the samples are nano globular form on the basis of scanning electron microscopy. The obtained yield percentage, grain size indicate that the nanomaterials prepared by the method adopted in the present study are important and more useful owing to the low cost of preparation.

Keywords: Metal sulphides, Nanomaterials, Microwave-assisted route, Morphology

1. Introduction

In recent years, sulphide semiconductors with nanometer size dimensions have been the focus of many researchers due to the quantum semiconductors[1]. Recently several researchers have shown interest on PbS and CdS nanoparticles because of their variety of applications such as photonic materials [2] Pb^{2+} ion selective sensors[3]. However, finding fast and energy efficient methods to produce metal sulphides is a challenge to synthetic chemists and material scientists. The microwave assisted route is yet another novel method to prepare metal chalcogenide nanoparticles and is a very rapidly developing area of research.

It is well known that micro waves containing electric and magnetic field components. The electric field applies a force on charged particles, as a result of which the charged particles start to migrate or rotate. Due to the movement of charged particles, further polarization of polar particles takes place. The concerted force applied by the electric and magnetic components of microwaves are rapidly changing in direction, which creates friction and collisions of the molecules, claimed effects of microwave irradiation include thermal and non -thermal effects[4]. Compared with conventional methods, microwave synthesis has the advantages of short reaction time, small particle size, and high purity[5]. In the present study we have prepared PbS and CdS nanoparticles by using a domestic oven without connecting any refluxing system , for the first time.

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2. EXPERIMENTAL

Analytical Grade reagent (AR) lead acetate and thiourea along with ethylene glycol were used for the preparation of PbS nanoparticles. Lead acetate and thiourea in 1:3 molecular ratio were mixed and dissolved in 100 ml ethylene glycol and. For the preparation of CdS nanoparticles, cadmium acetate and thiourea in 1:3 molar ratio were mixed and dissolved in 100 ml ethylene glycol solution then sodium hydroxide solution was added. Microwave irradiation was carried out till the solvent was evaporated completely in the two systems. The colloidal precipitate obtained was cooled and washed several times with distilled water and dried. The dried powders of the corresponding nanoparticles were characterised by X-Ray diffraction(XRD) and SEM analysis. The optical properties of the CdS nanoparticles have been studied by UV-visible absorption spectroscopy and photoluminescence to further decide its quality.

3. Results and discussion

Results of the X-Ray diffraction studies were carried out for PbS and CdS nanoparticles and a typical pattern for samples presented in Fig.1 and Fig.2 respectively .

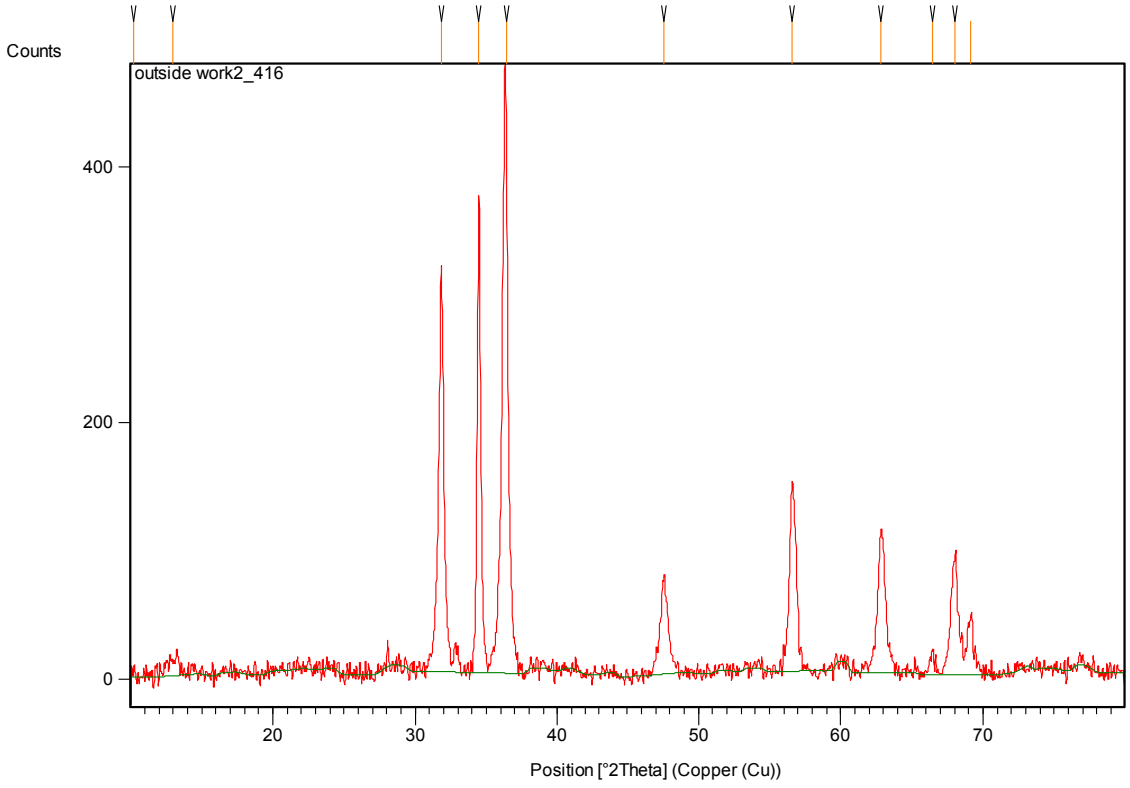

Fig.1 XRD pattern of PbS nanoparticles

The particle sizes (d) were calculated using Debye Scherrer equation.

$$d=0.96\lambda /\beta \cos\theta \quad (1)$$

Where, ƛ is the wavelength of the X-Ray used, θ is the angle of reflection and ß is the full width at half maximum.

The broadening of XRD peaks observed for the prepared samples indicate that the samples prepared in the present study are nano structured.

Fig.1 shows the XRD pattern of PbS nano crystals. The mean particle diameter is calculated to be 12 nm from the Scherrer formula.

Table.1. Interplanar distances of PbS nano particles and their corresponding crystalline lattice planes.

Interplanar distance of PbS nanoparticles (A°)	Crystalline plane
2.95	(200)
2.09	(220)
1.79	(311)
1.48	(440)
1.36	(331)

Table.1 indicates that PbS crystalline nanoparticles as-prepared with cubic structure.

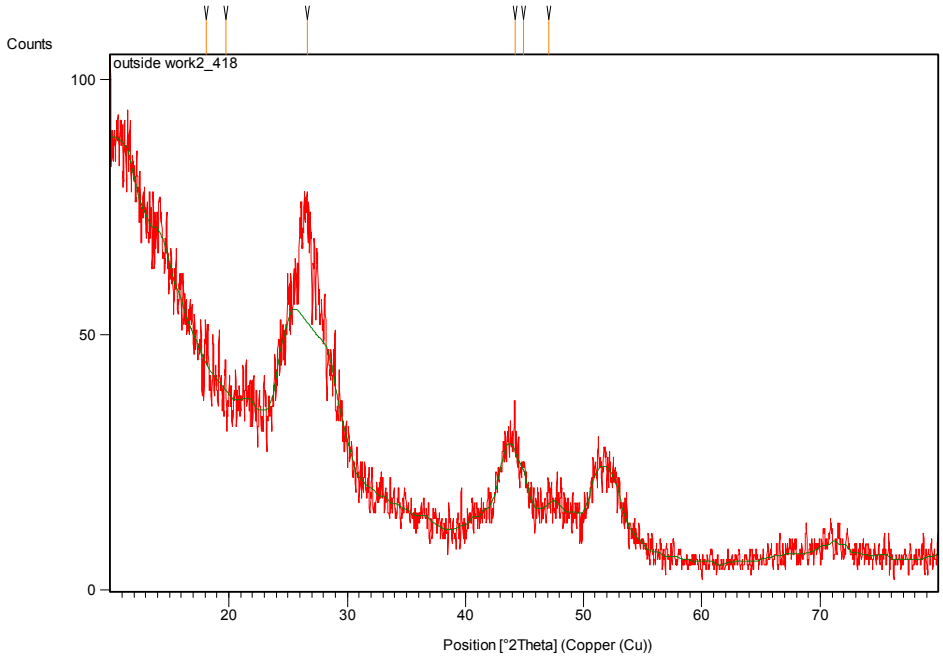

Fig.2. XRD pattern of CdS nanoparticles

From Fig. 2 the XRD patterns of CdS nanocrystals have a hexagonal structure. The presence of a diffraction peak 2θ = 28.4°, 53° correspond to the crystal planes of wurtzite CdS structure.A lack of 2θ = 31.5 is due to the lack of Zinc blend structure in the sample[6]. The mean paticle diameter is calculated to be 9.3 nm.

The particle morphology of the prepared metal sulphides were studied by using SEM images. Inset of Fig.3 (a and b) shows the particle morphology of PbS nanoparticles at low and high magnification respectively. The nanosized PbS particles form globular aggregates of microdimensions. These aggregates are almost similar throughout indicating uniform particle dimension (shape and size). However, in the higher magnification, some smooth solid blocks and nano rod may be due to very close packing of the PbS nanoparticles.

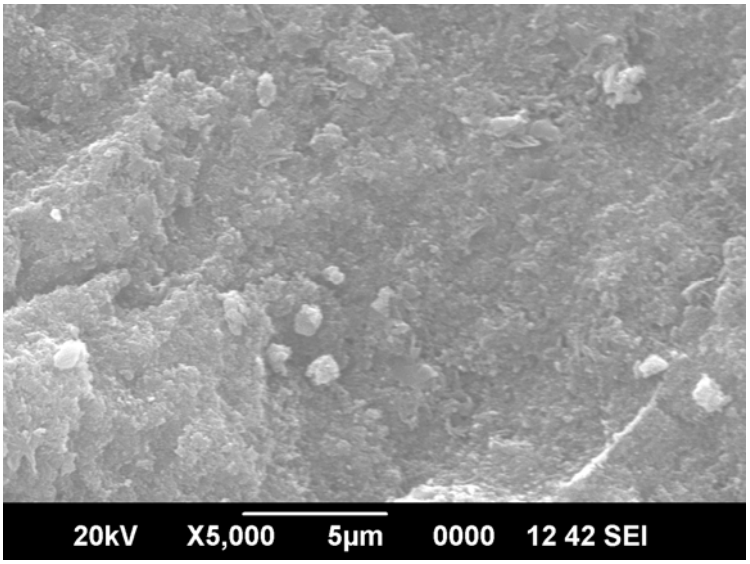

Fig.3a. SEM image of PbS nanoparticles at low magnification

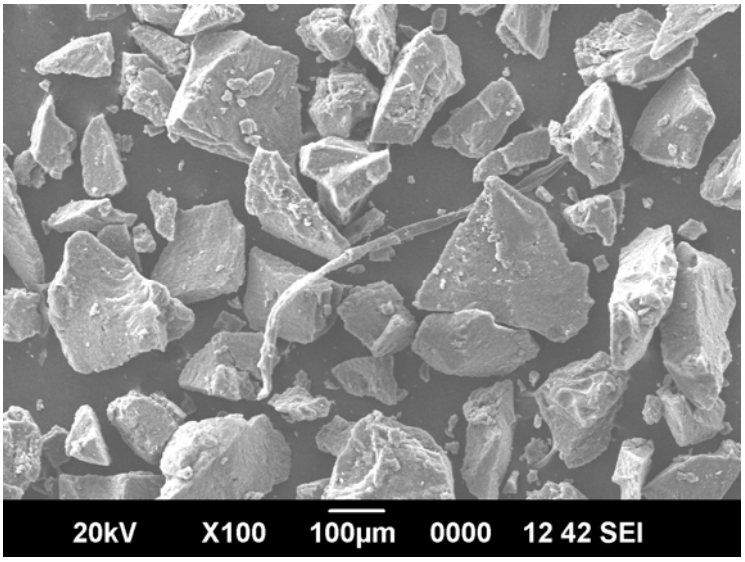

Fig.3b. SEM image of PbS nanoparticles at high magnification

Inset of Fig.4(a and b) shows the SEM images of the as synthesized CdS nanoparticles at low and high magnifications. Some particle agglomerates with globular shape are also observed. The high- magnification images clearly shows the presence of nanosized spherical particles are also observed.

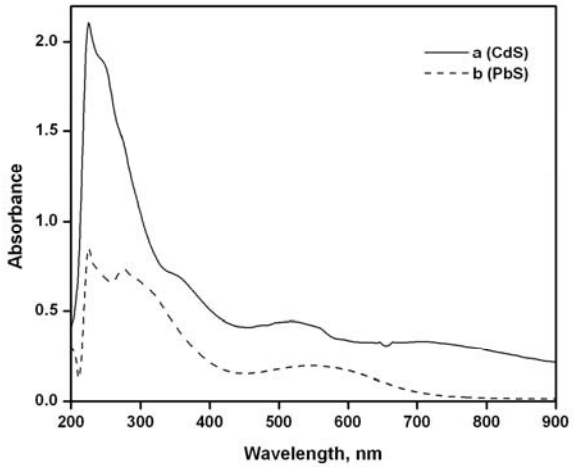

Fig.5. UV-visible absorption spectra of CdS and PbS nanoparticles.

UV-visible absorption spectra were recorded for as- synthesized CdS nanoparticles Figure. 5a displays UV-visible absorption spectra of CdS nanoparticles. The spectrum of CdS nanoparticles exhibit a broad absorption shoulder at 463 nm which was assigned to the optical transition of the first excitonic state. i.e this absorption band was due to the first optically allowed transition of CdS between the electronic state in the conduction band and hole state in the valence band [7] Figure. 5b shows the UV-visible absorption spectra of as-synthesised PbS nanoparticles. The position of the absorption edge in the spectra of the sample is strongly shifted to higher energies indicating the great influence of quantum confinement as consequence of the small size of the PbS nano particles .In addition, the spectra displays two well- defined absorption bands with maximum at 260 nm and 550 nm.

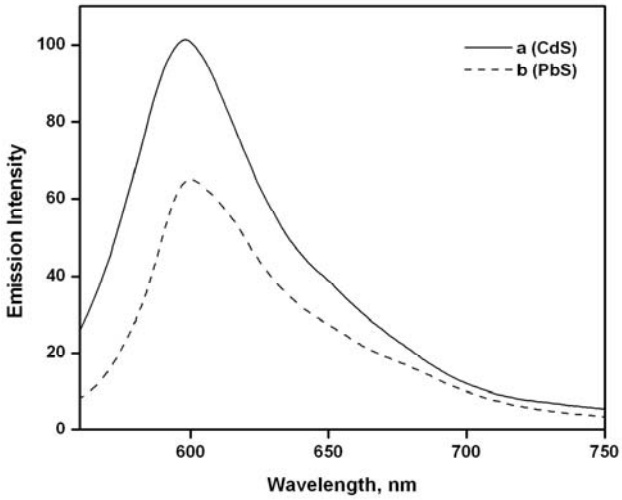

Fig.6. Photoluminescence spectra of CdS and PbS nanoparticles

The optical properties of sodium hydroxide capped metal sulphide nanoparticles were characterized by PL measurements at the room temperature . Figure 6 shows PL specra of the metal sulphide nanoparticles .The PL spectra of the CdS nanoparticles was dominated by very srong and broad emission peak spanning over a large part of visible range (490-600 nm) .The strong broad emission was attributed to the excitonic emission[8]. From the PL spectra of PbS nanoparticles, an emission band within the ultraviolet range (300-550nm).This band shifts from 360 to 380 nm.The unusual emission bands of PbS nanoparticles in the visible region are due to the presence of complexes of lead by thiol on the surface of the nanoparticles.

4. Conclusions

The metal sulphide nanoparticles were prepared by using a domestic microwave oven without any refluxing system and using sodium hydroxide as a capping agent .The yield, grain size and other characteristics observed indicate that the method followed in the present study can be considered as suitable and economical one.

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