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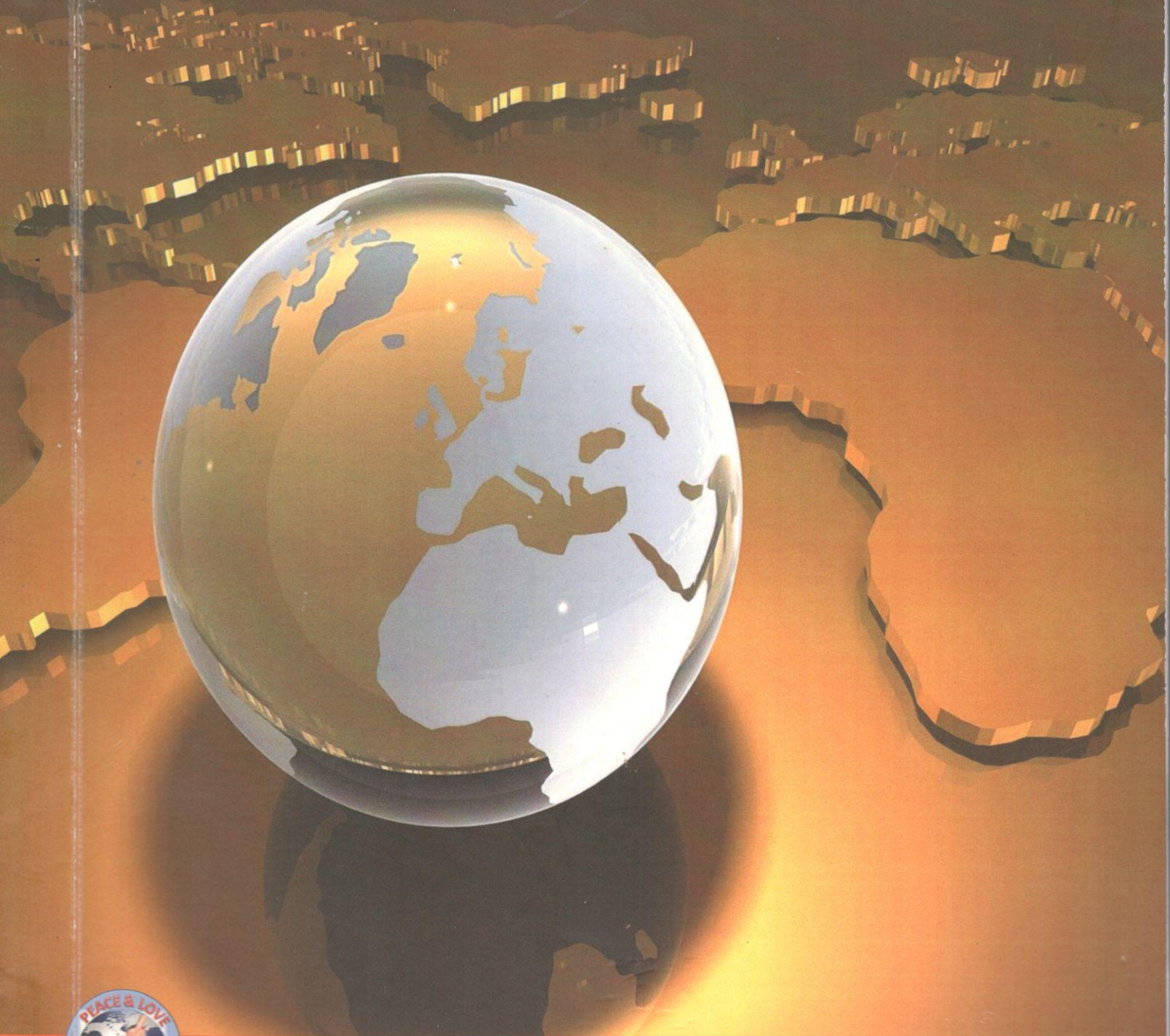

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Hydrothermal Synthesis and Characterization of Cobalt disulphide in polymer matrix

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Abstract

Nanopowders of cobalt disulphide and its polymer composite were synthesized using the simple hydrothermal technique. The as-prepared samples were characterized by X-Ray powder Diffraction, Scanning Electron Microscopy, EDAX and conductivity studies. The information about the crystal structure and particle size of the materials were obtained from XRD analysis. EDAX analysis shows the elemental composition of the synthesized nanopowders. The formation of a protective solid film like structure by the polymer over the agglomerated cobalt disulphide particles is seen from SEM analysis.

Keywords: *CoS_2, hydrothermal technique, XRD, SEM, EDAX, polymer, grain size.*

1. Introduction

Nanotechnology is the design, characterization, production and application of structures, devices and systems by controlling shape and size at the nano scale. Preparation of nanopowders in narrow size distribution is a major field of research in recent years. Control over particle size can tune band gap of semiconductor nanomaterials and catalytic efficacy of catalyzing agents [Henrick]. Lower the particle size and narrower the size distribution, the higher is the specificity of the excited states in the energy ladder, and the more specific is the photonic energy to induce transitions between these states.

Due to catalytic, electrical and magnetic properties of cobalt disulphide (CoS_2) nanoparticles, they have received recent attention. [Changzhou et. al] In preparation of metal sulphide nanoparticles, polymers are employed as steric stabilizers and as a means of controlling nanoparticles growth and spatial arrangement in films [Hong Bi et. al].

In this paper, poly vinyl pyrrolidone (PVP) has been chosen as protective colloid, with water as solvent and in the presence of some additional organic coordinating solvent, cobalt disulphide nanoparticles were successfully prepared. The structural, optical, conductivity and elemental composition features of the cobalt disulphide nanoparticles were determined with X-ray powder diffraction (XRD), SEM, EDAX and conductivity studies.

2. Materials and methods

2.1 Materials

Cobalt chloride hydrate ($CoCl_2.6H_2O$), thiourea (NH_2CSNH_2), PVP $(C_6H_9NO)_n$, Ethylenediamine, hydrazine hydrate and anhydrous alcohol of analytical reagent grade were all used.

2.2 Sample Preparation

The following is a typical experimental procedure for synthesis of sample, 0.05M Cobalt Chloride Hexahydrate was dissolved in 150 ml of water and stirred well for 5 minutes. To this solution 3g of PVP was added and then stirred for 1 hour at 90°C. Then 0.05M thiourea and 2ml of Ethylenediamine were added and stirred for half an hour. The solution was then centrifuged at the rate of 500rpm for 30 minutes. The solution was then transferred to an autoclave and heated at 160°C for 24 hours. The resulting solution was filtered and the precipitate was washed with water and ethanol [K. Byrappa et. al., Hamid Emadi et. al., Nagavarma et. al.]. The sample was collected after filtering and dried in vaccum at 60°C for 6 hours. The synthesized sample was characterized by XRD, UV-VIS NR, FESEM, EDAX.

3. Result and discussion

3.1 XRD Analysis of cobalt disulphide

The powder XRD analysis was carried out using Rich Seifert diffractometer with CuKα (λ=1.5406 Å) radiation. The intensity versus 2θ values was recorded between the ranges 10-70°. The fig.1 shows the X-ray diffraction spectrum of cobalt disulphide nanoparticle and cobalt disulphide nanoparticle in polymer matrix. It is observed that,

the peaks in the XRD patterns match well with those of the CoS_2 (hexagonal) reported in the JCPDS Powder Diffraction file no 42-0826. From JCPDS the structures of pure CoS_2 and CoS_2 in polymer matrix were confirmed and the crystal system were found to be hexagonal [Jihong Liu et. al.]. The lattice parameters of pure CoS_2 and CoS_2 in polymer matrix obtained using UNITCELL software are given in table 1. The PXRD spectra for the as prepared samples are shown in Fig. 1. The average crystallite size (d) was calculated using the Debye Schrrer formula, $\mathbf{d = k\,\lambda\,/\beta\,\cos\theta}$, Where, λ is the wavelength of copper Kα line (1.546 Å), θ is the diffraction angle, β is the full width at half maximum value.

Table 1

nanopowder	Unit cell parameters						average crystallite size (nm)
	a(nm)	b(nm)	c(nm)	α (°)	β (°)	γ (°)	
CoS_2	3.352	3.352	5.194	90	90	120	13.86
CoS_2 in polymer matrix	3.357	3.357	5.151	90	90	120	35.916

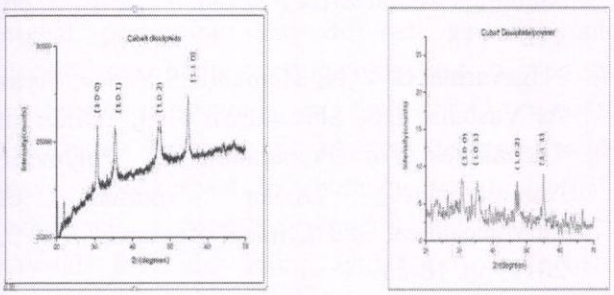

Fig. 1. XRD pattern of pure CoS_2 and CoS_2 in polymer matrix nanoparticles

3.2. SEM Analysis of cobalt disulphide

The surface structure of CoS_2 reveals the presence of agglomerates with a flake like appearance. The morphology can be controlled by further adjusting the temperature and time of the synthesis [Changzhou Yuan et. al.]. It is seen from the SEM image that when PVP is added to CoS_2, it forms a protective solid film like structure over the agglomerated cobalt disulphide particles. . The SEM images for the as prepared samples are shown in Fig. 2.

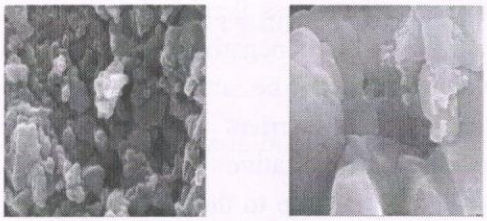

Fig. 2. SEM image of pure CoS_2 and CoS_2 in polymer matrix nanoparticles

3.3. EDAX Analysis of cobalt disulphide

The EDAX spectrum for the as prepared samples are shown in Fig. 3. From the presented spectrum one can clearly see six peaks located between 0 keV and 8 keV. The maximum is directly related to the Sulphur characteristic line. The peaks located at around1keV and between 6 keV and 8 keV show the presence of cobalt characteristic lines. The maximum located on the left part of the spectrum at 0.2 kV clearly comes from carbon. The hardly visible maximum located at 0.5 keV is connected with the oxygen characteristic line. The detected elements of Co, O, N and S in the spectra are related to CoS_2 and PVP polymer nanoparticles.

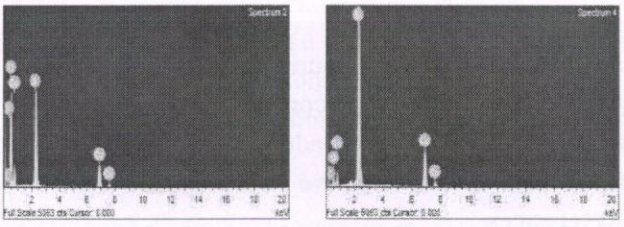

Fig. 3. EDAX spectra of pure CoS_2 and CoS_2 in polymer matrix nanoparticles

3.4. Conductivity Studies of cobalt disulphide

The photoconductivity experiment was performed at room temperature using the Keithley 6514 electrometer. Dark conductivity of the sample was studied by connecting the sample in series to a DC power supply and the Keithley meter. The DC input was increased from 1 to 5 volts in steps and the corresponding dark current was noted from the electrometer. For measuring the photocurrent, the sample was illuminated with an incandescent light bulb (40 W). The DC imput was increased in the same range as done in the previous case and the corresponding photocurrents were measured. The variation of photocurrent (I_p) and dark current (I_d) with applied field are shown in Fig. 4. Both photo and dark currents of CoS_2 increases linearly with applied field. It is observed from the plot that the

photo current is less than dark current, suggesting that CoS_2 exhibits negative photoconductivity. This phenomenon can be attributed to generation of mobile charge carriers caused by absorption of photons. The negative photoconductivity of the sample may be due to the reduction in the number of charge carriers to reveal the dielectric nature of the material. [10]

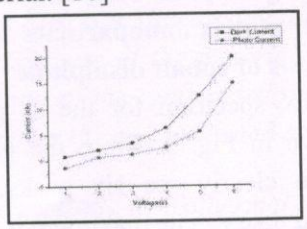

Fig. 4. Voltage Vs Current for CoS_2

Conclusions

Nanopowders of cobalt disulphide and its polymer composite were synthesized using the simple hydrothermal technique and their structural, optical, conductivity and elemental composition features were discussed. XRD studies show that the grain size increases in CoS_2 in polymer matrix. The formation of a protective solid film like structure by the polymer over the agglomerated cobalt disulphide particles is confirmed from SEM analysis. No other impurity peak is seen from EDAX spectrum which confirms the purity of the as-prepared samples. Conductivity studies suggest that CoS_2 exhibits Negative Photoconductivity.

References

1. Byrappa K. and Adschiri, T.," Hydrothermal technology for nanotechnology" Science Direct, vol. 53, 2007, pp. 117-166.
2. Changzhou Yuan, LaifaShen, Fang Zhang, Xiangjun Lu, Diankai Li and Xiaogang Zhang, " Interface-hydrothermal synthesis and electrochemical properties of CoSxnanodots/poly(sodium-4-styrene sulfonate) functionalized multi-walled carbon nanotubes nanocomposite", Journal of Colloid and Interface Science, 2010, pp 181–185.
3. Hamid Emadi, Masoud Salavati, Fatemah Davar, "Synthesis and Characterization of cobalt sulphide nanocrystals in the presence of thioglycolic acid via a Hydrothermal method", Polyhedron, vol. 31, 2012, pp. 438-442.
4. Henrick and Bruus, " Introduction to Nanotechnology." Springer, 2004.
5. Hong Bi, Xiqun Jiang, Changzheng Yang and Jianming Hong, "Synthesis of cobalt disulfide nanoparticles in polymer matrix", Science Direct, Materials Letters 57, 2003, pp. 2606–2611
6. Jihong Liu, Yan Xing, Xianchun Liu, Xiaodan Yu, ZhongminSu, "Hydrothermal synthesis and magnetic properties of nanoplate-assembled hierarchical structured Co1-xS microrods", Materials Characterization, vol. 6 7, 2 0 1 2, pp. 112 – 118.
7. Nagavarma B V N, HemantK.S.Yadav, Ayaz A, Vasudha L.S, Shivakumar H.G, "Different Techniques For Preparation Of Polymeric Nanoparticles", Asian Journal of Pharmaceutical and Clinical Research, Vol 5, 2012, pp. 16-23.