Conductivity study of Polyaniline- PbO Composites

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Abstract
Polyaniline-lead oxide (PANI- PbO) composites with 10, 20, 30, 40 and 50 wt% of PbO were prepared by insitu polymerization method. Variation in the oxide composition with polymer matrix maintained to enhance its conductivity. The structural, morphological study and chemical bonding of prepared composite materials were carried out by X-ray diffraction (XRD), Scanning Electron Micrograph (SEM) and Infrared (IR) spectroscopy respectively. The conductivity behavior investigated in the frequency range 10²–10⁷ Hz at room temperature is reported in this paper. The PbO particles in the matrix of PANI has a greater influence on the observed conductivity

Keywords: Synthesis and characterization, Polyaniline-PbO composite and Conductivity

INTRODUCTION
Synthesis of polymer composites of core shell metallic particle-polymer have attracted attention of researchers in the recent years because of its unique electrical properties [1-2] particularly, 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 broaden their applicability in the fields of catalysis, electronics and optics [3]. Many efforts have been made to successfully prepare polymer composites with metal oxide (MO) core shell 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. The resulting conducting polymers with controllable hollow structure have shown promising prospective applications [6]. The challenge for the preparation of the MO-Polymer is to generate the polymer coating uniformly and completely on the surface of the metal

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oxide core by a polymerization reaction in a solution phase. The key issue aims at slowing down the rate of uniform polymerization and controlling the polymerization on the surface of the core. 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 method of synthesis of PANI and PbO dispersed PANI composite materials through oxidative polymerization of aniline. The conductivity of the prepared PANI composite material is well studied to understand its electrical behavior.

EXPERIMENTAL

Materials and Methods

Chemicals used in the preparation of PANI and its composites were Ammonium persulphate (NH₄)₂S₂O₈, Hydrochloric acid (HCl), aniline and lead oxide (PbO) are of AR grade. The synthesis process was carried out using double distilled water. Polyaniline and its composite materials were prepared as described.

Synthesis of Polyaniline- PbO Composites

Aniline was dissolved in 1M HCl to form aniline hydrochloride. Lead oxide 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 lead oxide suspended in the solution. 0.1M of ammonium persulphate [(NH₄)₂S₂O₈] as an oxidant was added slowly to the reaction mixture with continuous stirring for 4-6 hours at 0-5°C. The precipitated powder recover was vacuum-filtered and washed with deionized water. Finally, the resultant precipitate was dried in an oven for 24 hours to achieve a constant weight. Similarly five different PANI- PbO composites with different weight percentage of PbO (10, 20, 30, 40 and 50) in PANI have been synthesized. Pure ,polyaniline was prepared by chemical oxidation of aniline without adding lead oxide [9]

Characterization Technique:

The synthesized samples were characterized using XRD, FTIR and SEM. The X-ray diffraction patterns of the prepared composites were obtained by advance X-ray diffractometer (Bruker AXS D8) using CuKα radiation (λ=1.5418 Å). The XRD pattern was recorded in 20 angle ranging from 10° to 80°. The FTIR spectra of the composites were recorded at room temperature using ThermoNicolet, Avatar 370 spectrometer in KBr medium. The surface morphology of polyaniline, PbO particles and PANI/PbO composites were studied by using Joel model JSM-6390 LV scanning electron microscope (SEM). AC/DC conducting properties were studied using (instrument)
RESULTS AND DISCUSSION

X-ray diffraction

Figure 1 shows X-ray diffraction pattern of Polyaniline. The analysis of X-ray diffraction pattern of polyaniline suggests that it has amorphous nature with a broad peak centered on $2\theta \approx 26.40^\circ$. Figure 2 shows the X-ray diffraction pattern of polyaniline – PbO composite with 50 wt% of PbO in polyaniline. It is seen from figure that, the peak of PbO indicates the crystalline nature of the composite. By comparing the XRD pattern of PANI-PbO composite with that of PANI, the additional peaks in Figure 2 corresponds to the dispersion of PbO in PANI.

![Figure 1](image1)

**Figure 1:** X-ray diffraction pattern of Polyaniline

![Figure 2](image2)

**Figure 2:** X-ray diffraction pattern of Polyaniline – PbO composite
Figure 3 shows the IR spectra of polyaniline. The analysis of the spectra shows intensity peaks at 2322.42 cm$^{-1}$, 2089.99 cm$^{-1}$, 1789.70 cm$^{-1}$, 1537.79 cm$^{-1}$, 1403.15 cm$^{-1}$, 1260.34 cm$^{-1}$, 925.41 cm$^{-1}$, 666.13 cm$^{-1}$, 562.42 cm$^{-1}$, 576.66 cm$^{-1}$, 552.64 cm$^{-1}$, 545.81 cm$^{-1}$ and 538.62 cm$^{-1}$. The spectra show the presence of four intense bands at 1537.79 cm$^{-1}$, 1403.15 cm$^{-1}$ and 1260.34 cm$^{-1}$. The peak at 2322.42 cm$^{-1}$ is due to secondary amine. The intense peaks at 1537.79 cm$^{-1}$ and 1260.34 cm$^{-1}$ may be attributed due to the presence of quinoid (N=Q=N) and benzenoid (N=B=N) ring stretching. The peak at 2089.99 cm$^{-1}$ is due to CH$_2$ symmetric stretching. The intense peaks at 1537.79 cm$^{-1}$ and 1260.34 cm$^{-1}$ may be attributed due to the presence of quinoid (N=Q=N) and benzenoid (N=B=N) ring stretching.

Figure 4 shows the IR transmittance with wave number (cm$^{-1}$). The analysis of the spectra reveals the presence of PbO metal oxide with intensity peaks at 3741.92 cm$^{-1}$, 2545.52 cm$^{-1}$, 2160.65 cm$^{-1}$, 709.10 cm$^{-1}$, 566.65 cm$^{-1}$, 554.98 cm$^{-1}$, 547.17 cm$^{-1}$, 543.44 cm$^{-1}$, 539.68 cm$^{-1}$, 535.63 cm$^{-1}$ and 532.04 cm$^{-1}$. The IR spectrum of polyaniline–PbO with 50 wt% of PbO is shown in Figure 5. The characteristic stretching frequencies are observed at 2162.11 cm$^{-1}$, 1566.75 cm$^{-1}$, 1483 cm$^{-1}$, 1290.36 cm$^{-1}$, 1024.28 cm$^{-1}$, 799.84 cm$^{-1}$, 624.28 cm$^{-1}$, 574.22 cm$^{-1}$, 566.34 cm$^{-1}$, 547.48 cm$^{-1}$, 543.28 cm$^{-1}$, 539.60 cm$^{-1}$, 535.42 cm$^{-1}$, 531.90 cm$^{-1}$ and 528.09 cm$^{-1}$ which are due to the presence of metal oxygen stretching frequencies. The IR spectra of polyaniline – PbO composite (50 wt % of PbO in PANI). The characteristic stretching frequencies are observed at 2162.11 cm$^{-1}$, 1566.75 cm$^{-1}$, 1483 cm$^{-1}$, 1290.36 cm$^{-1}$, 1024.28 cm$^{-1}$, 799.84 cm$^{-1}$, 624.28 cm$^{-1}$, 566.34 cm$^{-1}$, 547.48 cm$^{-1}$, 543.28 cm$^{-1}$, 539.60 cm$^{-1}$, 535.42 cm$^{-1}$, 531.90 cm$^{-1}$ and 528.09 cm$^{-1}$. By comparing the IR spectra of polyaniline and polyaniline – PbO composite, it is observed that in the composite the characteristic stretching frequencies are shifted toward higher frequency side which may be attributed due to the Vander walls kind of interaction between PbO and polyaniline chain.

![Figure 3: FTIR spectra of Polyaniline.](image-url)
Figure 4: FTIR spectra of Pure PbO

Figure 5: FTIR spectra of Polyaniline – PbO composite (50 wt %)

Scanning Electron Micrograph of conducting polyaniline synthesized by chemical oxidative method is shown in Figure 6. It can be clearly seen that the micrograph of polyaniline is branched and homogeneous. Since Hydrochloric acid is used as protonic...
acid in the preparation of polyaniline, the presence of microcrystalline structure can be seen in the micrograph.

The SEM micrograph of pure PbO is shown in Figure 7. Circular or plate like morphology is observed with overlapping of most of the grains. The SEM micrograph of polyaniline / PbO composite with 50 wt% of PbO in polyaniline is shown in Figure 8. The SEM image reveals the presence of PbO grains distributed uniformly throughout the composite. A small variation in the grains dimensions of PbO dispersed in polyaniline has been observed. The contrast in the image is due to the difference in scattering from different surface areas as a result of geometrical differences between polyaniline and PbO.

Figure 6: SEM Micrograph of Polyaniline

Figure 7: SEM Micrograph of PbO
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Figure 8: SEM Micrograph of Polyaniline – PbO composite

Figure 9 shows the variation of dc conductivity as a function of temperature for PbO in polyaniline. It is observed that the value of dc conductivity of these composites increases exponentially with temperature. It remains nearly constant up to 100°C and there after it increases exponentially. The conductivity behavior is the characteristic of amorphous materials.

The saturation in the values of conductivity till 40 wt % of PbO is due to partial blocking of charge carriers. Further, gradual increase in conductivity is noticed due to the variation in distribution of PbO particles in PANI. The exponential increase in the values of conductivity for 50 wt % of PbO in PANI is due to the extended chain length of polyaniline due to which the charge carriers can hop between the favorable localized sites.

Figure 9: Variation of DC conductivity of PANI-PbO composites with temperatures)
CONCLUSION
Insitu polymerization was used for preparation of conducting PANI-PbO composites. Structural changes of pure PANI and pure metal oxide is taken place due to the presence of oxide material in the PANI is observed by XRD pattern. The morphology and bonding changes is observed in composite material compared to pure PANI. The results of conductivity show a strong dependence on the wt. % of PbO in PANI.

REFERENCES