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Department of Physics, Materials of Science, Polymer Research Center, University of Basrah, Basrah, Iraq Synthesis and structural studies of CdS nanoparticles in PMMA polymer coordination

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Abstract

The UV-Vis with CdS end-point analysis was used to produce and investigate CdS. As a result, the PMMA nanocomposites as well as the hexadecylamine capped metal sulfide nanoparticles were heated to 180 °C in order to get them. The absorption and retronscopy techniques were utilized in order to study the optical properties of nanoparticles and the PMMA/nanocomposite. Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray Spectroscopy (EDS), and Powder X-Ray Diffraction (XRD) have been among the methods applied for such structural investigations. From 10. 15 to 37. CdS nanoparticles could be, a 25 nm aspect, characterized by the TEM imaging. The new research showed a widening the diffraction peak of polymer composite in XRD and EDS proofs of presence of sulfur nanoparticles in PMMA nanocomposite products.

Keywords: Nanocomposites; metal sulfide, poly (methyl methacrylate)

Introduction

The variations which occur on the basis of particle size and shape of semiconductor metal sulfur nanoparticles hold a great deal of significance and therefore has led towards the fabrication of the latter in recent times ^[1-6]. Dithiolates which have been under the researches of group 12 metal in the same source of the same precursor, as well as sulfide semiconductors have been developed to the formation of metallic sulfide materials such as NPs [7-10]. Our work focuses on dithiocarbamate which is common amine referred to as dithiolate ligand among researchers, in order to synthesize, characterize, and utilize 12 dithiocarbamate as precursors for nanoparticles ^[11-13]. They carry out countless studies and experiments related to transitions and transition ions, many of which have been applied to industry ^[14-15]. The reduction of the size is described already in the Chemical and physical character of the material change. The particles are called nanoparticles which refer to the particles that are considered to have an advantage that can be applied for the creation of the new materials. The energy gap for the CdS semiconductor is the required 2. 5 eV; As ZnS nanoparticles are also used for creation of optoelectronic devices, they posses energy band gap of 3. 68 eV. Composites of polymers and inorganic nanoparticles possess special features and they are considered to be very important in the science and technology today because the nanoparticles and polymers both exhibit structure as the nature of the hybrid material ^[16]. Looking at the synthetic approaches we worked out on with regard to the dithiocarbamate compounds' usage as precursors for the formation of metal sulfide nanoparticles is what we are presently engaged in ^[17-20]. Prepared is then a genesis and illustration on the CDSS nanoparticles/PMMA composites. Analysis of spectroscopic, optical, structural properties and the analytical properties about the sulfide nanoparticles embedded in the PMMA nanocomposite sample by absorption and emission spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray diffraction spectroscopy (EDS).

Experimental Procedures and measurements Materials

Merck chemicals supplied the hexamine, water-soluble ammonia (15 M), and metal chloride salts-cadmium (II) chloride utilized in this investigation, which was utilized as is without additional purification.

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Metal Sulfide Nanoparticle Synthesis

Using techniques described in the literature, metal sulfide nanoparticles coupled to hexadecylamine (HDA) were created ^[21]. A standard synthesis procedure involved dissolving 0.4 g of each cadmium (II) complex (CdL2) in tri-octyl phosphine (TOP), injecting the solution into 4.0 g of HDA, and heating the mixture to 180 °C. The combination was allowed to reach the appropriate temperature after °C refluxing was maintained for two hours, following which the mixture was allowed to cool to 80 °C. A drop in temperature of 30 °C was seen. After the solution was removed from the combination owing to low pressure, methanol was added to eliminate excess HDA, and centrifugation was used to separate the flocculant in order to extract CdS nanoparticles from CDA. The resulting metal sulfide nanoparticles were washed three times with methanol, filtered, and dried in smoke at room temperature.

Metal Sulfur Dioxide Nanoparticle and PMMA Nanocomposites Synthesis

10 ml toluene and 0.5 g polymer (Approximately 3% by weight) the polymer dissolved after 25 min, yielding a dark solution (Yellow color for CdS nanoparticles). The solution is poured onto the beaker and air is blown. Sulfite/PMMA nanocomposite material was obtained in powder form ^[22].

Measurements and requirements

Elemental analysis was carried out on Perkin Elmer elemental analyzer. Emission spectra of the nanoparticles was determined using Perkin Elmer Lambda 25 spectrophotometer while powder X-ray diffraction patterns of the metal sulphide nanoparticle and metal sulphide nanoparticle-polymer nanocomposites were recorded on a Bruker D8 Advanced, equipped with a proportional counter using Cu K α radiation (λ =1.5405 A). The scanning electron microscope (SEM) for the nanoparticle and nanocomposite were obtained using JEOL JSM-6390 LV-SEM at a rating voltage of 15-20 kV at different magnifications after they were coated with Au/Pd using the Eiko IB.3 Ion coater.

Results and Discussion

Synthesis

A fumigant for oil is produced by heating hexamine with carbon dioxide and hydrolyzed ammonium salt solution at room temperature. By conducting an equimolar substitution utilizing the ligand molecules and two chloride salts of metal as precursors, the metal complexes with Cd (II) were formed. The estimated values (C, H, N) are accurately in line with the experimental results acquired by means of elemental analysis based on the analysis results.

Morphology investigations of PMMA/metal sulfide nanocomposites

The information about the CdS nanoparticles reactions with PMMA and its surface morphology were taken using SEM technique. The exposure of the single gradients of polymers without added nanoparticles is shown in Fig. 1A, and SEM images of nanoparticles and polymer-based composite materials (That is, PMMA with CdS) are shown in Fig. 1B. SEM images show particles of the sulfide polymer (CdS/PMMA) have larger size diameters compared to CdS layer alone, which suggest strong interactions between itself and the polymer (PMMA). There is higher concordance in contact of diverse nanoparticles of SMS with polymer, which can be seen from the TEM micrographs in Figure 2. This interaction allows the threading of characters from the same program material and results in an improved hydrid with fresh features ^[22]. This was accomplished through employing energy dispersive spectroscopy (EDS) and thereby concluding the elements formations that is the constitution of the nano-composite. In the achieved spectra (Fig. 2), the detected Cd core and S of CdS nanoparticles are marked.



Fig 1: Images from the corresponding nanocomposites captured by SEM/TEM B; CdS/PMMA; PMMA (A)



Fig 2: EDS traces of the respective nanocomposites: CdS/PMMA

The X-ray Diffraction (XRD) patterns of PMMA/metal sulfide nanocomposites

The crystalline pattern of the XRD graphs for those samples can hardly be distinguished. However, it becomes obvious that there is some variation in the data and for example this is indicated by some fluctuations in the amplitude (the height of the peak) and the width of the peaks. What concerning the phenomenon of the wetpmma infiltration in the pores, this is the reason for the different appearance of extreme obsernosense that PAT dawn the difference between sarity and PMMA. White models made matrix for CdS/PMMA showed a slight decline in the energy, that is also in agreement with Flores-Acosta et all and Sathish et all. ^[23, 24] provided similar findings through zeolite research. White PMMA crystal diffraction diagram (without nanoparticles) - The CdS/PMMA nano composite is presented in the photos (3A, 3B) below.



Fig 3: XRD diagrams of the various nanotechnology: PMMA (A); B = CdS/PMMA

Optical properties of the metal Sulphide Nanoparticles

Besides excitonic peaks and shoulders, they are also proof of quantum confinement in nanomaterials and could also be used to calculate this measure. It has been demonstrated that nanoparticles have enhanced contrast as they are considered nanocrystalline materials which was evident ^[24]. The features of the cadmium sulfide nano particles are represented in Fig. 4 which summarized in Figure 5. This peak of the exciton creation in the CdS nanoparticles is seen at 289 nm. The optical absorption of nanoparticles measured here iwas found to be at 399 nm. The power stroke slip comes from the self-locking of the feed belt. This is because the quantized sizes of the nanomaterials were reduced as the confinement between dimensionality increased. Table 1 provides a summary of the different material types and their absorption maxima.

 Table 1: The metal sulfur's band gaps as determined by the absorbed peak:

Samples	Samples under Investigation	
	Wavelengths (nm)	Band gaps (eV)
CdS	289	4.29

Expectation spectra in semiconductor nanoparticles are wide bands; the ones that emit trapped emission have this shape, whereas spectroscopic line emission is sharp with the property of excitonic emission. The SrTiO3 has been reported to have a small shift in emission peaks, which can be explained by two distinctive peaks ^[22]. Emission at 388. Avoir une longueur de 5nm was seen for the CdS nanoparticles of the nanoparticles prepared. Nanoparticles which show their maxima absorbance shifts down to red because they contain absorbing centers.



Fig 4: Absorption and emission spectra of the nanoparticles: CdS.

Conclusion

By using a particular type of synthesis of the impurity, it was possible to to produce a perimeter that will be further characterized by spectrum and analytical chemistry methods. It was found that PMMA, using metallurgy as a base was a precursor for the synthesis of nanodimensional metal sulfides and metal sulfide nanocomposites. TEM image revealed the size of the nanoparticles which vary from 10 nm to several 00 nm sizes CdS nanoparticle size ranges from 1.5 to $3.7 \mu m$ while CdS nanoparticle size is

between 25 to 100 nm. The fact of CdS mean values difference of 13 in the peak diffraction is a key observation. 21 nm. According to EDS findings of the metal sulfides nanoparticles/PMMA nanocomposite, the polymer chain of the nanoparticles can be confirmed. This becomes more evident in the XRD of the nanocomposite, where the peaks are seen to be widening.

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