

# Nanocomposite Characterization through Solid Dispersion: Structural Analysis of Polystyrene - Cadmium Sulphide Systems using XRD, FTIR, and Raman Spectroscopy

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**Abstract:** Solid dispersion technique is used to make nanocomposites complex of polystyrene (PS) and cadmium sulphide (CdS) at various weight percentage of CdS. Several methodologies are used to investigate the material, including X-ray Diffraction, FTIR, and Raman Spectra. X-ray scattering was used to characterize the structure. The outcome shows that it is partially crystallization and amorphous structure exists for the nanoparticles. The sample's FTIR spectroscopy and Raman spectroscopy are captured and briefly described.

**Keywords:** FTIR spectroscopy, CdS nanocomposites (NPs), Raman spectroscopy, XRD diffraction

## 1. Introduction

The composites referred to as nanocomposites contain at least one phase with nano metre - scale dimensions (1 nm = 10<sup>-9</sup> m). [1]Polymers are filled by mixing them with solid particles like silica or minerals. In addition to promoting mechanical properties like fatigue resistance, wear resistance, and hardness, these filler materials can improve processing, lower costs, control density, improve optical and thermal properties, reduce thermal expansion, improve flame retardancy, and improve magnetic and electrical characteristics. [2]The food business, which includes agriculture, food processing, food packaging, and dietary supplements, appears to gain from nanotechnology to the greatest extent. [3]

Polystyrene (PS) is one of the most fascinating polymers because of its distinctive properties. When heated to its temperature of glass transition (95°C) for casting or extrusion, a thermoplastic substance called polystyrene flows and then solidifies after cooling. Polystyrene is stable (glassy) at room temperature. Solid Polystyrene is a rigid, colourless substance with limited elasticity. Polystyrenesol could be carefully poured into moulds. Polystyrene can be produced in a range of colours or in clear forms. [4][5]

Styrene monomers are polymerized to create PS, an aromatic polymer. Ethylene and benzene are combined to produce styrene (vinylbenzene). Massive production of PS is conducted by catalytic dehydrogenation of ethylbenzene which leads to in the polymer industry usually do not exceed 20 ppm, which is much below the determined value of chronic toxicity. [6] When polymers such as polystyrene goes under sonication it dispersed nanotubes in the matrix of polymer. [7] Although cadmium is toxic for humans and exist in very low amount in environment, [8] but cadmium sulphide CdS has low toxicity and it is fluorescent material with high optical, photocatalytic and electric properties. [9][10][11]

## 2. Experimental

Dissolving PS resin produced thin films of unfilled polystyrene and CdS - filled polystyrene. As a result, we dissolve CdS in dichloromethane for 1 hour with vigorous stirring. When we add dichloromethane to polystyrene, it becomes like jelly, and we mix it at 300 rpm, and CdS is mixed in dichloromethane using a solarimeter. The UV rays present in the sonicator decompose the CdS, but there are some sonicator instructions that we must stop for 5 minutes every 10 minutes stirring and that the sonicator nob is not too high or touch the beaker. The magnetic stirrer, on the other hand, will continue to rotate the PS and dichloromethane for another 30 - 35 minutes. After that, combine both and stir for 40 minutes with the help of a solarimeter. In PS, CdS remained in the dispersed phase. The percentages of CdS in PS are 1, 2, 3, 4, and 5. Make a PS/CdS solution before casting the mixture.

The CdS powder was uniformly mixed with PS solution after being stirred. To ensure uniform thickness, the glass plate was kept over a pool of mercury for perfect levelling. To keep dust at bay, the entire assembly was placed in a sealed chamber. At room temperature, the solution was allowed to evaporate. The film on the glass substrate was kept at 313 K for 12 hours for out gassing and another 12 hours at room temperature for solvent removal. It was then cut into small pieces of the desired size and washed with ethyl alcohol to remove surface impurities. The thickness of all the samples was found to be in the order of 50 micro metre and was taken nearly constant throughout the entire work.



(a)



(b)

**Figure 1:** (a) Dispersion of CdS by using sonicator (b) CdS and PS nanocomposite film

**Calculation**

By using formula,

For CdS

$$\rho = m/v$$

$$m = \rho \times v$$

$$m = \pi r^2 \times \text{thickness}$$

$$m = 4.82 \times 3.14 \times 4.5 \times 4.5 \times 50 \times 10^{-4}$$

$$m = 1.5323 \text{ gm}$$

CdS	MASS (gm)	PS	Mass (gm)
1%	0.0153	99%	1.51323
2%	0.0306	98%	1.4994
3%	0.0459	97%	1.484
4%	0.0613	96%	1.4710
5%	0.0766	95%	1.4556

**3. Result and Discussion**

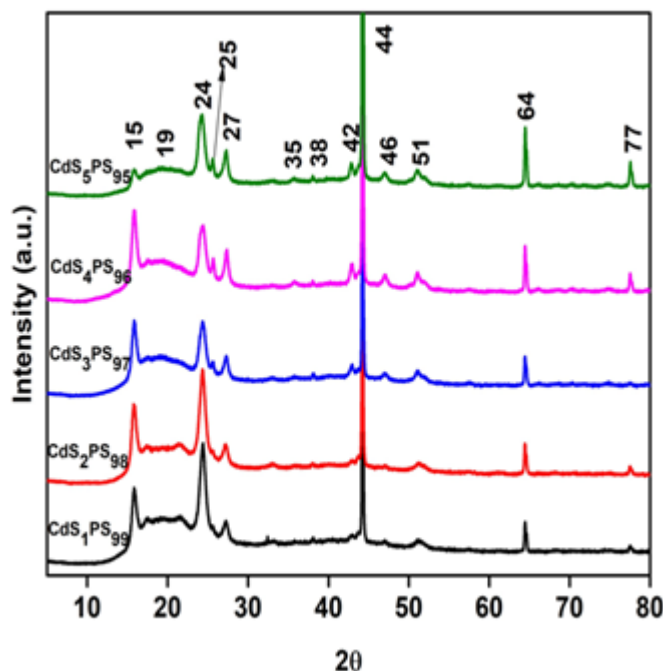
Small - angle x - ray diffraction (XRD) provides information about the topography of a polymer on length scales ranging from a few nanometres to some micrometres. Changes in charge distribution cause the absorbed x - rays to spread, resulting in recognizable patterns. [12]The XRD pattern of empty PS has Two significant peaks with centres at  $2\theta=9.51^\circ$  and  $18.88^\circ$  define the amorphous structure of the commonly produced PS. [13][14] The presence of halos is a defining feature of the design. Halos indicate that the compound's.

Major phase is the amorphous phase. It is determined from the XRD diffractogram that the empty PS thin film is amorphous. [15]

Our findings are well - aligned with Figures 3 which represent the diffractograms for weight percentages of CdS filled PS composite thin films of 1, 2, 3, 4, and 5 correspondingly.

The conventional XRD finding suggests that polystyrene has an amorphous nature and verifies the presence of CdS in PS nano - composite films. The amorphous character of the nanostructured thin films is represented by a broad peak. Peak positions are detected at  $2\theta= 24^\circ, 44^\circ$  and  $64^\circ$  for all filled samples. Diffraction patterns show that the peak shifts towards a lower elevation and rises in width while the peak height lowers as in PS the percentage of CdS rises. These kinds of results might result from the CdS particles' modest size. [16]

In addition to the broad peak, most XRD patterns in the range of  $2\theta = 19^\circ, 35^\circ, 38^\circ, 46^\circ$  and  $51^\circ$  show a minor hump - like pattern that is identical to the existence of CdS in PS, which appears in the XRD graph figure 2. Nanostructures created by polymers are somewhat amorphous and somewhat crystalline. [17]



**Figure 2:** Display the XRD pattern a) **Black** (CdS 1% and PS 99%) b) **Red** (CdS 2% and PS 98%) c) **Blue** (CdS 3%

and PS 97%) d) **Pink** (CdS 4% and PS 96%) e) **Green** (CdS 5% and PS 95%)

Amorphous solid microparticles are a significant approach to increase the seeming solubility, solubilization, and ultimately bioavailability of pharmaceuticals that are weakly water - soluble due to the greater solubility of amorphous chemicals (relative to their crystalline equivalents). [18]

The XRD of nanocomposites of CdS filled PS shows both character of amorphous solid which is suitable for oral drug delivery.

**FTIR (Fourier Transform Infrared Spectroscopy)**

In table 2, unexposed PS polymer film's FTIR spectroscopy graph is displayed along with their related frequency connections in Table 3.

In the table 2 PS FTIR spectra between 3700 and 400 cm<sup>-1</sup>. The five peaks that make up the major frequency bands, which are rings and in the main chain in the 2800–3100 cm<sup>-1</sup> region, are caused by C–H stretching vibrations in aromatic the peaks are located at 1601, 1492, 1451, 1029, 757, and 698 cm<sup>-1</sup>, which are caused by C - H bond skeletal vibrations and deformation in PS polymer. [19][20]

**Table 2:** Unfilled polystyrene (PS) polymer film and frequency connections

Sr. no.	Wavenumber (Cm <sup>-1</sup> )	Connections
1	1601	C - H bond stretch
2	1492	C - H bond stretch
3	1451	C - H bond stretch
4	1027	C - H bond stretch
5	757	C - H bond stretch
6	698	C - H bond stretch

In figure 3 shows FTIR spectra (a), (b), (c), (d) and (e) represents 5%, 4%, 3%, 2% and 1% CdS respectively. The range of frequency is between 400 - 3000 cm<sup>-1</sup>. The absorption peaks of FTIR spectra 1%, 2%, 3%, 4% and 5% CdS in polystyrene are given in table4, reveals the relation with frequency get from different bending and stretching vibrations surrounded.

In figure 5, exhibits some bands which are quite similar as the bands present in Table 2 with truly little variations due to the dispersion of CdS in polystyrene. Some other peaks are also there which represent the different atomic bonding such the peaks around 2849cm<sup>-1</sup> and 2922 cm<sup>-1</sup> due C - H bond stretching present in macromolecule of PS connected with aliphatic nature hydrocarbons are mainstay.

**Table 3:** CdS and PS nanocomposites relation with frequency

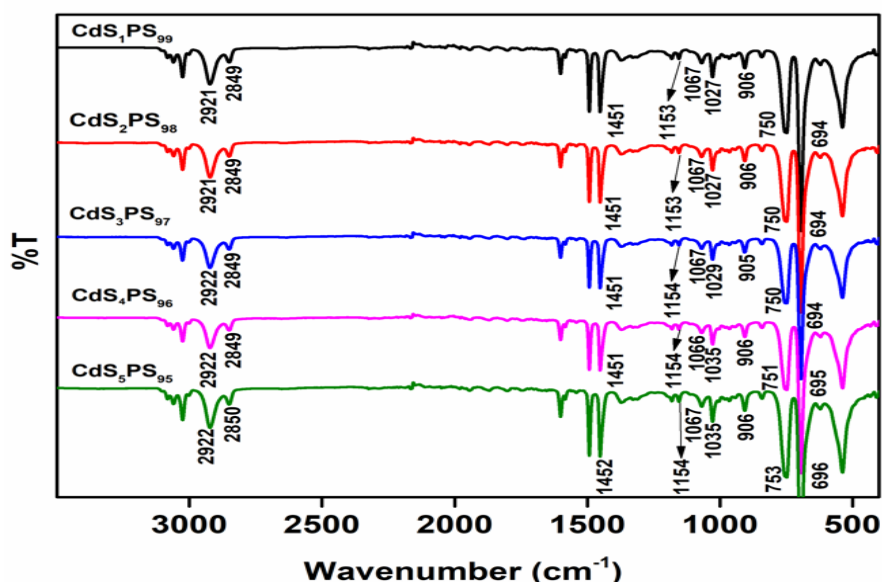
Sr. no.	Frequency (Cm <sup>-1</sup> )	Frequency (Cm <sup>-1</sup> )	Frequency (Cm <sup>-1</sup> )	Frequency (Cm <sup>-1</sup> )	Frequency (Cm <sup>-1</sup> )	CONNECTIONS
	CdS 1%	CdS 2%	CdS 3%	CdS 4%	CdS 5%	
1	694	694	694	695	696	C - H bond bending
2	750	750	750	751	753	C=C double bond stretching asymmetric
3	906	906	905	906	906	C - H bond bending
4	1027	1027	1029	1035	1035	O - H bond bending
5	1067	1067	1067	1066	1067	C=C double bond stretching asymmetric
6	1153	1153	1154	1154	1154	C - H bond stretching
7	1451	1451	1451	1451	1452	C - H bond stretching
8	2849	2849	2849	2849	2850	C - H bond stretching
9	2921	2921	2922	2922	2922	C - H bond stretching

Peaks exhibits C - H bond bending frequency are at 694 Cm<sup>-1</sup> and 906 Cm<sup>-1</sup>. Two weak peaks are also visible at 750 Cm<sup>-1</sup> and 1067 Cm<sup>-1</sup>. One peak around 1027Cm<sup>-1</sup> represents the O - H bond bending.

After comparing, table 3 and table 2 our findings is in good compliance with the functional groups of unfilled PS. Although, the intensity of peaks is decreasing but the

frequency of all the vibrational peaks are verifies the presence of PS and CdS composite. [21]

The conclusion drawn from the foregoing explanation is that the emergence of new peaks and modifications to existing peaks in the FTIR Spectral data directly point to the formation of complexes of CdS with PS and are congruent with the XRD results.



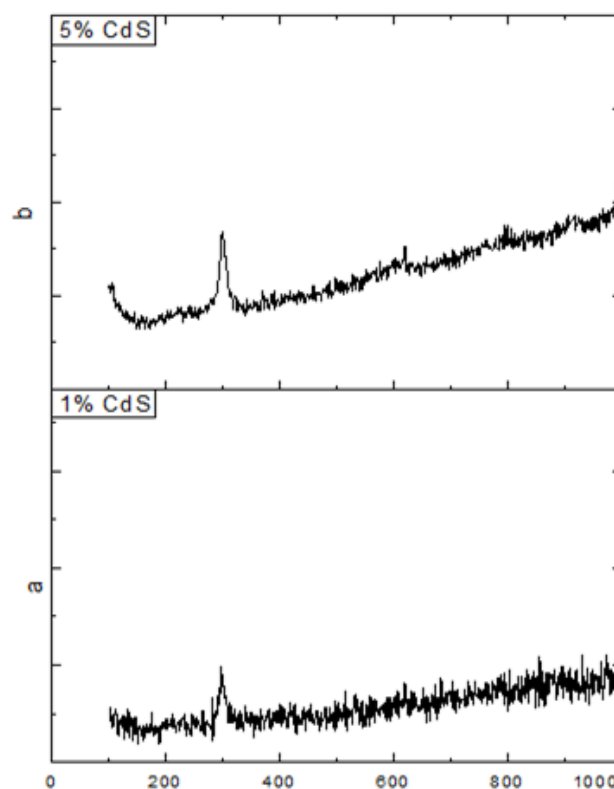
**Figure 3:** (a) Green colour  $\text{Cd}_5\text{PS}_{95}$  (CdS 5% and PS 95%) (b) Pink colour  $\text{Cd}_4\text{PS}_{96}$  (CdS 4% and PS 96%) (c) Blue colour  $\text{Cd}_3\text{PS}_{97}$  (CdS 3% and 97%) (d) Red colour  $\text{Cd}_2\text{PS}_{98}$  (CdS 2% PS 98%) (e) Black colour  $\text{Cd}_1\text{PS}_{99}$  (CdS 1% and PS 99%)

### Raman Spectroscopy

Figure 6 shows the Raman Spectral data of the synthesized nanoparticles of CdS and PS for 1% and 5% of CdS in the region of  $100 - 1000 \text{ cm}^{-1}$ . All nanostructured thin films had their Raman spectroscopy data obtained, however only for lower and upper percentages, i. e., for 1 and 5 percentage of CdS are displayed in the graph. Raman scattering of CdS and PS nanostructured thin films with of 2, 3, and 4 percentage of CdS as well as void PS film are excluded from graph to conserve space.

According to Figure 6, the longitudinally optical phonon scattering (LO) phase of CdS is represented by Raman shift about  $296.64 \text{ cm}^{-1}$ . For PS film loaded with CdS at 1%, minute wave number shifting is seen. Some other peaks that are also visible are caused by the organic particles' vibrations. Both the first and second order LO phonon Raman peaks of Nanocomposites are fully evident in their Raman spectra around  $296.64$  and  $619.30 \text{ cm}^{-1}$ , respectively. The third peak is a hump - shaped configuration about  $915.57 \text{ cm}^{-1}$ .

A CdS loaded PS nanocomposite film with wt% of 1 and 5 was created, based on Raman spectroscopy. Additionally, it affirms to the stability of nanostructured thin film.



**Figure 6:** RAMAN spectroscopy of (a) 1% CdS (b) 5% CdS

### 4. Conclusion

By adopting the solution casting approach, we were able to successfully create a novel PS nanocomposite film utilizing CdS as filler. The XRD measurements have verified that compound formation has occurred in the CdS and PS composite structure having both amorphous and crystalline is suitable for drug delivery. The synthesized sample is a thin composite film of PS/CdS, according to FTIR. Asymmetry inside the Spectra shows the impact of phonon restriction in the Raman line form which give information that our film is stable.

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