Comparative Study of Performance of CdS, CdSe thin filmCdS-PTh, CdSe-PTh nanocomposite thin films Using SEM-EDXA(Scanning Electron Microscope) and FTIR (Fourier Transform Infrared Spectroscopy)

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Abstract

A comparative study of the performance of nanomaterial CdS, CdSe and nanocomposite CdS-PTh, CdSe-PTh has been made through of SEM-EDXA analysis and FTIR. The results indicate that CdS and CdSe nanocomposite are of uniform size and monodisperse having size less than 200nm.

Keywords: Nanocomposite, Fourier transform infrared spectroscopy, Scanning electron microscopy with energy dispersive X-ray analysis.

Resumen

Se presenta un estudio comparativo de la actuación de los nano materiales CdS, CdSe y CdS nano compuestos con la hormona paratiroidea, PTH CdSe a través de SEM-EDXA análisis y FTIR. Los resultados indican que los nano compuestos CdS y CdSe son de tamaño uniforme y mono dispersos de tamaños inferiores a 200nm.

Palabras clave: Nano compuestos, espectroscopia infrarroja con transformada de Fourier, microscopía electrónica de barrido con análisis de energía dispersiva de rayos-X.

PACS: 81.05.-t, 01.40.-d **ISSN 1870-9095**

I. INTRODUCTION

Now-a-days the energy demand over the world increases more and more. Everything what happens in the world is the expression of flow of energy in one of its forms. Mainly there are two types of energy sources; these are conventional and non-conventional energy source. Conventional energy sources include fossil fuels, oil, coal, natural gas etc., which are running out and not going to long last. These resources are limited and may supply energy for about next 50 to 60 years. Therefore extensive research and development activities are being undertaken to develop efficient, reliable and cost effective technologies to use non-conventional energy sources such as solar, biomass, wind, sea, geothermal, and hydrogen etc. Due to day-by-day increase in demand for electricity and fast depleting conventional resources, men have been forced to find an alternative, by which electricity can be produced efficiently and cheaply. Again the source would be non-depleting. Since now-a-days pollution is a great concern, it has been stressed to find alternative, which is non-polluting also Solar energy is the most important source of energy. It is an inexhaustible and pollution free source. Several technologies have been developed for utilization of solar energy. Photovoltaic devices also known as solar cells convert solar energy to the electricity, which is the most convenient form of energy. It is needless to mention that K. Ravichandran *et al.* investigated microstructural and optical properties of CdS films fabricated by a low-cost, simplified spray technique using perfume atomizer for solar cell applications solar cell application [1], A. A. M. Farag *etal* studied the Electrical and photovoltaic characteristics of Al/n-CdS Schottky diode [2] and R. Mendoza-Pérez *et al.* studied CdTe solar cell degradation studies with the use of CdS as the window material [3].

For terrestrial applications, along with reliability, the cost of solar cell is important. Hence efforts are being made to produce efficient, reliable and cost effective solar cells using various types of semiconductors. Presently most successful photovoltaic device is using crystalline silicon. However the generating cost is very high hence cannot be used extensively for terrestrial applications. A method to reduce the cost of solar cell and give good energy conversion efficiency is to utilize nanocomposite semiconductors rather than bulk materials. Hence different nanocomposite thin film photovoltaic technologies are being developed as a means of substantially reducing the cost of the photovoltaic systems.

A. Cadmium selenide(CdSe) and Cadmium Sulphide (CdS)

Cadmium selenide is also a solid hexagonal or cubic crystal. It is a direct band gap n-type semiconducting material with a band gap of 1.74 eV at 300K. Its molecular weight is 191.37 g/mol where Cd is 58.74% and Se is 41.26%. Its appearance is dark red colour [4].

CdSe in the bulk form is not very interesting. The most interesting form of CdSe is nanoparticles. Much current research on CdSe has focused on nanoparticles. Researchers are concentrating on developing controlled synthesis of CdSe nanoparticles. It has useful properties for optoelectronic devices, laser diodes, nanosensing, biomedical imaging and high efficiency solar cells. The structure of CdSe is shown in figure 2. It is a point of interest to note that A. V. Kokate *et al.* studied the Structural, compositional, and optical properties of electrochemically deposited stoichiometric CdSe thin films from non-aqueous bath because of its potential application in solar energy conversion [5].

Cadmium sulphide is a solid hexagonal or cubic crystal. It is a direct band gap n-type semiconducting material with a band gap of 2.42 eV at 300K. Its molecular weight is 144.47 g/mol where Cd is 77.81% and S is 22.19%. Its appearance is yellowish-orange colour. It has useful properties for optoelectronics, being used in both photosensitive and photovoltaic devices [6]. The structure of CdS is shown in figure 1.In this interest it is mention that Jun Zhang *et al.* studied the Multicolored luminescent CdS nanocrystals and observed the efficient blue, green, orange and red luminescence from CdS nanocrystals [7].

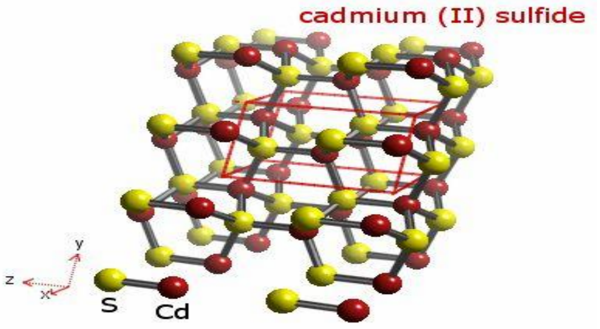


FIGURE 1. Cadmium sulphide.

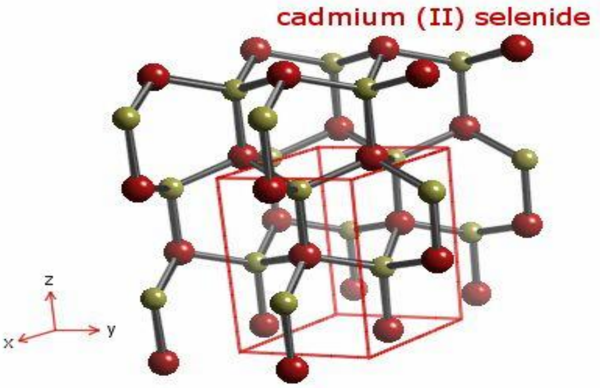


FIGURE 2. Cadmium selenide.

B. Polythiophenes (PTh)

Polythiophenes are one of the most valuable types of conducting polymers that may be easily modified to afford a variety of useful electrical and physical properties such as solubility, electrical conductivity, mobility and others.

Polythiophenes are the polymerization of thiophenes (a sulphur heterocycle) *i.e.,* a linear chain of thiophene monomers. This is shown in the figure-1.4. It possesses lower band gap and better electronic properties. It may give rise also some very useful properties such as increased ionization potential and stability [8, 9].

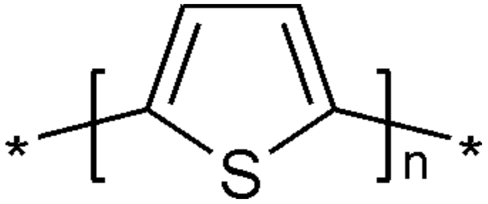


FIGURE 3. The monomer repeat unit of unsubstituted polythiophene.

Polythiophenes usually do not possess metallic type conductivity even in a doped state. Therefore, they are much more commonly used as organic semiconductors. Many of them possess also good luminescent, nonlinear-optical, and other useful optoelectronic properties. The simplest polythiophene shown in figure 1.4 is insoluble and infusible, therefore inconvenient for semiconductor processing. Hence, it is not widely used even though it is very cheap.

II. EXPERIMENTAL WORK

This experimental work has been done in the Center for Materials For Electronics Technology (C-MET), Pune.

Synthesis of Cadmium sulphide (CdS), Cadmium selenide (CdSe) and polythiophene (PTh) were tried out by different methods either by changing reactants, concentration of the reactants and by changing reaction conditions.

A. Synthesis of Nanocrystalline CdS

A.1 Microemulsion Mediated Sonochemical Route

In a typical procedure, 50 ml of distilled water, 50 ml of n-hexane and 1 ml of cetyl trimethyl ammonium bromide (CTAB a surfactant) were added together in order to prepare water-in-oil microemulsion solution, which was divided into two 50 ml aliquots. Then 0.1 M solutions of cadmium acetate and thioacetamide were prepared from these two aliquots under stirred condition. These solutions were added together and irradiated at 20 KHz frequency for 1 hour in an ultrasonic bath (Elma Ultrasonic Bath, Germany). The products of yellowish precipitates were collected. These products were centrifuged (Research Centrifuge, R 23, Remi Instruments Ltd., Mumbai), filtered and washed thoroughly with distilled water followed by absolute ethanol. The final products were dried in a vacuum oven at 70° C for 5 hours. Then it was ground mechanically using a mortar and pestle to get fine powders of nanocrystalline CdS.

A.2 Sonochemical Route

In this procedure, 95 ml of distilled water and 5 ml of CTAB (surfactant) were added together and divided into two 50 ml aliquots. Then 0.01 M solutions of cadmium nitrate and ethylene diamine tetraacetic acid (EDTA) were prepared in one aliquot and 0.01 M solutions of sodium thoisulphate were prepared in another aliquot. These solutions were added together slowly (drop by drop) under stirring and then irradiated at 20 KHz frequency for 1 hour in an ultrasonic bath. The products of greenish-yellow precipitates were collected. These products were filtered and washed thoroughly with distilled water followed by ethanol and then dried in a vacuum oven at 70° C for 5 hours and ground these to get powder form of nanocrystalline CdS [10].

A.2 Synthesis of nanocrystalline CdSe:(Wet Chemical Synthesis)

In this procedure, first 0.4 M Se metal powders were dissolved in 0.4 M sodium sulphite solution. The solution was carried out at 60° C under stirring for 4-5 hours. Then the undissolved particles were filtered out after the solution cooled to the room temperature. That solution was sodium selenosulphate used as a selenium source. Second, in 35 ml of distilled water and 15 ml of isopropanol, 0.1 M of cadmium sulphate solution was prepared. The prepared sodium selenosulphate solution was added drop wise to the stirred cadmium sulphate solution. After one hour, the products of dichromate coloured precipitates were collected and then washed with distilled water followed by absolute ethanol. The products were dried in a vacuum oven at 70°C for 6 hours. Finally the powders of nanocrystalline CdSe were collected [11, 12].

A.3 Synthesis of Polythiophene (PTh): (Oxidative Polymerization of Thiophene)

Here 0.1 M of thiophene was dissolved in 25 ml of acetonitrile or methanol (non-aqueous media) with 1ml of CTAB as surfactant. Then 0.3 M of lithium perchlorate was added as a supporting electrolyte to increase the bath conductivity and also added 0.1 M of ferric chloride as an oxidant to the above stirred solution. Again stirred the above mixture for 2 hours and kept several hours for polymerization. Then the products of black coloured precipitates were collected. These products were washed thoroughly with acetonitrile to remove any residual ferric chloride. These products were dried in a vacuum oven at 70°C for 10 hours to get powder form of polythiophene [9, 13].

A.4 Synthesis of CdS/PTh Nanocomposite

In a typical procedure, 50 ml of methanol, 45 ml of distilled water and 5 ml of CTAB stock solution were added together and then divided into two equal volumes. 0.1 M of thiophene, 0.3 M of lithium perchlorate and 0.1 M of ferric chloride solutions were prepared in one part. Again 0.1 M of cadmium nitrate, 0.1 M of EDTA and 0.1 M of sodium thiosulphate solutions were prepared in another part. Then these solutions were mixed slowly under stirring for 1 hour and irradiated 20 KHz frequency for 1 hour in an ultrasonic bath. The products of brown coloured precipitates were collected and then washed thoroughly with ethanol. The final products were dried in a vacuum oven at 70°C for 10 hours and ground to get CdS/polythiophene nanocomposite powders.

A.5 Synthesis of CdSe / PTh Nanocomposite

Here first Se metal powders were dissolved in 0.4 M sodium sulphite solution at 60°C under stirring for 4-5 hours and then the undissolved particles were filtered out after the solution cooled to the room temperature. That solution was sodium selenosulphate used as a selenium ion source. Second, 50 ml of methanol, 45 ml of distilled water and 5 ml of CTAB stock solution were added together and then divided into two equal volumes. 0.1 M of thiophene, 0.3 M of lithium perchlorate and 0.1 M of ferric chloride solutions were prepared in one part. Again 0.1 M of cadmium nitrate and the above prepared sodium selenosulphate solutions were prepared in another part. These solutions were mixed slowly under stirring for 1 hour and irradiated 20 KHz frequency for 1 hour in an ultrasonic bath. The products of blackish-brown coloured precipitates were collected and then washed thoroughly with absolute ethanol. The final products were dried in a vacuum oven at 70°C for 10 hours. Finally CdSe/polythiophene nanocomposite powders were collected [14].

A.6 Deposition of Nanocomposite of Thin Films

Different techniques such as physical vapor deposition, chemical vapor deposition, chemical bath deposition [16, 17], electrodeposition, spray pyrolysis, spin coating and sputtering have been adopted by different groups for the deposition of thin films. We have tried to deposit the nanocomposite thin films on different substrates like Ti, SS, Sn, ITO, glass and Mo by the following methods.

- DIP COATING
- ELECTRODEPOSITION, etc.

A.6.1 Dip Coating

Before depositing, the above substrates were cleaned by using soap solution, distilled water, acetone and final washing with trichloroethylene (TCE), and then dried under IR lamp.

The prepared CdS/polythiophene or CdSe/polythiophene powders were dispersed well in equal volume of chloroform and pyridine mixture. After mixed properly, a cleaned glass substrate was dipped in it for 10 minutes, a uniformed film appeared on it and then the film was dried in IR lamp.

A.6.2 Electrodeposition

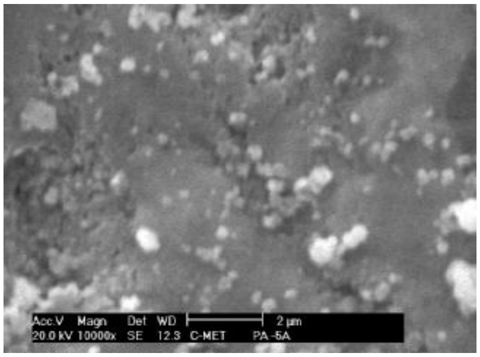
The nanocomposite thin films were galvanostatically deposited on copper substrates. Here 25 ml of n-hexane, 25 ml of distilled water and 1 ml of CTAB were added together and divided into two equal parts. For CdS/polythiophene film, 0.1 M of thiophene and 0.3 M of lithium perchlorate were added in one of the above solution and 0.1 M of cadmium nitrate, 0.1 M of EDTA and 0.1 M of sodium thiosulphate were added in another solution under stirring. But for CdSe/polythiophene film, 0.1 M of sodium selenite was added instead of sodium thiosulphate. These solutions were added together slowly and applied 1.2-1.5 mA current for electrodeposition. After 75 minutes a brown coloured uniform film deposited on the copper substrate. Here copper substrate was connected to the cathode and graphite electrode was used as anode [18].

III. RESULTS AND DISCUSSION

The surface morphology, particle size and elemental composition were determined by using Scanning Electron Microscope with Energy Dispersive X-Ray Analysis (SEM-EDXA, Model XL 30, Philips, Holland). The infrared spectra were recorded in the wave number range 400-4000 cm^{-1} by Fourier Transform Infrared Spectrometer (FTIR, Perkin-Elmer, Model Spectrum 2000, USA) by making the pellet of samples in KBr matrix.

A. Scanning Electron Microscopy with Energy Dispersive X-Ray Analysis (SEM-EDXA)

The scanning electron microscopy with energy dispersive X-ray analysis (SEM-EDXA) for different prepared materials are explained here. Figure 4 (a) shows the SEM image of nanocrystalline CdS synthesized by microemulsion mediated sonochemical route. This SEM image clearly shows the morphology of monodisperse CdS nanoparticles with an average particle size of 50-100 nm. Figure 4 (c) is the EDX spectrum of nanocrystalline CdS. Table 4 (e) gives the element composition of CdS, which matched with standard reported value.Figure 4 (b) is the SEM image of CdS/polythiophene nanocomposite. This SEM image clearly indicates that the CdS nanoparticles were immersed and well separated in polythiophene matrix. Figure 4 (d) is the EDX spectrum of CdS/polythiophene nanocomposite. Table 4 (f) gives the element composition of CdS/polythiophene nanocomposite. This shows that the prepared nanocomposites are stoichiometric in composition.



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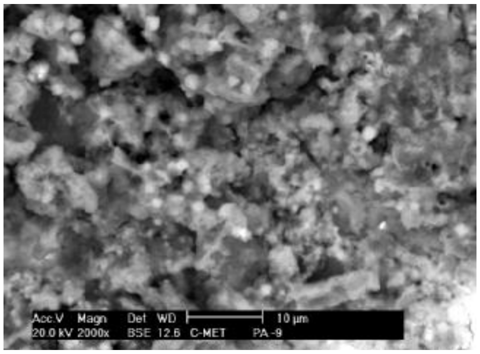

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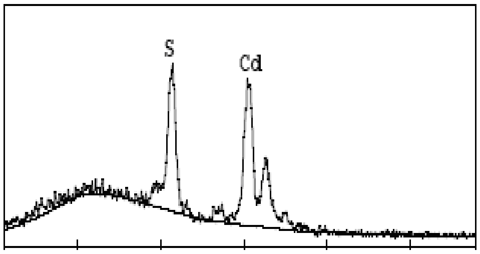

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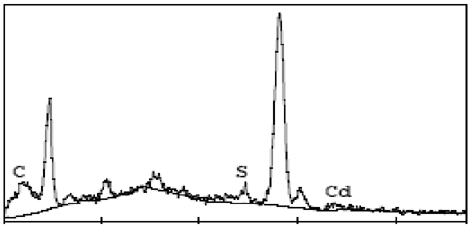

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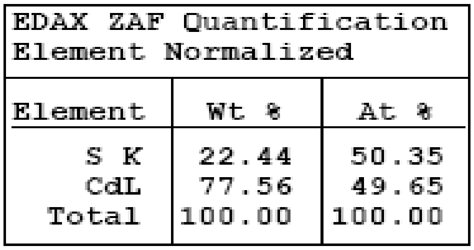
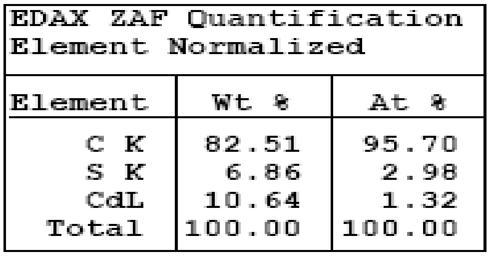


EDAX ZAF Quantification
Element Normalized

Element	Wt %	At %
S K	22.44	50.35
CdL	77.56	49.65
Total	100.00	100.00

(e)

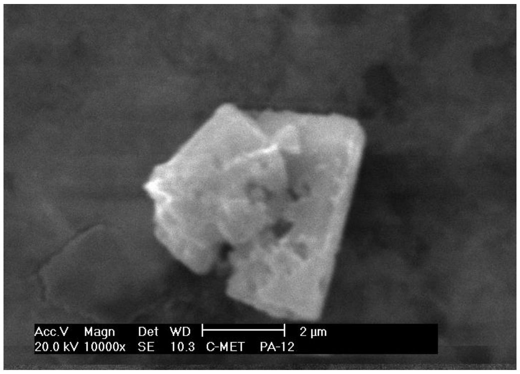


EDAX ZAF Quantification Element Normalized		
Element	Wt %	At %
C K	82.51	95.70
S K	6.86	2.98
CdL	10.64	1.32
Total	100.00	100.00

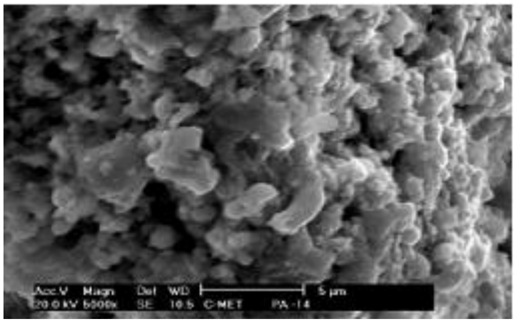
(f)

FIGURE 4. SEM image of (a) nano CdS and (b) CdS/PTh nanocomposite. EDX spectra of (c) nano CdS and (d) CdS/PTh nanocomposite EDX element composition table of (e) CdS and (f) CdS/PTh.

Figure 5 (a) gives the SEM image of nanocrystalline CdSe synthesized by wet chemical route. This SEM image clearly shows the morphology of monodisperse CdSe nanoparticles with an average particle size of 100 nm. Figure 5 (c) is the EDX spectrum of nanocrystalline CdSe. Table 5 (e) gives the element composition of CdSe. The presence of some impurities is due to the use of sodium sulphite solution in the synthesis process.



(a)



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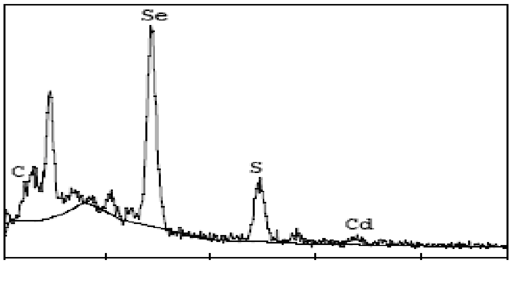

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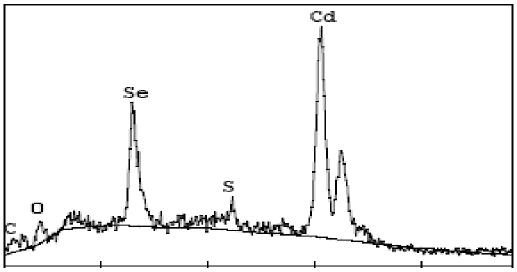

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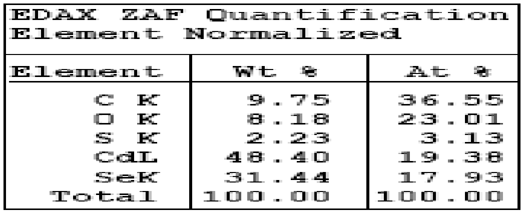
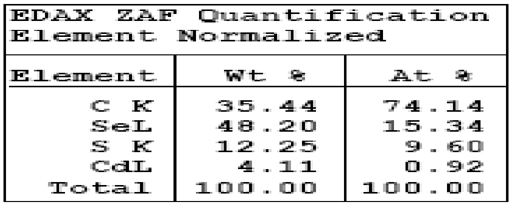


EDAX ZAF Quantification Element Normalized		
Element	Wt %	At %
C K	9.75	36.55
O K	8.18	23.01
S K	2.23	3.13
CdL	48.40	19.38
SeK	31.44	17.93
Total	100.00	100.00

(e)



EDAX ZAF Quantification Element Normalized		
Element	Wt %	At %
C K	35.44	74.14
SeL	48.20	15.34
S K	12.25	9.60
CdL	4.11	0.92
Total	100.00	100.00

(f)

FIGURE 5. SEM image of (a) nano CdSe and (b) CdSe/PTh nanocomposite. EDX spectra of (c) nano CdSe and (d) CdSe/PTh nanocomposite. EDX element composition table of (e) CdSe and (f) CdSe/PTh.

Figure 5 (b) gives the SEM image of CdSe/polythiophene nanocomposite. This SEM image clearly indicates that the CdS nanoparticles were immersed in the polythiophene matrix. Figure 5 (d) is the EDX spectrum of CdSe/polythiophene nanocomposite. Table 5 (f) gives the element composition of CdSe/polythiophene nanocomposite.

B. Fourier Transform Infrared Spectroscopy

The Fourier transform infrared spectroscopy (FTIR) for different prepared materials are discussed here. Fig.6 shows the FTIR spectrum in the frequency range (400–4000 cm^{-1}) of nanocrystalline CdS synthesized by microemulsion mediated sonochemical route. There is a band at 635.54 cm^{-1} is due to the stretching frequency of Cd-S bond. Strong interaction of water with CdS is reflected by peaks at 3429.20 cm^{-1} and 1629.17 cm^{-1} due to O–H stretching and O–H bending modes, respectively.

Fig. 7 shows the FTIR spectrum in the frequency range (400–4000 cm^{-1}) of CdS/polythiophene nanocomposite. The major peaks at 630.29 cm^{-1}, 1089.82 cm^{-1}, 1374.73 cm^{-1}, 1634.48 cm^{-1} and 3380.52 cm^{-1} are due to the presence of different bonds in both CdS and polythiophene in the nanocomposite.

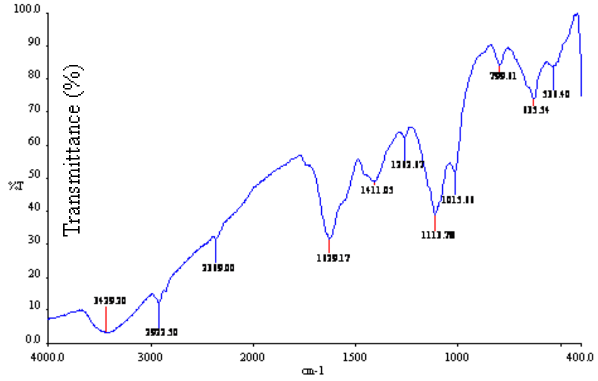


FIGURE 6. FTIR spectrum of nanocrystalline CdS.

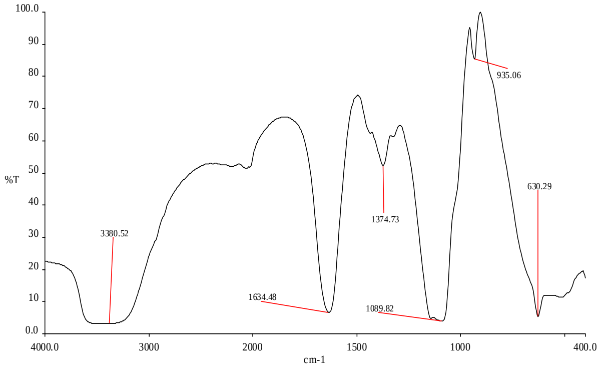


FIGURE 7. FTIR spectrum of CdS/Polythiophene nanocomposite.

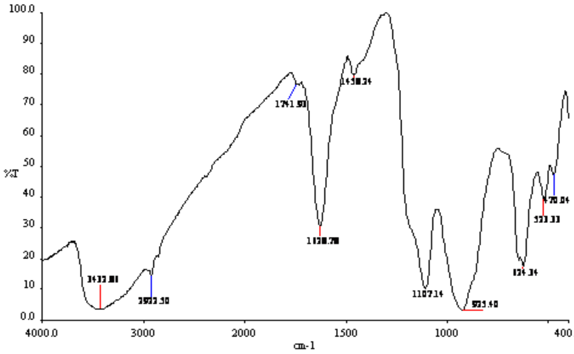


FIGURE 8. FTIR spectrum of nanocrystalline CdSe.

Fig. 8 is the FTIR spectrum in the frequency range (400–4000 cm^{-1}) of nanocrystalline CdSe synthesized by wet chemical synthesis. There is a band at 624.34 cm^{-1} is due to the stretching frequency of Cd-Se bond. Here also the peaks at 3432.86 cm^{-1} and 1628.78 cm^{-1} due to O–H stretching and O–H bending modes, respectively.

Fig. 9 gives the FTIR spectrum in the frequency range (400–4000 cm^{-1}) of CdSe/polythiophene nanocomposite. The peaks at 626.33 cm^{-1}, 1124.27 cm^{-1}, 1635.94 cm^{-1}, 2355.68 cm^{-1} and 3434.87 cm^{-1} are due to the existence of different bonds in both CdSe and polythiophene in the nanocomposite.

C. Electrical Characterization of Thin Films

The direct method of measuring dc electrical resistivity, ρ_{dc} or sheet resistance, R_s is to prepare a rectangular sample of film and measure its resistance. It is necessary to ensure that the two gold-coated contacts at the two ends of the rectangle are ohmic or as close to ohmic as possible, so that the bulk resistivity, not the contact resistance, is measured. R_s of squarely cut samples is independent of the size of square and depends only on resistivity and thickness, d, of films, *i.e.*, $R_s = \rho_{dc}/d$. Van der Pauw resistivity measurements were performed on squarely cut samples of 10 mm each side length provided with four symmetrically spaced gold coated contacts. For the electrical measurements, very low resistance silver wires and gold-coated contacts were used.

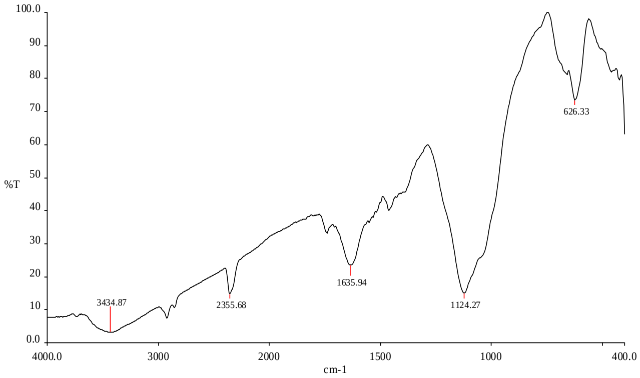


FIGURE 9. FTIR spectrum of CdSe/Polythiophene nanocomposite.

TABLE I. Electrical characterization of thin films.

Thin Films	Condition	Electrical resistivity
Electrodeposited polythiophene / CdS thin film	As prepared	Surface resistance 29 KΩ
		Bulk resistance 11 KΩ
	Heated 200°C for half an hour.	Surface resistance 4.6 KΩ
		Bulk resistance 1.5 KΩ
Electrodeposited polythiophene / CdSe thin film	As prepared	Surface resistance 34 KΩ
		Bulk resistance 15 KΩ
	Heated 200°C for half an hour.	Surface resistance 5.3 KΩ
		Bulk resistance 1.8 KΩ

The resistivity measurements were carried out for the as-deposited and annealed hybrid thin films. The above table I shows the electrical resistivity of as prepared and thermal annealed CdS/polythiophene and CdSe/polythiophene nanocomposite hybrid thin films. The decrease in resistivity after thermal annealing may be due to the increase in effective grain size and also to the formation of S and Se vacancies [19].

IV. CONCLUSION

In conclusion, it is summarized that CdS/polythiophene and CdSe/polythiophene nanocomposites are successfully synthesized in room temperature and pressure. The inorganic CdS and CdSe nanocrystals are of uniform size and nearly monodispersion, the SEM images of nanocrystals and nanocomposites are shows that, they are ideal materials having particle sizes < 200 nm. The EDX spectra showing that the powders are stoichiometric compositions. In FTIR, it confirms that the successful polymerization of thiophene and the formation of CdS/polythiophene and CdSe/polythiophene nanocomposites due to the existence of peaks of both inorganic and organic materials. Again we observed that, the electrical conductivity of polythiophene CdS and CdSe as-prepared films have less conductivity than that in thermally annealed state. This is because of the increase in grain sizes of the materials and becomes more crystalline. Further investigations on photovoltaic studies on hybrid thin films are in progress.

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