

Synthesis, Characterization and Electrical Properties of Polyaniline/Nickel Oxide Nanocomposites

¹Sharanabasamma M Ambalgi, Hajeebaba K Inamdar, Manjula V T, Sannakki Nagaraja, Shrishail G Hogade and ^{1*}Basavaraja Sannakki

Department of Post graduate and Research in Physics Gulbarga University Kalaburagi.585106 Karnataka, India.

Email id: shakti6585@gmail.com

Abstract: Super paramagnetic Metal oxide nanoparticles with appropriate surface chemistry have been widely used experimentally for numerous in vivo applications such as magnetic resonance imaging contrast enhancement, tissue repair, immunoassay, detoxification of biological fluids, hyperthermia, drug delivery and in cell separation, etc. In the present investigation, Polyaniline/Nickel oxide nanocomposites with various weight percentage of Nickel oxide (10%, 20%, 30, 40% and 50%) were synthesized by in-situ polymerization method. The prepared nanocomposites were characterized by X-Ray diffraction (XRD), Scanning Electron Microscopy (SEM). The DC conductivity of the samples was measured as a function of temperature in the range 30-190°C and it was found that the increasing the concentration of Nickel nanoparticles increases the conductivity.

Keywords: NiO₂, combustion method, Scanning Electron Microscope, Electrical properties

1 INTRODUCTION

Nanoparticles of transition metal oxides have been investigated by several workers in the last few years. Besides their structural aspects, magnetic properties of the oxide nanoparticles are of particular interest [1]. Thus, it would be of value to know if the nanoparticles of antiferromagnetic oxides generally show evidence for ferromagnetic interaction at low temperatures, a behavior that has been reported by a few workers [2-3]. Nanoscale oxide particles of transition metals are gaining continuous importance for various applications such as catalysts, passive electronic components and ceramic. Due to their small size, nanoparticles exhibit novel material properties that are significantly different from those of their bulk counterparts [4-6]. Chemically synthesized magnetic nanoparticles have drawn much attention due to the unique magnetic properties associated to their size magnitude and distribution uniformity. Among these particles, magnetite nanoparticles have been widely studied with biomedical applications in view, such as magnetic resonance imaging for clinical diagnosis, magnetic drug targeting, hyperthermia anti-cancer strategy, and enzyme immobilization [7].

Nickel oxide is one of the important metal oxide prepared by different methods like, combustion, sol-gel, Hydrothermal and solvent thermal technique. Among these, combustion technique has received considerable attention, based on their interesting properties and applications in gas sensor device, Magnetic storage devices, catalysis and ferrofluids [8-9].

Conductive polymers had been the topic of the large number of investigations during last decades because of their unique properties such as mechanical strength, electrical conductivity, corrosion, stability and possibility of both oxidative and electrochemical synthesis. Hence PANI is useful in wide area of application such as solar energy conversion, rechargeable batteries, electrochromic displays, electrochemical sensors, capacitors and active corrosion protector [10-12]. Due to ease of synthesis, processing environmental stability and low synthetic cost, so polyaniline is probably the most important industrial conducting polymer today [13]. The use of conducting polymers for photovoltaic applications has been reported [14].

In the present paper nickel oxide nanoparticles were synthesized by combustion method. The synthesized Polyaniline/Nickel oxide is characterized by X-ray diffraction (XRD), Scanning electron microscopy (SEM) and their Electrical properties are also studied.

2 EXPERIMENTAL

2.1 CHEMICAL SYNTHESIS OF POLYANILINE.

The synthesis was based on mixing aqueous solution of aniline hydrochloride and ammonium persulphate at room temperature, followed by the separation of PANI hydrochloride precipitate by filtration and drying. Aniline hydrochloride (equi molar volume of aniline and hydrochloride acid) was dissolved in distilled water in a volumetric flask to 100 ml of solution. Ammonium persulphate (0.25M) was dissolved in water also to 100ml of solution. Both solutions were kept for 1 hour at room temperature, then mixed in a beaker, stirred with a mechanical stirrer and left at rest to polymerize. Next day, the PANI precipitate was collected on a filter paper, washed with 0.2 M HCL and similarly with acetone. Polyaniline hydrochloride powder was dried in air and then in vacuum at 60°C for 24 hours. Polyaniline prepared under these reaction and processing conditions are further referred to as "standard" samples.

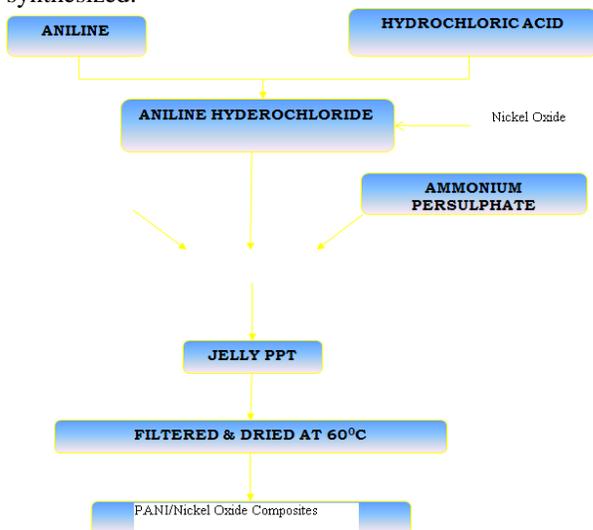
2.2 SYNTHESIS OF NICKEL OXIDE NANOPARTICLES

The nickel oxide nanoparticles were synthesized by self-propagating low temperature combustion method, employing nickel oxalate as precursor. Precursor is prepared by dissolving equimolar quantity of Nickel chloride and oxalic acid in distilled water. This solution is stirred for ½ hour on magnetic stirrer. Light green precipitate of nickel oxalate dehydrate so obtained is filtered and washed with distilled water. The prepared Nickel Oxalate was mixed with Polyethylene glycol (PEG) in the weight ratio 1:5. The resultant compound was

placed in a crucible and heated in air by using electrical heater and it was observed that initially PEG is melted, then frothed and finally ignited to give nickel oxide as a residue, then the prepared compound was then calcinated for 2 hours to remove impurities. Finally pure nickel oxide nanoparticles were obtained.

2.2 SYNTHESIS OF POLYANILINE/NICKEL OXIDE NANOCOMPOSITES

Synthesis of the Polyaniline– nickel oxide nanocomposites was carried out by in-situ polymerization method. Aniline (0.1 M) was mixed in 1M HCl and stirred for 15 min to form aniline hydrochloride. Nickel oxide nanoparticles were added in the mass fraction to the above solution with vigorous stirring in order to keep the nickel oxide homogeneously suspended in the solution. To this solution, 0.1M of ammonium persulphate, 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 then washed with deionized water, Acetone and finally dried in an hot air oven for 24 hour to achieve a constant mass. In these way, Polyaniline–Nickel oxide nanocomposites containing various weight percentage of Nickel oxide (10%, 20%, 30%, 40% and 50 %) wt% in PANI were synthesized.



Flow chart for preparation of Polyaniline/ NiO_2 Nanocomposite

3 Characterization

X-ray diffraction studies were performed using Philips X-ray diffractometer with CuK_{α} as the radiation source. The morphology of the nano Nickel oxide and nanocomposites in the form of powder was investigated using SEM Model-EVO-18 Special Edison, Zein Germany. DC conductivity of these nanocomposites are also studied by using Keithley 6514 electrometer.

4 Result and discussion

4.1 XRD Analysis

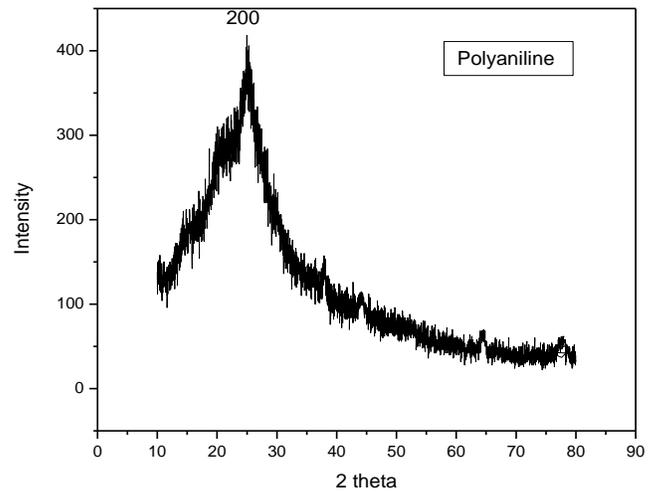
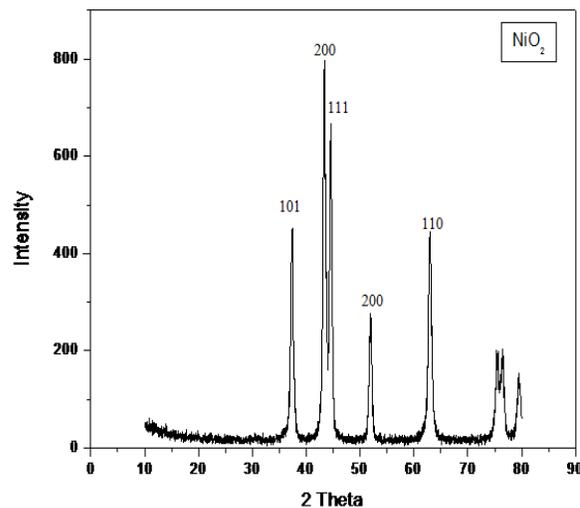


Figure 4.1 (a) X-ray diffraction pattern of Polyaniline

Figure 4.1 (a) shows X-ray diffraction pattern of Polyaniline. A broad peak centered at $2\theta = 25.53^{\circ}$ may be assigned to the scattering from the polyaniline chains at interplanar spacing which clearly implies the amorphous nature of polyaniline and it corresponds to (200) diffraction planes of pure Polyaniline.

X-ray diffraction pattern of Nickel oxide nanoparticles



4.1(b) XRD of Nickel oxide(NiO_2) Nanoparticles

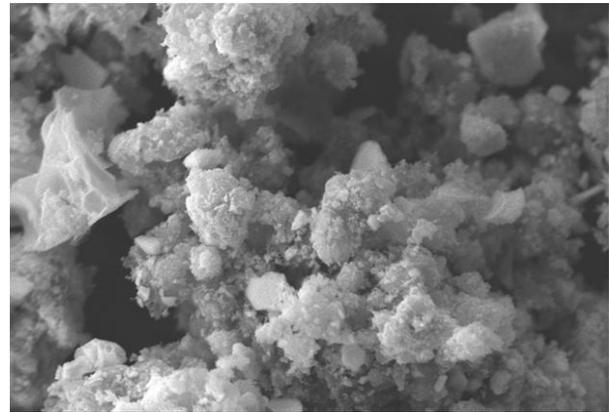
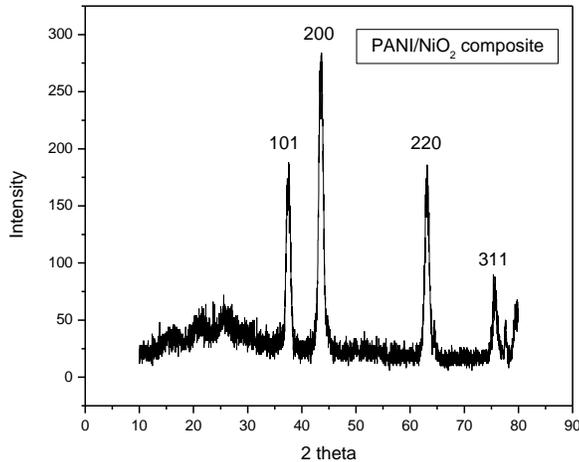


Figure 4.2 (b) Polyaniline/Nickel oxide nanoco

Figure 4.2 (a) shows the SEM image of Pure NiO₂. The particles exhibit a compact arrangement of homogeneous nanoparticles and are roughly spherical in shape. Most of the particles are found to be grouped, so it is difficult to calculate the particle size and of the NiO₂ compound. Hence, the exact size and shape of the NiO₂ particles were calculated by XRD. Fig.4.2 (b) that the Polyaniline/Nickel oxide nanocomposites are cluster like structure and the presence of such sharp crystal of Nickel oxide nanoparticles has a strong influence on various electrical parameters of these nanocomposites.

5 . DC CONDUCTIVITY

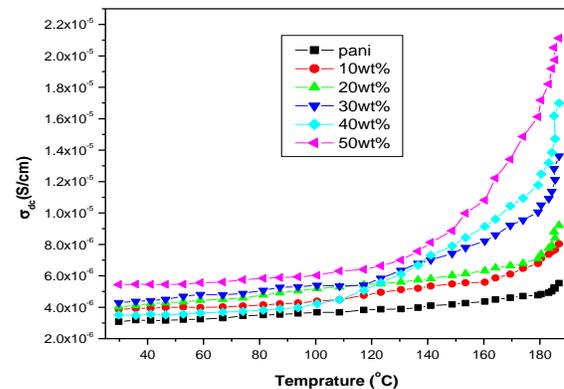


Fig. 5. Temperature versus Dc conductivity graph

The variation of conductivity with temperature for PANI-NiO₂ composites are shown in Fig.(5). The increase in conductivity for the composites may be due to the formation of more number of polarons the change in the conductivity of the composites indicates a change in the doping state of the polymer. Though the composite having 50wt% of PANI/ NiO₂ shows the higher conductivity than pure PANI such enhancement of DC conductivity values can be attributed to the uncoiling of

4.1 (c) Polyaniline /Nickel oxide nanocomposite

Figure 4.1(b) shows a broad peak at $2\theta=43.35^\circ$ which has a sharp and well defined peak and it indicates the good crystallinity. Figure 4.1(c) shows the intensity of diffraction peaks for PAN/NICKEL OXIDE nanocomposite and it is found to be lower than that for pure nickel oxide. The pure nickel oxide nanoparticles peaks are also present in PANI/NICKEL Oxide nanocomposite. The XRD peaks are broad indicating that the particles are in nanometer size range (JCPDS No.04-0835). The amorphous background hump comes from the polyaniline. The average particle size calculated by using Debye Scherrer equation. It is found that the particle size of Nickel Oxide is 35nm.

4.2 Scanning Electron Microscopy

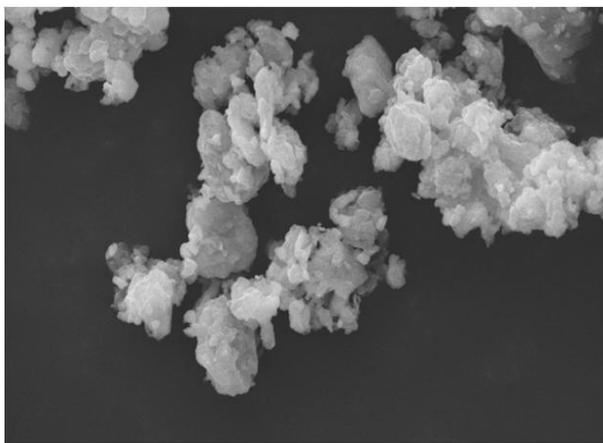


Figure 4.2 (a) shows the SEM image of Pure NiO

polymeric chains due to strong interfacial interaction between NiO₂ with PANI caused by their composition indicating that they have enough binding energy to combine with neighbors grains or molecules. The electrical properties in ferrites can be explained on the basis of exchange of electrons between ions of the same element that are present in more than one valence state distributed randomly over equivalent crystallographic lattice sites. The conductivity varies directly with the temperature, obeying an expression of the following form:

$$\sigma(T) = \sigma_o \exp \left[- \left(\frac{T_o}{T} \right)^{1/4} \right]$$

where σ is the conductivity, T is the temperature and σ_o is the conductivity at characteristic temperature T_o.

Conclusion

The nanocomposites were doped with Nickel oxide in polyaniline at different weight percentage using ammonium persulphate as an oxidant. The prepared nanocomposites were characterized by XRD for particle size studies and SEM to study the morphology of the nanocomposites. The electrical study indicates here was a strong influence on the conductivity of small dopant added to the conducting polymer. It is observed that conductivity increases with the increase in the concentration of Nickel oxide nanoparticles. Among all nanocomposites 50wt% show high conductivity. Hence these nanocomposites are found to be promising material for potential applications.

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