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Polyaniline-SbO_2 Composites: Preparation, Characterization and a c conductivity Study

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Abstract: *Polymeric materials containing metal oxide particles constitute a new class of polymer composites materials. Chemical oxidation of aniline is carried out for polyaniline (PANI) and SbO2 inserted PANI (PANI-SbO_2) composite material. Varied weight percentage of SbO_2 in PANI constitutes different PANI- SbO_2 composite materials to know detailed changes. Pure SbO_2 and prepared PANI composites were characterized by various characterization tools. Structural changes of SbO2 and composite materials were carried out by X-ray diffraction (XRD) tool, morphological study by Scanning Electron Micrograph (SEM) tool and bonding changes was observed by Infrared (IR) study. Structural, morphology and bonding variation is observed in PANI- SbO_2 composite materials compared to pure oxide and PANI samples. Dielectric study of the composite materials is undertaken for its dielectric behavior. The study shows the variation of the dielectric behavior for different weight percentage composite materials.*

Key words: *Polymeric materials, Oxidation, Dielectric, SEM, XRD, IR, SbO_2*

I. Introduction

Synthesis of polymer composites of core shell inorganic particle-polymer has attracted much research attention in recent years because its properties [1-2]. In particular, the composites of core shell metal oxide particles-conducting polymer combine the electrical properties of the polymer shell and the magnetic, optical, electrical or catalytic characteristics of the metal oxide core, which could greatly widen their applicability in the fields of catalysis, electronics and optics [3]. Many efforts have been made to successfully prepare composites such as Fe_2O_3-polypyrrole by chemical preparation and electrochemical method [4-5]. Besides the preparation of MO-Polymer, the synthesis of hollow conducting polymer capsules is expected to become much feasible by the chemical removal of the metal oxide core of the MO-Polymer. The resulting conducting polymer capsules with controllable hollow structure have shown promising prospective applications [6]. The challenge for the preparation of the MO-Polymer is how to generate the polymer coating uniformly and completely on the surface of the metal oxide core by a polymerization reaction in a solution phase. The key issue aims at slowing down the rate of polymerization and controlling the polymerization on the surface of the core rather than in the solution. The fabrication of MO-Polyaniline is particularly of interest because polyaniline (PANI) is one of the most important conducting polymers with high conductivity, ease of synthesis, and good environmental stability [7].
In this paper, we describe the synthesis of PANI and SbO_2 dispersed PANI composite materials through oxidative polymerization of aniline. As prepared PANI and its SbO_2 composite is well characterized by various characterization techniques. a.c conductivity study of the as prepared PANI composite material is also well studied for its conducting behavior.

II. Experimental

2.1. Materials and Methods

PANI and PANI composites were prepared by chemicals i.e Ammonium persulphate $(NH4)_2S_2O_8$, Hydrochloric acid (HCl), aniline and SbO_2 are of AR grade. Double distilled water was used in the synthetic process. In situ polymerization of aniline was carried out for PANI and SbO_2 composite materials.

2.2. Synthesis of PANI-SbO_2 Composites

0.1 M aniline was dissolved in 1M HCl to form aniline hydrochloride. SbO_2 was added in the weight percent of 10, 20, 30, 40 and 50 to the above solution with vigorous stirring in order to keep the SbO_2 materail suspended in the solution. 0.1M of ammonium persulphate $[(NH_4)_2S_2O_8]$ as an oxidant was added slowly to the reaction mixture with continuous stirring for 4-6 hours at 0-5^{O}C. The precipitated powder recover was vacuum-filtered and washed with deionizer water. Finally, the resultant precipitate was dried in an oven for 24 hours to achieve a constant weight. Similarly five different PANI- SbO_2 composites with different weight of SbO_2 (10, 20, 30, 40 and 50) in PANI have been synthesized. Pure polyaniline was prepared by chemical oxidation of aniline without adding Indium oxide [8].

2.3. Preparation of Pellets

Varied concentrations of prepared composites were pressed under pressure for its pellet form. The test samples to be used were prepared in pellet form of diameter 10mm and thickness 3mm by applying pressure of 7t using Pye-Unicam dye. The contacts for these composites were made using silver paste as electrodes on both sides.

2.4. Characterization

The structures of as prepared polymer composite were studied by X–ray diffraction using Phillips X–ray diffractometer (PW3710) with Cu Kα as source of radiation. Morphology and bonding of the above polymer composites were studied by Phillips XL 30 ESEM and Perkin–Elmer 1600 spectrophotometer in KBr medium tools respectively. Dielectric measurements were carried out at room temperature over the frequency range 10^2-10^7Hz using the Hiokie LCR Q meter.

III. Results And Discussion

1. X-ray diffraction

Figure-1 shows XRD pattern of PANI- SbO_2 at 50% weight composition. The pattern shows large number of peaks confirms shows the presence of SbO_2 reflections in the composite pattern and is comparison with literature. [9]. Some of the SbO_2 peaks are disappeared in the composite pattern compared to XRD pattern of pure SbO_2 is due to demasking of the oxide in the polymer matrix. This oxide peaks in the composite pattern confirms the formation of SbO_2 dispersed polyaniline composite.

2. Scanning Electron Microscopy (SEM)

Figure-2 shows SEM image of SbO_2 sample. This image shows, the most of the particles are in spherical shape and also a compact arrangement. The particle nature clearly indicates the crystalline structure of the sample. Figure-3 shows SEM image of pure PANI obtained by chemical oxidation of aniline. The close packing with amorphous nature is observed in the image. This image also shows the high particle size with self assembly. Figure-4 shows the SEM image of PANI- SbO_2 at 50% weight percentage. The close mapping of spherical Sb particles in the polyaniline matrix is observed in the image. Clear enhanced crystalline morphology PANI composite is observed. Morphological change in the composite compared to pure PANI and pure SbO_2 show the formation of PANI composites. Figure 5 shows a representative energy-dispersive X ray (EDX) spectrum of as prepared SbO_2 sample. The pattern shows the presence of Sb metal peaks, which again confirms the presence of SbO_2 in polyaniline matrix.

3. Infrared Study

The bonding nature in pure SbO2 and PANI- SbO_2 composite was well studied by infrared tool. This study is to ascertain the metal- oxygen (M-O) in SbO_2 and shift in frequency in metal oxide inserted PANI composite sample. Metal oxides generally give absorption bands below 1000cm^{-1} arising from inter-atomic vibrations [9]. Figure-6 shows FTIR spectrum of pure SbO_2. The sample shows the absorption peak at 1659, 1261, 724, 555, 535, 531, 528cm^{-1}. The peaks below 1000cm-1 is due the presence of Sb-O bonding.

Figure-7 shows the FTIR spectrum of as prepared PANI- SbO_2 composite. The spectrum shows the peaks at 2287, 2113, 1915, 1552, 1408, 1290, 1081, 1000, 886, 806, 577, 561, 527 cm-1. Peaks below 1000cm-1clearly shows presence of SbO_2 sample. The peak at 1081cm-1 is due to the B-NH+ = Q vibration, indicating that the PANI is conductive and is in the form of emeraldine salt. The absorption peak at 1000 cm-1 is due the C-H bonding of the aromatic ring. The peak 577 is attributed to the out of plane deformation of C-H aromatic ring. The additional peaks at 2287, 2113, 1915, 1552, 1408, 1290 may be due to some overtones.. Some additional peaks and shift in vibrational frequency were also observed on comparison with pure SbO_2 spectrum. This confirms the formation PANI- SbO_2 composite.

4. a.c conductivity

Figure 8 shows the variation of ε' as a function of frequency for PANI – SbO_2 composites (different wt%). In all the cases it is observed that, the dielectric constant is quite high at low frequency and decreases with increase in applied frequency. it is also observed that the values of dielectric constant decreases up to 40 wt% and then increases to 50wt%.

The observed behavior may be due to the Debye relaxation mechanism taking place in these materials [10]. (19)

IV. Conclusion

The preparation of PANI composites with various metal oxide materials is simple and this method may adopt for the preparation pervoskites and garnets polymer composites. Structural and morphological changes has taken place due to insertion of oxide material in to polymer matrix. Demasking of the particles is also observed in the composite material. Shifts in vibrational frequencies of oxide material or polymer is also observed in the composites.

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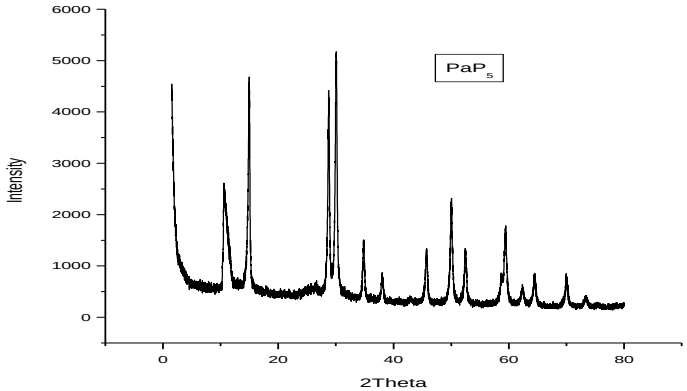

Figure 1: XRD pattern of pure PANI- SbO2 at 50% weight composition

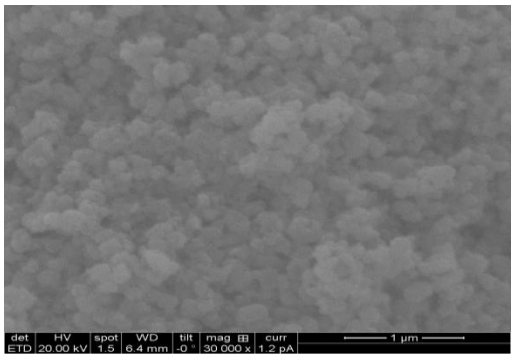

Figure 2: SEM image of SbO_2

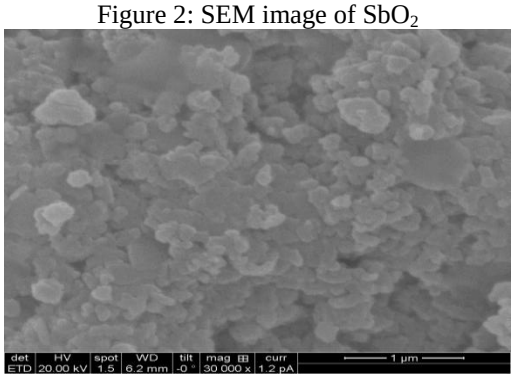

Figure 3: SEM image of pure PANI

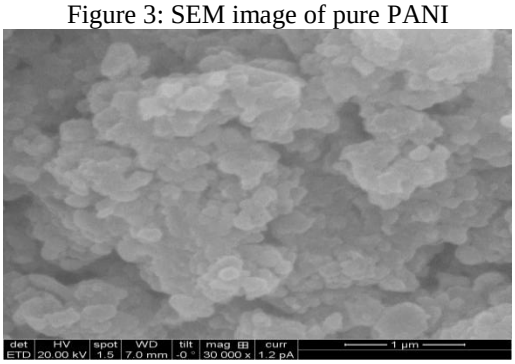

Figure 4: SEM image of PANI- SbO2 at 50% weight composition

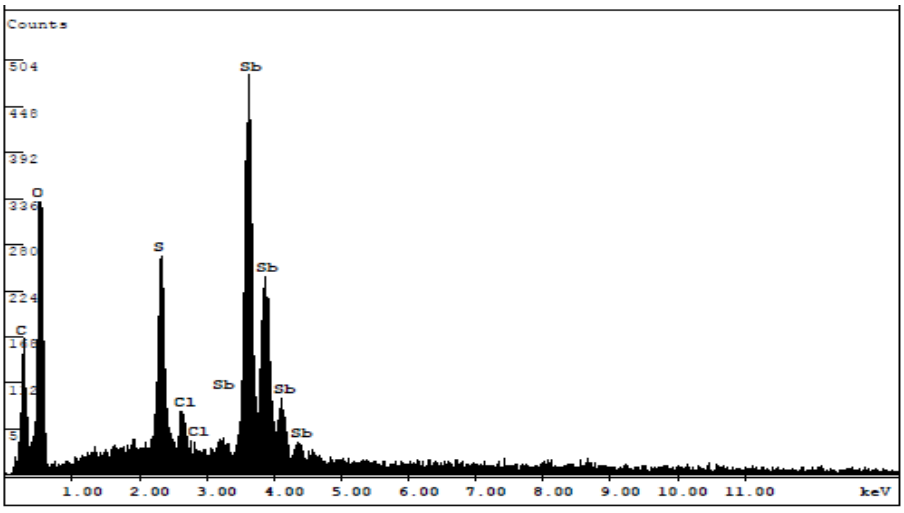

Figure 5: EDAX of PANI- SbO_2 at 50% weight composition

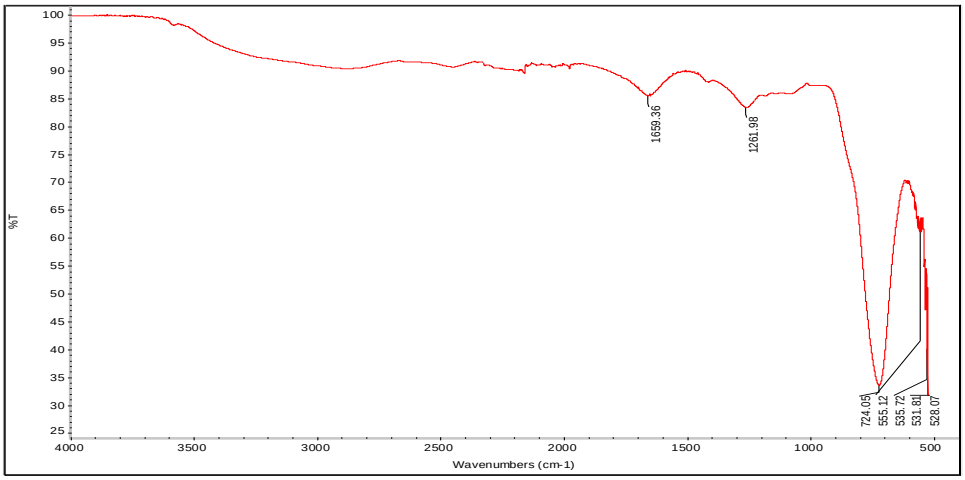

Figure 6: FTIR of pure SbO_2

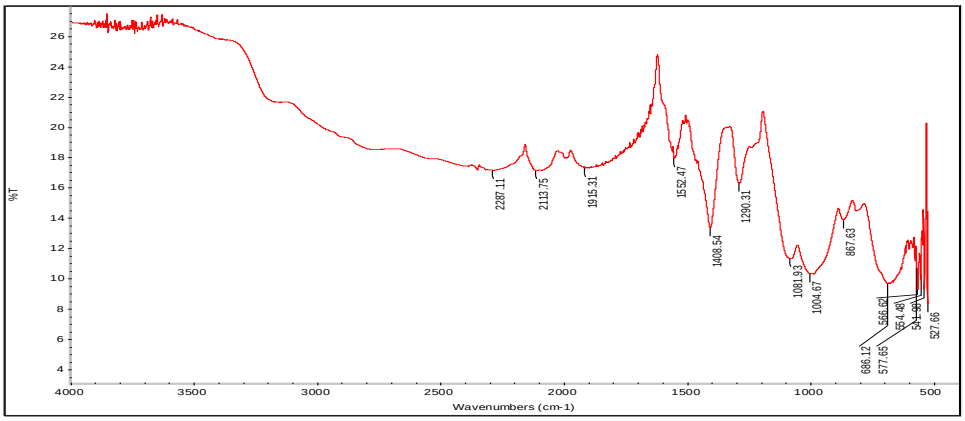

Figure 7: FTIR spectrum of PANI- SbO_2 composite

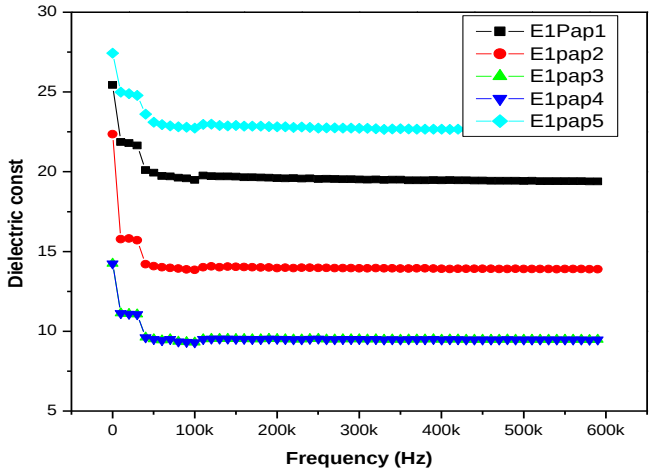

Figure 8: a c conductivity of PANI- InO composite