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# Synthesis, conductivity and sensitivity studies of Polyaniline-Iron oxide nanocomposites

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**ABSTRACT**
Polyaniline and polyaniline/Iron oxide nanocomposites were synthesized by in-situ polymerization technique. The formation of PANI/Iron oxide nanocomposites with regards to the structural properties of the materials were investigated by X-Ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) techniques. The crystallite size of iron oxide was found to be 36 nm as indicated by XRD along surface normal direction. The frequency dependent conductivity of these nanocomposites has been studied. Large variations in conductivity were observed, this could be due to lattice polarization around the charge localized state and due to variation of distribution of Iron oxide in polymer matrix. Compare to all the composites 50 wt% shows highest sensitivity. The results obtained for these composites are of scientific and technological interest for variety of applications such as in batteries, microelectronics displays, antistatic coatings, electromagnetic shielding materials, sensors and actuators. Its good environmental as well as thermal stability and electrical conductivity tunable by appropriate doping make PANI an ideal active material.

***Keywords*** - Polyaniline and Polyaniline, Nanocomposites, X-Ray diffraction, Scanning Electron MicroscopyTransmission Electron Microscopy

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## I. INTRODUCTION

The conducting polymer/inorganic nanoparticles composites show unique physical and chemical properties that have attracted more and more attention, as they combine the merits of conducting polymers and inorganic nanoparticles. These composites can have wide potential applications in diverse areas such as chemistry, physics, electronic, optics, materials, and biomedical science [1–3]. Among conducting polymers, polyaniline (PANI) is an air-stable conducting polymer and has attracted considerable attention because of its moderate electrical conductivity. PANI is unique as its electronic properties can be controlled both by varying the oxidation state of the main chain and by protonation [1]. Recent investigations have revealed that PANI can be an excellent matrix for forming polymer-nonmaterial composites [4–6].

Iron oxide nanoparticles with a uniform size and well dispersion are desirable for many applications in designing ceramic, magnetic, electro-chromic and heterogeneous catalytic materials [7]. One can prepare g-Fe2O3 nanoparticles by various methods like sol–gel [8], surfactant-mediated synthesis [9] and spray-Pyrolysis [10]. Ultrasonic radiation, hydrothermal synthesis, carbonyl method, laser chemical method, Pyrolysis by microwave, precipitation-calcinations, micro emulsion method, and so on [11–17]. However, to the best of our knowledge, most of the reported experimental techniques for the synthesis of nanopowders are still limited in laboratory scale due to some unresolved problems, such as special conditions, tedious processes, complex apparatus, low yield, and high cost [18]. Since they exhibit interesting properties with many applications such as quantum electronic devices, magnetic recording materials, sensors, capacitors, smart windows, toners in photocopying, conducting paints and rechargeable batteries [19–27]. These composites are often prepared by dispersing nonmaterial in a non-conducting polymer matrix [28].

## II. 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 is stirred for 1/2 hour on magnetic stirrer. Yellow precipitate of iron oxalate dehydrates 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 and it was observed that initially PEG is melted, then frothed and finally ignited to give iron oxide as a residue, then the prepared compound was then calcinated for 2 hours to remove impurities. Finally, pure nickel oxide nanoparticles were obtained.

### 2.1 Synthesis of pani/iron oxide nanocomposites.

Synthesis of the Polyaniline–iron oxide nanocomposites were carried out by in-situ polymerization method. Aniline (0.1 M) was mixed in 1MHCl 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, 0.1M of ammonium per sulphate, which acts as an oxidizer was slowly added drop wise with continuous stirring at 5 °C for 4 hours 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, Polyaniline– iron nanocomposites containing various weight percentage of iron oxide (10%, 20%, 30%, 40%, and 50%) wt% in PANI were synthesized.

## III. PREPARATION OF PELLETS

The powders of Polyaniline, Polyaniline/Iron Oxide composites, obtained from synthesis techniques discussed in early sections were crushed and finely ground in agatemortar in the presence of acetone medium. The powder is then pressed to form pellets of 10 mm diameter and thickness varying up to 2 mm by applying pressure of 90 MPa in a hydraulic press. For temperature dependent conductivity and sensor studies, the pellets of Polyaniline and its metal oxide nanocomposites are coated with silver paste on either side of the surfaces to obtain better contacts.

### 3.1. Polyaniline/iron nanocomposites film

Polyvinyl alcohol (PVA) with molecular weight approx. 1,25,000 was obtained commercially with AR grade, and Polyaniline/Iron oxide nanocomposites was synthesized by In-situ polymerization method. Powdered PVA of about 2.5 g was dissolved in 50 ml of double distilled

water by stirring. The solution was then warmed up to 333 K and thoroughly stirred, using a magnetic stirrer, for about 1 h until the polymer became completely soluble. A thick film of the sample was prepared by solution casting method in the following manner, the synthesizedPolyaniline/Iron nanocomposites powder was dissolved in PVA solution and this was sonicated for 15–20 min. The sonicated solution was stirred for 1/2 hour. Then the paste of PVA/ Polyaniline/Iron oxide nanocomposites was formed. Then known volume of viscous PVA/ Polyaniline/Iron oxide nanocomposites solution was poured onto a leveled clean glass plate and left to dry at room temperature for about 48 h. The dried films were peeled off from the glass plate and cut into suitable pieces for characterization and applications.

In this paper, the author has reported Pani/Iron nanocomposites, which were synthesized by in-situpolymerizationmethod.The presence of iron oxide nanoparticle in Pani nano composites influences the electrical and sensing parameter such as conductivity and sensitivity of these nanocomposites.

## IV. RESULT AND DISCUSSION

### 4.1. X-ray diffraction

The X-ray diffraction patterns of the samples in this present study are obtained on Philips X-ray diffractometer using CuKα radiation (λ =1.541 Å). The diffractograms were recorded in terms of 2θ in the range 10°- 160° with a scanning rate of 2° per minute. Figure 1 (a)shows X-ray diffraction pattern of oxide nanoparticles and one can notice the multiple peaks with reasonable intensities, this indicates the polycrystalline nature of the iron oxide particles, along surface normal direction. Using Scherrer formula, the average crystallite size of iron oxide particles along surface normal direction was found to be 36 nm (check one more time).

However, after the addition of iron oxide nanoparticles into PANI during polymerization, high-intensity peaks were masked in the composite due to the overwhelming amorphous nature of PANI as shown in Figure 1 (b). Once again, the appearance of multiple peaks with reasonable intensity indicates the polycrystalline nature of the Iron oxide-PANI based composite. The sharp peaks observed are found to be due to the crystalline nature of iron oxide nanoparticles. This also confirms the uniform molecular level dispersion of iron oxide in PANI, which is responsible for a decrease in the

crystallinity of iron oxide particles upon dispersion into the PANI matrix.

### 4.2. Scanning electron microscopy

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. Shows the SEM micrographs of the synthesized Iron oxide the particles these are spherical in shape. Figure 2 (a, b) shows that Scanning Electronic Micrograph (SEM) image of 50 wt % of Polyaniline/Iron Oxide nanocomposite. The particles are highly agglomerated granular in shape is found. The grains are well interconnected with each other.

### 4.3. Transmission electron microscopy

Figure 4.3 shows TEM micrographof Iron oxide nanoparticles. The shape of the particles is spherical and the average diameter is 200 nm. This may be attributed to the formation of intra residue hydrogen bonds between the molecules, resulting in a zig–zag-shapedStructure

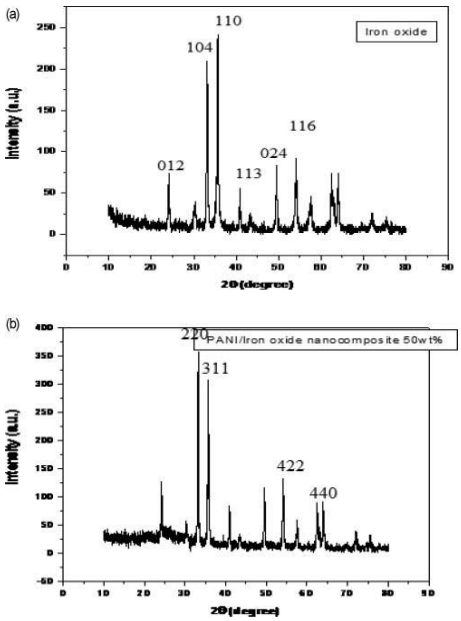



**Figure 1.** (a) & (b) shows X-ray diffraction pattern of Iron oxide nanoparticles and Polyaniline /iron oxide nanocomposite.

### 4.4. AC conductivity Pani and Pani/iron oxide nanocomposites

Figure. 4 Shows the variation of ac conductivity as a function of frequency for polyaniline– iron oxide nanocomposites for different wt %. It is observed that in all the cases, sac remains constant up to $3.5x10^5$ Hz, there after conductivity increases for all the nanocomposites.

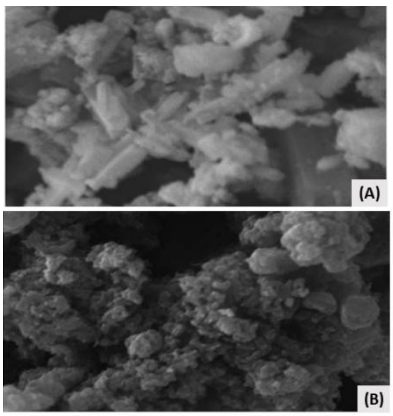
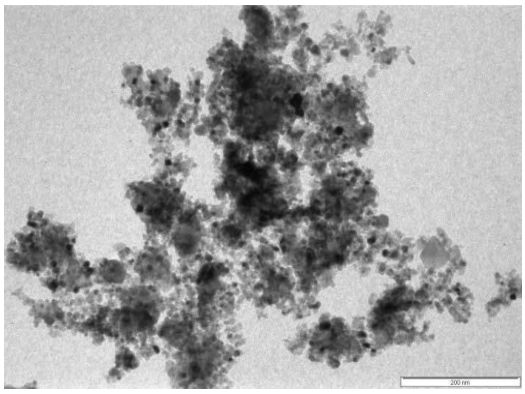



**Figure 2. (A)** Shows SEM Image of Iron oxide nanoparticles. (B) Shows SEM Image of PANI/ Iron Oxide nanocomposites.

### 4.5. Sensing studies

Figure 5 shows sensitivity vs. time for Polyaniline /Iron Oxide nanocomposites, it is observed that the sensitivity increases with increase in the weight percentage of Iron oxide nanoparticles. and in the case of Pani Sensitivity is low because of lower surface area. Among all the nanocomposites, 50 wt% shows maximum sensitivity when compared to pure polyaniline and other nanocomposites and this is due to the reaction between metal nano particle and LPG. In the case of semiconducting metal oxide –based gas sensor.



Sensitivity depends on the chemisorbed oxygen ions, oxygen vacancies and the interstitial ions. The interaction of gas molecules with the surface of pellet cuses the transfer of electron between the semiconducting surfaces of the pellet of Iron Oxide

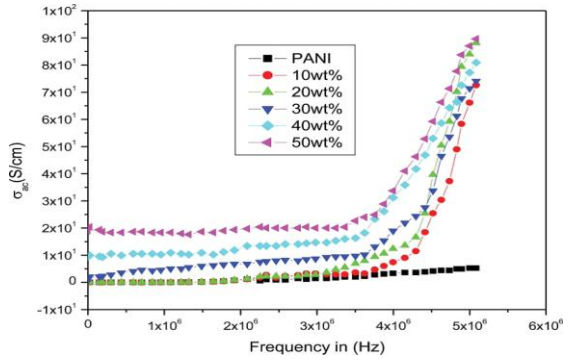



**Figure 4**. Shows sac conductivity of PANI and PANI /Iron Oxide composites against applied frequency

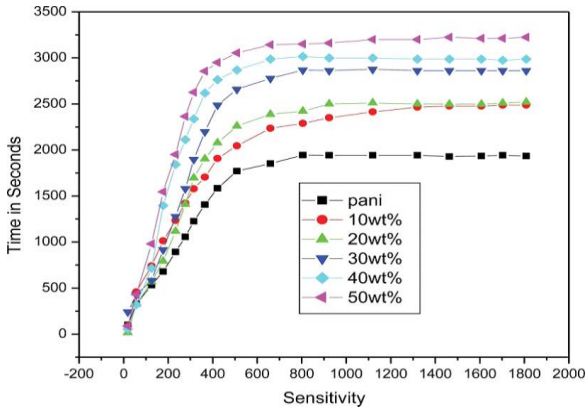



**Figure 5.** Shows Sensitivity Vs Time for PANI thin films in seconds.

## V. CONCLUSION

In this study, Polyaniline/Iron Oxide nanocomposites were successfully synthesized by insitu polymerization method. The XRD, SEM and TEM results shows the formation of nanocomposites and indicates an interaction between Polyaniline and Iron oxide nanoparticles.

The electrical conductivity in these nanocomposites shows a strong dependence on content of iron oxide nanoparticles in Polyaniline/Iron oxide nanocomposites. Among the entire composites 50 wt% shows highest Sensitivity. Hence these nanocomposites are found to be promising material for potential applications.

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