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Synthesis and Characterization of Polyaniline Cadmium Sulphide Nanocomposite via in Situ Polymerization

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Abstract

Nanocomposite of conducting Polyaniline with CdS nanoparticles have been synthesized by In Situ polymerization method. A series of the Nanocomposite have been synthesized by varying the weight percentage of CdS (5, 10, 15, 20, 25 %) with the polymerization of aniline (monomer). PANI-CdS 5 % Nanocomposite was optimized sample with high conductivity. Crystalline nature and size of the particles have been analyzed by X-ray diffraction study. The particle size was found to be range from 53 nm to 400 nm. This has been confirmed from SEM images.

Keywords: Polyaniline, Nanocomposite, In Situ Polymerization.

1 Introduction

Recently, conductive polymer composites have attracted much attention because of their variety of properties and functionalities, such as mechanical strength, Processibility [1-3] .Among conducting polymers, Polyaniline (PANI) is probably the most widely studied due to its several unique properties [4,5]. It ease of preparation, light weight, low cost, better electronic, optical properties, highly stable in air and soluble in various solvents, and good processibility [6-8]. Polyaniline (PANI) is one of the most interesting conducting polymers due to its low cost, good processability, environmental stability, unique active conduction mechanism [9] and reversible control of conductivity both by charge-transfer doping and protonation [10]. Inorganic semiconductors CdS, ZnS & PbS nanoparticles are the most promising materials used in various applications like sensors, optoelectronic devices and in solar cells. Polymeric nanocomposites consisting of organic polymer and inorganic nanoparticles in a nanoscale regime represent a novel class of materials that have motivated considerable interest in recent years. These composites exhibit new advantageous properties and can be very different from those of their individual counterparts. It is therefore expected that this type of materials will play increasingly important roles in research and in numerous applications. They frequently have special properties and are significant for many technological applications, ranging from microelectronics to catalysis, optoelectronic devices, and synthesis of lubricant and preparation of electrolytes for rechargeable batteries [11-14].

2 Methods and Materials

2,.1 Materials used

The Aniline Hydrochloride (AR grade) and Ammonium Persulphate (AR grade). Cadmium Nitrate Tetrahydrate (AR garde) obtained from Loba Chem. Mumbai, (India). Ammonium Sulphide received from National chemicals ,Vadodara, Gujrat(India). All the chemicals were used as received.

2.2 Synthesis of Nano CdS

100mL aqueous solution of $Cd(NO_3)_2$ (0.085M) was in One beaker and100 mL aqueous solution of $(NH_4)_2S$ (0.1M) was in another beaker. Aqueous Solution of $Cd(NO_3)_2$ (0.085M) was added drop wise to 100 mL aqueous solution of $(NH_4)_2S$ (0.1M) with vigorous stirring. Stirring was continued for 5 hours. The dark yellow precipitates of CdS nanoparticles were obtained. Left the precipitates overnight then filtered with Distilled water and acetone. Then dry in air and kept at 80 °C in Oven for 5 hr.

2.3 Synthesis of Polyaniline (PANI):

The synthesis was based on mixing aqueous solutions of aniline hydrochloride 0.2M and ammonium persulfate(APS) 0.25M at room temperature, followed by the separation of PANI hydrochloride precipitate by filtration and drying. More precisely, 2.59 g of Anilinium hydrochloride was dissolved in distilled water in a volumetric flask to 50 mL of solution. 5.71 g of ammonium persulfate(APS) was dissolved in water also to 50 mL of solution. Both solutions were kept for 1 h at room temperature (~18 − 24 °C), then mixed in a beaker, briefly stirred, and left at rest to polymerize. Next day, the resulting dark green PANI precipitate was collected on a filter, washed with three 100 mL portions of 0.2 M HCl, and similarly with acetone.

2.4 Synthesis of PANI-CdS Nanocomposite:

In the Present work polymer composite film of Polyaniline (PANI)-CdS was prepared for different weight percent of Cadmium Sulphide (CdS).

Synthesis steps of PAni/CdS nanocomposite are similar to the synthesis method of PAni. Different amount of CdS were dispersed into the APS solution and stirred for 1 hour prior to the addition of aniline. Aniline (0.4 mol) stirred with 0.4M H_2SO_4 in 100 ml of distilled water were added drop-wised using burette into the APS-CdS solution and stirred vigorously to form homogeneous dispersion. For convenience, PAni Composites were prepared with different weight percentages of CdS[15]. Same synthesis conditions were maintained for all composites as that of pure PAni.

3 CHARACTERIZATIONS

3.1 Scanning Electron Microscopy (SEM):

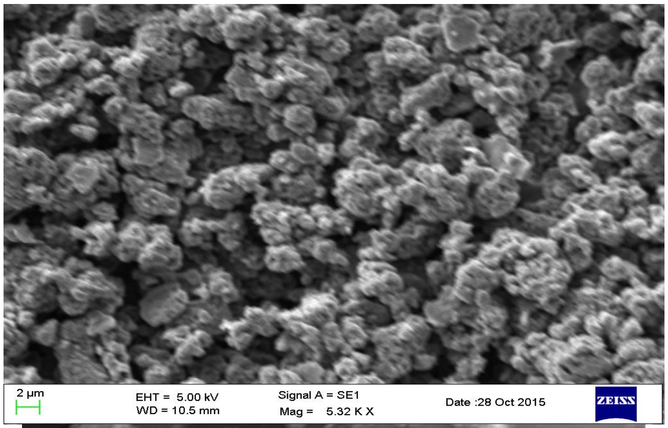
For the present work Field Emission Gun-Scanning Electron Microscope with model number JSM-7600F, was used. The resolution of the instrument was 1.0 nm at 15KV and 1.5 nm at 1KV.The accelerating voltage was in the range 0.1 to 30KV and magnification was about 25 times to 1,000,000 times. This technique gives the morphology synthesizes composite.

3.2 X-Ray Diffraction Technique:

XRD is an indispensable method for structural materials characterization and quality control which makes use of Debye-Scherrer method. In this technique diffraction pattern of X-ray is studied for powder/microcrystalline samples, where ideally every possible crystalline orientation is represented equally. New sixth generation MiniFlex 600 X-ray diffractometer (XRD) is used for the determination of crystallite size ,strain and molecular structure of sample.

4 RESULT AND DISCUSSION

The surface morphology of PANI, CdS and PANI-CdS Nanocomposite was carried out by scanning electron microscope (SEM) as shown in figures 1 (a,b,c). The fine microspheroidal surface was observed with poor matrix. Nanoparticles were not of homogenous size. The SEM was in good agreement with the experimental results. These materials are polycrystalline in nature. Particle sizes of PANI, CdS and PANI-CdS Nanocomposite are found to be range between 200 nm-550 nm , 75–400 nm and 25nm -340nm respectively. There may be agglomeration of crystallites which is clearly seen from the figure 1.(b). Amorphous structure with very fine particles is seen. Definite particle shape was not visible due to more fine amorphous powder. These images indicates that, particles grow via bond formation of source ions with their surface atoms, resulting in a continuous increase of the particle size. Further reaction between any two nanoparticles can lead to stable aggregates.



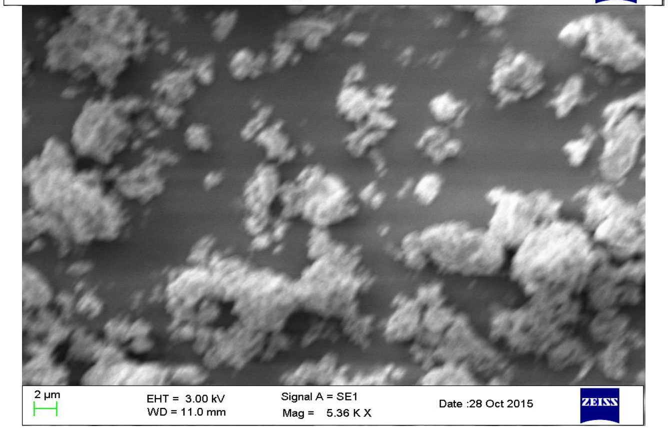


Fig 1(a) Fig 1(b)

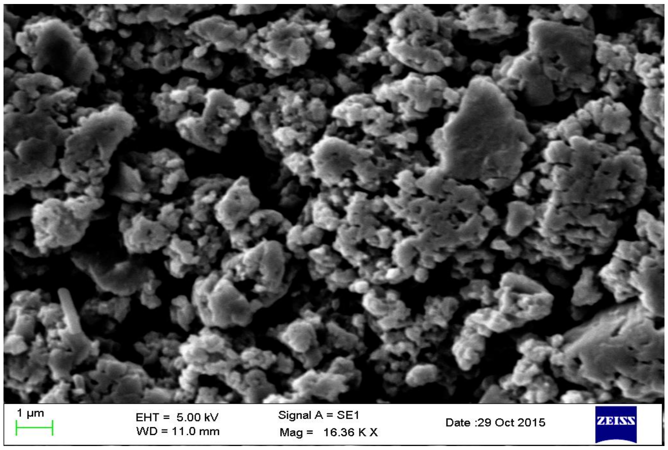
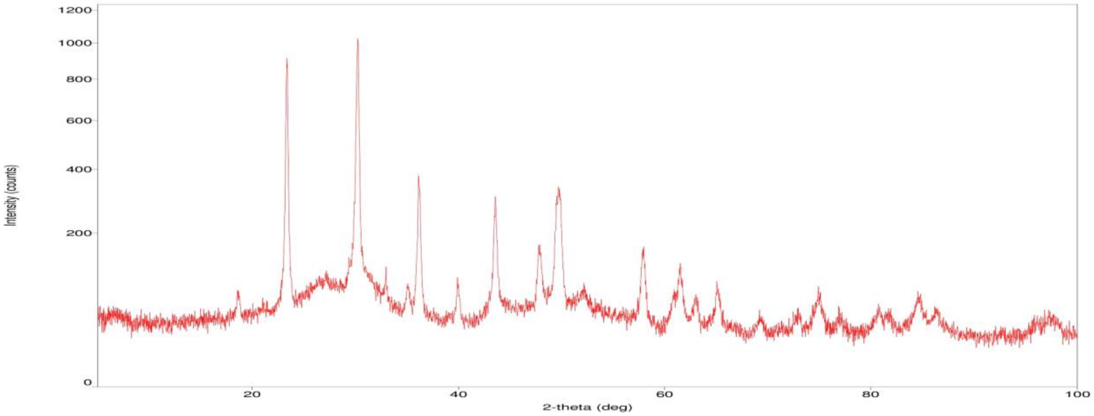


Fig 1(c)

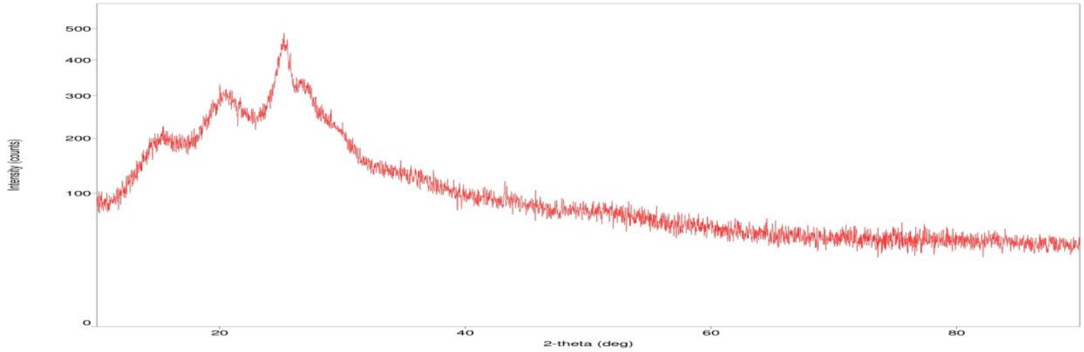
Fig 1(a)SEM of Pure PANI, Fig 1(b) SEM of Pure CdS, Fig 1(c) SEM of PANI-CdS Nanocomposite

The degree of crystallinity increased in Pani/Cds nanocomposite than pure Pani and Cds, clearly indicated the homogeneous distribution of nanoparticles in the polymer matrix. The crystalline size of the crystalline particle can be determined using Debye Scherer formula [$\frac{0.9\,\lambda}{\beta\cos\theta}$] and it is found that the grain size of PAni/Cds nanocomposite is (75 nm). The XRD pattern of PAni shows the three broad peaks at 2θ= 15.13^0 , 20.34^0, 25.20^0.

CdS – Fig. 2(a)PANI



PANI – Fig. 2(b)



PANI-CdS 5 %-- Fig. 2(c)

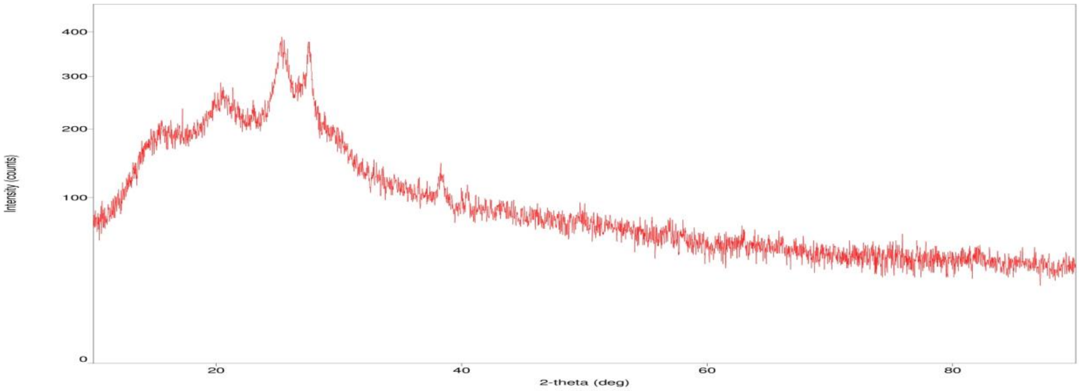


Fig 2(a)XRD of Pure PANI, Fig 2(b) XRD of Pure CdS, Fig 2(c) XRD of PANI-CdS Nanocomposite we can observe broad diffraction peaks for all composites indicating the presence of very small size crystallites. Again, it can be seen that the Nanocomposite show crystalline as compared to that of pure PANI due to uniform dispersion of the nanoparticles in the polymer matrix. The cristallinity of the Nanocomposite is dominated by polymer matrix.

PANI showed two peaks at $2\theta = 20.34^0$ and 25.20^0 and has an amorphous nature [12]. A Nanocomposite show the greater Crystallinity due to the addition of CdS in PANI matrix as compared to pure PANI and shows the peaks related to CdS with increasing concentration in PANI matrix as seen from figure 2(c).

5 Conclusion

PANI- CdS Nanocomposite was fabricated and characterized using the coprecipitation and polymerization techniques. It is seen from XRD Characterization the peak shows crystallite nature of CdS and each highly agglomerated irregular shape which reflects the amorphous state of the obtained PANI. SEM Morphology confirms PANI- CdS Nanocomposite are polycrystalline in nature. Particle sizes of PANI, CdS and PANI-CdS Nanocomposite are found to be range between 200 nm-550 nm , 75–400 nm and 25nm -340nm respectively.

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