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A Study on Structural and Electrical Properties of Cadmium Oxide (CDO) Doped Polyaniline (PANI) Composites

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ABSTRACT: This research work presents the synthesis and characterization of PANI-CdO composites by in situ polymerization technique. CdO content in the composites was varied from 2 to 10 wt% in the steps of 2 wt%. Microstructure and structural characterization of the developed composites was conducted using scanning electron microscope and X-ray diffraction. The SEM study shows a granular agglomerated morphology andhomogeneously distributed throughout the polymer sample which implies decrease in inter-granular distance between grains. The XRD analysis shows CdO particles is well dispersed in the polyaniline matrix.DC conductivity was studied in the 40 - 200°C temperature range. DC conductivity of all composites was found to increase with the increase in temperature. Hopping of polarons increases from one localized state to another when the temperature is increased which is why it is observed that higher DC conductivity values at high temperatures.

KEYWORDS: Polyaniline-CdO composites, Scanning electron microscope, X-Ray diffraction, DC conductivity.

I. INTRODUCTION

Polyaniline (PANI) is a conducting polymer of the semi-flexible rod polymer family. Indeed in spite of the fact that the compound itself was discovered above 150 years ago, only the early 1980s polyaniline has captured the intense attention of the scientific community.

Among the accessible conducting polymers, polyaniline(PANI) is found to be the foremost encouraging since it hastunable properties, ease of synthesis, stability and low cost monomer when compared to other conducting polymers. For this reason, several authors have accomplishedwide-rangeanalysis on the synthesis, characterization and use of PANI and its components. However, the solubility of few conducting polymers may be improved through doping with an appropriate dopant or adjusting the beginning monomer. Hence, there is plenty of scope for modifying the processability and conductivity of PANI by selecting an appropriate dopant and appropriate level of doping as well as by assuring its structure at the time of synthesis[1].

Blending the PANI with a commercially accessible polymer yields good compound whose processability and mechanical properties are quite worthy and they are successful utilized in many fields. Polymer composites containing PANI where used as a conducting filler in other polymer matrixes, these fillers have obtainedplenty of attention because of the combination of enhanced processability and good mechanical properties joined with good conductivity. Therefore, there is a wide opportunity for the practical applications of these composites[2,3]. It is also found that there exists a modification in the properties of PANI due toirradiation process [4]. To exploit and utilize the promising properties of conducting polymers, it is essential not only to accomplish a high amount of conductivity but also need to make them appropriate for processing either single or in a composite (matrix) of anallowed host polymer. In recent years, maximuminvestigations on PANI as well as other conducting polymers have mainly focussed on these two aspects.

Cadmium oxide (CdO) is n-type semiconductor used as a transparent conductive material. Cadmium oxide has been used in applications such as photodiodes, phototransistors, photovoltaic cells, transparent electrodes, liquid crystal displays, IR detectors, and anti-reflection coat. With this background of multi-functionality CdO, it was thought worthwhile to use theCdO as an inorganic counterpart in the composite preparation.



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II. LITERATURE SURVEY

Yuvraj Sing Negi et al[5] reviewed the article; recent research work on polyaniline-type conducting polymers has been surveyed. Research and development work have been reviewed with special interest in chemical, electrochemical, doping processes, and polyaniline-based composite processes. They found out that process development with high accuracy of product results will have a direct relationship with structure and properties. Polyaniline based on a variety of structures generated from molecular structural modification have given more interesting results for future applications.Faisal et al [6] synthesized conducting polyaniline-stannous oxide (PANI-SnO) composites by the in situ polymerization of aniline in the presence of SnO and investigations reveal that the PANI-SnO composites are potential candidates for EMI shielding applications for both the X and K_{α} bands. AlemtemsuOzukum et al[7] successfully synthesized PANI/CdOnanocomposites by in-situ polymerization method in the presence of CdO nanoparticles.The results of SEM show the formation of the composite and indicate an interaction between PANI and CdO nanoparticles. The electrical property of PANI/ CdOnanocomposite increases with increasing in frequency this is because of interaction between metal oxide and PANI. MeghaSawarka et al [8] successfully synthesized pure PANI, ZnO and CdO nanoparticles along with PANI/ZnO, PANI /CdOnanocomposites by cost effective technique. ZnO and CdO nanoparticles were synthesised by sol gel method. Pure PANI and PANI/CdOnanocomposites have been successfully prepared by a chemical polymerization method. The SEM observations revealed that PANI has irregular sheet like morphology and the morphology remained stable after the combination with CdO nanoparticles. Hence this composite is a promising material for electronic potential and sensing applications.

To the best of our knowledge, this is the first attempt by the authors reporting the synthesis, characterization anddc conductivity polyaniline and polyaniline/Cadmium oxide (PANI/CdO) composites prepared by in situ polymerization technique.

III. METHODOLOGY

A. SYNTHESIS OF PURE POLYANILINE (PANI)

Polyaniline was prepared by using extra pure and AR grade reagents aniline (99.9%), ammonium persulphate (99.9%), and hydrochloric acid (99.9%) using in situ polymerization method. Ammonium persulphate (0.2 M) was added drop wise to a stirred solution to avoid warming of the aniline (0.1 M) solution dissolved in 1 M of an aqueous solution of hydrochloric acid (1 N, aniline hydrochloride) at a temperature around 5°C. Along with this addition, stirring was continued for 2 hrs using a magnetic stirrer to affirm completion of the reaction. During the polymerization reaction, HCl was utilized as a protic acid and the temperature was asserted at about 0^0 C (low) by using a freezing mixture. The end prepared product was a green-colored precipitate (emarldine salt). This precipitate was filtered by using Buckner funnel along with vacuum pump. Further, it is rinsed with acetone and deionized water in order to remove the oligomers and excess ammonium persulphate and then with 1 N HCl solution to remove the Cl⁻ ions and unreacted aniline until clear filtrate. At last, the precipitate was dried in hot air furnace for 24 hours at room temperature to attain a constant mass.

B. SYNTHESIS OF PANI-CADMIUM OXIDE COMPOSITES

Synthesis of the PANI–Cadmium oxide (CdO) composites was carried out by polymerization *in situ*. Aniline (0.2 M) was dissolved in 1 M HCl and stirred for 2 hours to form aniline hydrochloride. Cadmium oxide was added in the mass fraction to the obtained solution with energetic stirring in order to keep the CdOhomogeneously suspended in the mixture. To this solution, 0.2 M of ammonium persulphate, which acts as an oxidant was slowly added drop-wise with continuous stirring at room temperature for 8 hours to completely polymerize the monomer aniline. The precipitate was filtrated, rinsed with demonized water, and finally dried in a furnace for 24 hours to attain a constant mass. In this way, PANI– CdO composites containing various mass fractions of CdO (2%, 4%, 6%, 8% and 10%) in PANI were synthesized.



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Figure-1: Flow Chart of the Synthesis of PANI/CdO composite

IV. EXPERIMENTAL RESULTS

A. STRUCTURALREVIEW

Presence of reinforcing phase and formation of any other phase was studied with the help of X-ray diffraction (Philips XPERT diffractometer) using Cu K α radiation ($\lambda = 1.54 \text{ A}^\circ$). The diffraction pattern of the pure polyaniline is shown in the figure 2(a). The broad peak is observed at $2\theta = 25.63^\circ$, which clearly indicates complete amorphous nature. These results clearly indicate the dominant amorphous nature which causes high mobility of the ions in the material [9]. Figure 2(b-f) shows the X-ray diffraction pattern of all composites with the varying CdO composite. The intense sharp peaks corresponding to CdO particles were observed at $2\theta = 33.22^\circ$, 40.14° , 49.54° , 67.43° , 78.60° to (2 1 1), (4 0 1), (1 1 2), (2 2 2), (3 1 1) planes which indicates the presence of CdO in PANI. The nature of peaks observed for these composites suggest the crystalline nature. By comparing the XRD patterns of pure polyaniline with its composites, it is observed that there is a homogeneous distribution of CdO in polyaniline matrix and also confirmed that CdO has retained its structure even though dispersed in PANI during polymerization reaction. Similar observations were reported by Aashis S Roy et al [10] and SubhashKondawar et al [11] in their work on PANI/CdOnanocomposites. On the other hand presence of broad peaks in between 15° and 30° suggests amorphous structure corresponding to PANI. The X-ray diffraction peaks of the material were analyzed by using Debye–Scherrer equation.

$$\mathbf{D} = \frac{k\lambda}{\beta \cos\theta}$$

Where, D is the crystalline size, λ is the wavelength of X-ray, β is the full-width at half-maximum and θ is the diffraction angle.



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Figure-2: XRD pattern of (a) PANI, (b) CdO (2%)/PANI, (c) CdO (4%)/PANI, (d) CdO (6%)/PANI, (e) CdO (8%)/PANI, (f) CdO (10%)/PANI

B. MORPHOLOGICAL STUDY

Scanning electron microscopy (SEM: Philips XL-30 ESEM) was used to study the powder morphology of developed PANI-CdOcomposites. Figure 3(a) shows the SEM image of pure PANI, which has highly agglomerated granular in shape. From the SEM micrographs it can be observed that pure PANI has well interconnected grains with amorphous nature. The SEM images of PANI-CdO composite figure 3(b-f) reveals that the particle size is in the range of nanometre and they are spherical and porous structure [12]. It shows highly agglomerated, more densely packed particles in which the CdO particles are embedded in PANI matrix [12] and are homogeneously distributed throughout the polymer sample which implies decrease in inter-granular distance between grains in that composites which facilitates easier charge transport through the carbon back-bone of its polymer chain[13].It is also observed that CdO particles are surroundedbypolyanilinematrixandhenceitappears as agglomeratedmacromolecules.



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Figure-3: SEM images of (a) PANI, (b) CdO (2%)/PANI, (c) CdO (4%)/PANI, (d) CdO (6%)/PANI, (e) CdO (8%)/PANI, (f) CdO (10%)/PANI

C. DC CONDUCTIVITY

DC conductivity (Keithley 6514 electrometer) of all composites with varying CdO concentration with was measured by standard four probe method for temperature range of 40°C - 200°C.Figure 4 shows the variation of σ_{dc} conductivity as a function of temperature. It was noted that conductivity increased with an increase in temperature. This may be due to the thermally activated behaviourfollowing quasi-1D the variable range hopping conductivity model as that of pure PANI. The increase in conductivity is due to the increase in efficiency of the charge transfer between CdO and polymer chains with the increase in temperature. The conductivity of polymers depends on the conjugation length and chain alignment of the polymer; this plays an important role in the transportation of charge carriers. There will also be a molecular reorganisation upon heating, which renders the molecules in a favourable state for electron-hopping from one PANI island to another.

Change in electrical conductivity of the conducting polymer composites also depends on the conductivity of the filler particles into conducting polymer matrix. The conductivity varies directly with the temperature, according to an expression of the following form[14]:

 $\sigma(T) = \sigma_0 \exp[(-T_0/T)^{1/4}],$



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where σ is the conductivity, T is the temperature, and σ_0 is the conductivity at the characteristic temperature T₀.

At lower temperature, conductivity of PANI-CdO composites was found to be slightly lower than that of pure PANI. This may be due to the partial blockage of the conductive paths by CdO particles embedded in the PANI matrix for the charge carriers which do not have sufficient energy to hop between favorable sites [11]. Although composites have less conductivity at lower temperature as compared to pure PANI but conductivity of composites is due the extended chain length of polyaniline where polarons possess sufficient energy to hop between favourable sites. The electrical conductivity of PANI-CdO composites shows semiconducting behavior as that of conducting polyaniline with filler concentration dependant variation of transport parameters. For all temperature range the conductivity of 6% and 10% CdO content incorporated composites showed higher values. This may be due to variable hopping from one polymer chain to another. Such composites materials can be useful for electronic devices. Similar observation was seen in PANI-NiOnanocomposites which was studied by B. I. Nandapure et al[15].



Figure-4: DC conductivity of PANI and PANI/CdO composites at different temperatures.

V. CONCLUSION

The following conclusions were drawn from the present work on PANI-CdO composites, PANI-CdO composites with differentCdO content were successfully developed by in situ polymerization.XRD pattern revealsamorphous nature of polyaniline and regularly arranged crystalline nature of polyaniline composites. The SEM studies reveal that they are agglomerated and granular in morphology with an increasing amount of CdO with different mass fractions, suggesting intermixing of the CdO particles with the PANI matrix. The DC conductivity as a function of temperature for polyaniline and polyaniline–CdO composites was studied in the temperature range from 40^o C to 200^o C at mass % of CdO in polyaniline. At higher temperatures, it was found that conductivity increases because of the polarons hopping from one localised state to another. This is attributed to the temperature dependence of the conductivity in the composites, which exhibits typical semiconductor behaviour.

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