

Studies on Electrical and Sensing Properties of Polyaniline / Iron Oxide (Γ -Fe₂O₃) Nanocomposites.

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ABSTRACT

Polyaniline iron oxide nanocomposites were prepared by in-situ polymerization method These nanocomposites were characterized by employing Fourier Transform Infrared (FTIR), Scanning Electron Microscope (SEM), Thermal study by (TGA). The dc conductivity of prepared nanocomposites was measured as a function of temperature which shows the strong interaction between Polyaniline and iron oxide nanoparticles and exhibits semiconducting behavior. Finally, the sensing properties of these nanocomposites are also studied at room temperature.

Keywords: Nanocomposites, FTIR, TGA, SEM, Conductivity,

I. INTRODUCTION

Conducting Polymer inorganic nanocomposites attracted both fundamental and practical interest because of their different chemical, biological and physical properties and application in high density magnetic recording, catalysis, magnetic resonance imaging, energy conversion etc.[1-4]. Among all conducting polymer Polyaniline is one of the most promising conducting polymers due to its ease preparation, good environmental stability, better electronic properties, low cost, low density and its applications in electrochromic display, electro catalysis, rechargeable batteries, sensors and biosensors [5-14]. Polymer nanocomposites are also known as nanostructure materials because it improve the performance properties of the polymer by doping inorganic nanoparticles[15]. Among all metal oxide nanoparticles, iron oxides have attracted technological importance and potential applications in catalysis such as magnetic storage devices, ferrofluids and other uses [16-17]. In the present work, the Polyaniline nanocomposites were synthesized by in-situ polymerization method, the synthesized nanocomposite were characterized by FT-IR, SEM, Thermal Studies like TGA and also we study the DC-conductivity and sensing properties of these prepared nanocomposites.

II. EXPERIMENTAL

2.1 SYNTHESIS OF IRON OXIDE NANOPARTICLES.

The iron oxide nanoparticles were synthesized by self-propagating low temperature combustion method, employing iron oxalate as precursor. The Precursor is prepared by dissolving equimolar quantity of ferrous ammonium sulphate and oxalic

acid in distilled water. This solution stirred for ½ hour on magnetic stirrer. Yellow precipitate of iron oxalate hydrate obtained is filtered and washed with distilled water. The prepared iron Oxalate was mixed with Polyethylene glycol (PEG) in the weight ratio 1:5. The resultant compound was placed in a crucible and heated by using electrical heater. It was observed that initially PEG is melted, then frothed and finally ignited to give iron oxide as a residue, the prepared compound was then calcinated for 2 hours to remove impurities. Finally pure iron oxide nanoparticles were obtained.

2.1 PREPARATION OF PANI/IRON OXIDE NANOCOMPOSITES.

Synthesis of the PANI- iron oxide nanocomposites was carried out by in-situ polymerization method. Aniline (0.1 M) was mixed in 1 M HCl and stirred for 15 min to form aniline hydrochloride. Iron oxide nanoparticles were added in the mass fraction to the above solution with vigorous stirring in order to keep the iron oxide homogeneously suspended in the solution. To this solution, 0.1 M of ammonium persulphate, which acts as an oxidizer was slowly added drop-wise with continuous stirring at 5°C for 4 h to completely polymerize. The precipitate was filtered, washed with deionized water, acetone, and finally dried in an oven for 24 h to achieve a constant mass. In this way, PANI- iron oxide nanocomposites containing various weight percentage of iron oxide (10 %, 20 %, 30 %, 40 %, and 50 %) in PANI were synthesized.

III. CHARACTERIZATION.

Fourier transformed infrared spectra of these nanocomposite were recorded on Thermo Fisher

ATR Nicolet model using diamond (iS5) in the range 4000-400 cm^{-1} . The morphology of the nano iron oxide and nanocomposites in the form of powder was investigated using SEM Model-EVO-18 Special Edison, Zein Germany. Thermal studies were carried out employing STA PT1600 Thermal Analyzer from

Linseis under nitrogen atmosphere with a heating rate of 10 $^{\circ}\text{C}/\text{minute}$ at a flow rate of 100 ml/min. Electrical properties of these nanocomposites are studied by using Keithley 6514 electrometer and sensing properties of these nanocomposites were studied using laboratory set up.

IV. RESULT AND DISCUSSION 4.1 FTIR ANALYSIS

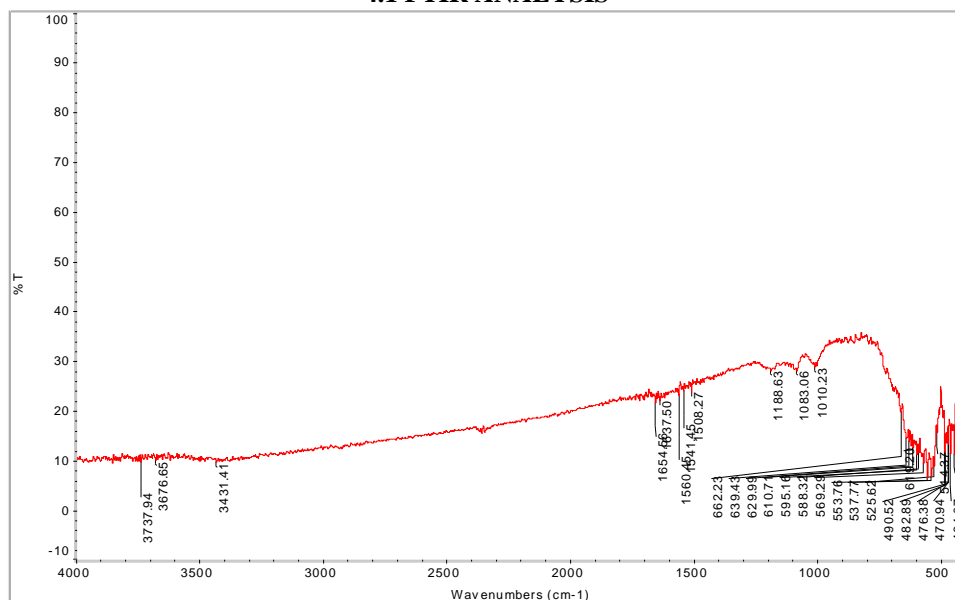


Fig (a) pure iron oxide.

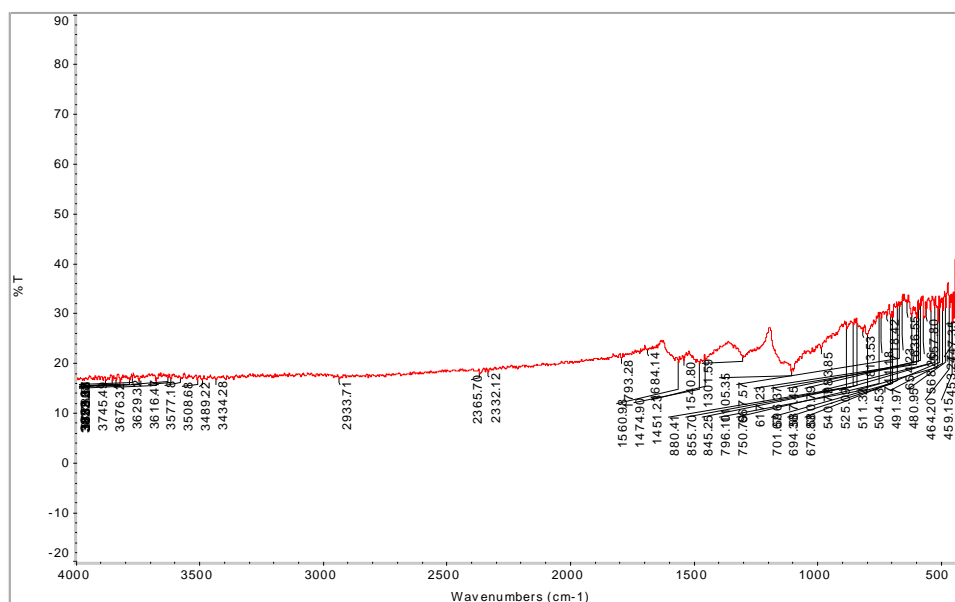


Fig (b) pani/iron oxide nanocomposite..

Fig.4.1 (a) & (b) shows the FTIR spectra iron oxide and pani/iron oxide nannocomposites.

Fig 4.1(a) shows FTIR spectra of pure iron oxide nanoparticles the peaks at 3737 to 3431 cm^{-1} is due to -OH stretching because of absorption of water molecules, the characteristic stretching frequencies at 1654 cm^{-1} is due to -N-H stretching, the peaks at

1083 to 1010 cm^{-1} is due to -C-H bending the peaks from 657 to 424 cm^{-1} is due to metallic stretching. Fig 4.1(b) shows the IR spectra of Pani/iron oxide nanocomposite the characteristic stretching frequency was observed at 3745 to 3434 cm^{-1} is due to -O-H

stretching ,the peaks at 2933 to 2332 cm^{-1} is due to C-H stretch, the peaks at 1793 and 1684 cm^{-1} is due to C=O stretch ,the peaks 1560 to 1451 cm^{-1} is due to -C-H bending ,the peaks at 880 to 676 cm^{-1} is due to Alkene=C-H bending and the peaks at 540 to 429 cm^{-1} is due to metallic stretch .By comparing IR spectra

of pani and pani/iron oxide nanocomposite, it is observed that in the composite the characteristic stretching frequencies are shifted towards higher frequencies side which may be due to Vander walls kind of interaction between iron oxide and pani chain.

4.2 SCANNING ELECTRON MICROSCOPY

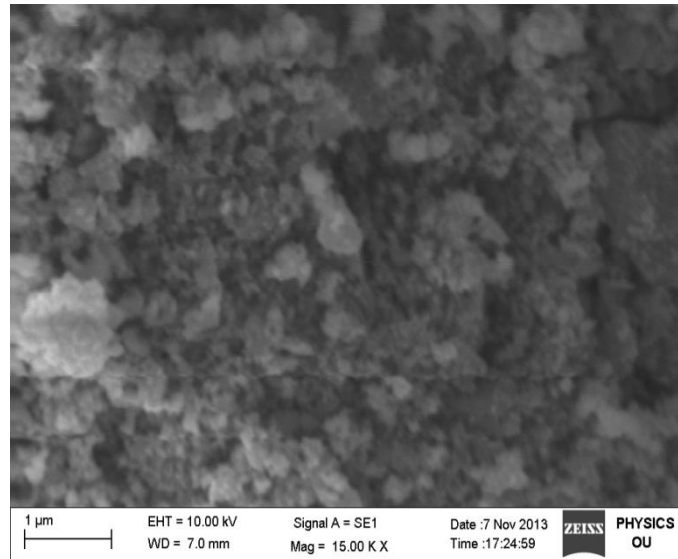


Fig (a)Pure Iron oxide

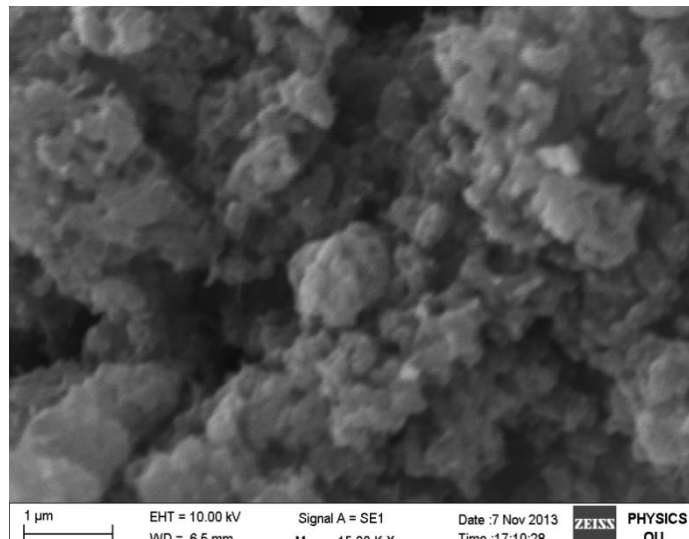


Fig (b)Pani/iron oxide nanocomposite.

Figure 4.2 Scanning electronic micrograph (SEM) image of Pure Iron oxide and Pani/Iron oxide nanocomposite.

Figure 4.2(a) & (b) shows the micrograph of iron oxide and Pani/Iron oxide nanocomposites. It is seen clearly from the SEM micrograph of iron oxide, it has a cluster spherical shaped particles. Figure 4.2.(b) Shows the micrograph of iron oxide

nanocomposite it is observed that the iron oxide nanocomposite are cluster like structure. The presence of such sharp crystal of iron oxide nanoparticles has a strong influence on various electrical parameters of these nanocomposites.

4.3 Thermal analysis

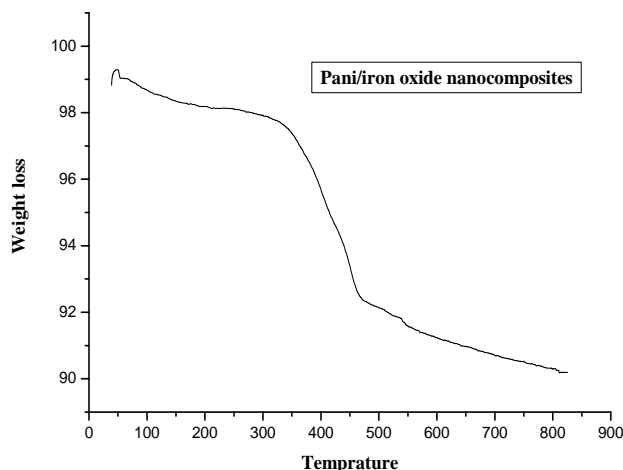


Figure 4.3 shows the TGA graph of 50wt% Pani/Iron oxide nanocomposite

Figure 3 shows the thermo graph of Pani/Iron oxide nanocomposites. the thermograph shows two weight losses as seen in fig 3. The first weight loss

corresponds to evaporation of water molecules from the prepared nanocomposites. The second weight loss is due to the decomposition of organic moiety.

4.4 DC CONDUCTIVITY STUDIES.

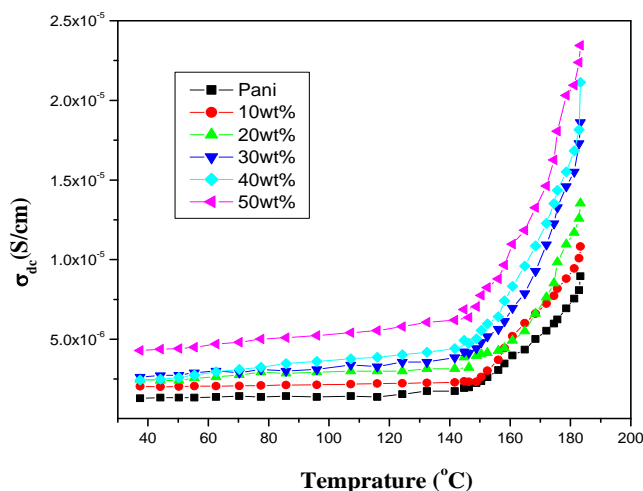


Fig.4 shows the dc conductivity of pani/iron oxide nanocomposites..

Fig 4 shows the DC conductivity of Polyaniline and Pani/iron oxide nanocomposite of different weight percentage as a function of temperature range from 30 to 180^oc. The DC conductivity remains almost constant up to 120^oc and thereafter it increases up to 180^oc, which is the characteristic behavior of semiconducting materials. The conductivity increases with an increase in temperatures due to the flow of ions from one localized state to another and it is also suggested here that the thermal curling effects of the

chain alignment of the Polyaniline leads to the increase in conjugation length and that brings about the increase of conductivity.

The conductivity varies directly with the temperature obeying an expression of the following form:

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

Where: σ is the conductivity, T is the temperature and σ_0 is the conductivity at characteristic temperature T_0 .

4.5 Sensing studies

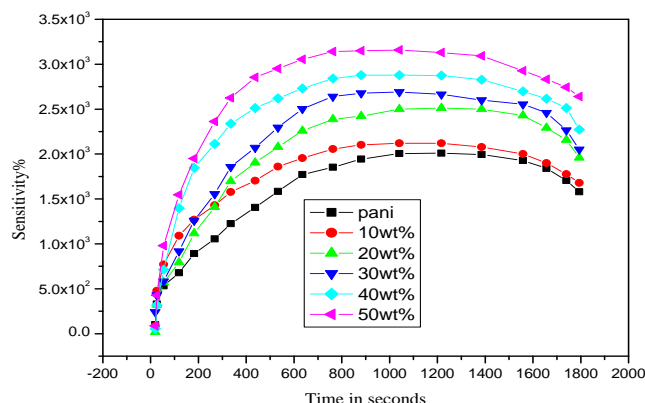


Fig.5. Variation of sensitivity against LPG

Fig (5). Shows sensitivity vs time for Polyaniline /Iron oxide nanocomposites it is seen that the Pani/iron oxide nanocomposites shows better sensitivity to gas vapor. Compared to pure Polyaniline and other nanocomposites such as (10,20,30,40 wt%) 50wt% nanocomposites shows high sensitivity due to the strong interaction between the Polyaniline and iron oxide nanoparticles. In the case of pure Polyaniline and 10wt% of nanocomposites sensitivity is very low because of lower surface area.

V. CONCLUSIONS:

Polyaniline Iron oxide nanocomposites were prepared by in-situ polymerization method, the FTIR spectra confirms the formation of pani/iron oxide nanocomposites. The SEM image shows the presence of iron oxide nanoparticles which are uniformly distributed throughout the nanocomposite sample. The presence of iron oxide nanoparticle in Pani nanocomposites influences the electrical and Sensing parameter such as conductivity and sensitivity of these nanocomposites. The DC conductivity studies show the prepared nanocomposites exhibits typical semiconductor behavior. It is concluded that this Pani iron oxide nanocomposites is one of the promising materials for the potential applications.

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