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Structural and Optical Properties of Polypyrrole/NiO Doped Nanocomposites

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ABSTRACT

This study reports the synthesis of polypyrrole/NiO doped nanocomposites (NCs) as a shielding pigment in organic coatings. Polypyrrole/NiO doped NCs were prepared by in-situ chemical oxidative polymerization of pyrrole monomer. Here dopant NiO nanoparticles (NPs) were synthesized by solution combustion method using *Aloe vera* gel as fuel with ammonium per sulphate (APS) as oxidant. Different concentrations of NiO (10-50 wt%) NPs were incorporated into the polypyrrole. The synthesized nanocomposites were characterized by means of powder X-ray diffraction (PXRD), scanning electron microscopy (SEM) and atomic force microscopy (AFM). The size of the NPs were calculated by Scherer method, and found to be around 20 nm. TEM results are in consistent with the XRD results. The morphology of the obtained composites shows the porous and agglomerated particles which are due to large amount of gas evolution during NPs synthesis by solution combustion method and optical properties were studied.

1. Introduction

Conductive polymers such as polyaniline (PANI), polypyrrole (PPy), and their composites are widely uses as sensors [1, 2], energy storage materials, due to the facile synthesis and flexibility in processing [3]. Conducting polymers including polypyrrole (PPy), polyaniline (PANI), PPV, polyethylene di-oxythiophene (PEDOT), and polythiophene (PT) have both electrochemical double layer capacitance and pseudo-capacitance arising from the fast and reversible oxidation and reduction processes related to their p-conjugated polymer chains [1-3]. Among these conducting polymers, PPy is outstanding as a promising electrode material for supercapacitors, because it has various significant advantages such as high specific capacitance, good conductivity, bio-compatibility and outstanding mechanical properties [4]. However, like other electronically conducting polymers, PPy suffers from volumetric shrinkage during discharge process which can lead to decrease in cyclic stability [5-9]. To improve the cyclic stability of the conducting polymer, researchers have recently synthesized various PPy-based composites with hierarchical structured materials to hinder the volume change of the conducting polymer during the charge–discharge processes [10-12]. As it is known to all, transition metal oxides have been massively reported as electrode materials for pseudo-capacitors because of their large capacitance and fast redox kinetics [3]. In this paper we report the synthesis of NiO nanoparticles using *Aloe vera* gel, synthesis of polypyrrole/Ni nanocomposites and their structural, morphological and optical studies in detail.

2. Experimental Methods

2.1 Synthesis of Polypyrrole

The synthesis of polypyrrole (PPy) was based on mixing aqueous solution of pyrrole and ammonium persulphate at ice temperature, followed by the separation of pyrrole precipitate by filtration and drying. An equimolar volume (0.1 M) of pyrrole and hydrochloride acid was dissolved in distilled water in a volumetric flask to obtain 100 mL of solution. Likewise, ammoniumpersulphate (0.2 M) was dissolved in 100 mL water. Both solutions were kept for 1 hour at room temperature and then mixed in a beaker, stirred with a mechanical stirrer and allowed to polymerizing. After a day, the PPy precipitate was collected using filter water, washed with acetone and deionised water repeatedly. The obtained polypyrrole powder was then dried in air in vacuum at 60 °C for 24 hours, then make it as a fine powder.

2.2 Facile Green Fabrication of NiO Nanoparticles

The nickel oxide nanoparticles were synthesized using self-propagating low temperature solution combustion method, employing nickel nitrate ($Ni\ (NO_3)_2.6H_2O$)) as precursor and *Aloe vera* gel as a fuel. 2.14 g of nickel nitrate was taken in 300 mL petri-dish and 10 mL of *Aloe vera* gel was added to the petri dish and kept on a magnetic stirrer for ~10 min. The uniform mixture of both oxidizer as well as the fuel was then introduced into the pre-heated muffle furnace kept at 450 °C. The mixture boils with froth yielding finally a black powder of NiO nanoparticles. The average particle size of the NiO was calculated by Debye-Scherer formula and it was found to be ~20 nm.

2.3 Synthesis of Polypyrrole/Nickel Oxide Nano-Composite

Synthesis of polypyrrole–nickel oxide nano-composites were carried out by in-situ polymerization method. Pyrrole (0.1 M) was mixed in 1 M HCl and stirred for 15 min. 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, 0.2 M of ammonium persulphate, which acts as an oxidizer was slowly added drop-wise with continuous stirring at ice temperature for 4 hours to completely polymerize. The precipitate was filtered, washed with de-ionized water and acetone, and finally dried in an oven for 24 hours to achieve a constant mass. The Polypyrrole -nickel oxide nano-composites were thus obtained containing various weight percentage of nickel oxide (i.e., 10, 20, 30, 40, annd 50%).

3. Results and Discussion

3.1 XRD Analysis

X-Ray Diffraction studies were performed using Shimadzu-7000 diffractometer with Cu as the target (1.54 Å) and nickel as the filter. Fig. 1 shows X-ray diffraction pattern of polypyrrole. A broad peak centered at $2\theta = 25.53°$ may be assigned to the scattering from the pyrrole chains at interplanar spacing which clearly implies the amorphous nature of

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polypyrrole and it corresponds to diffraction planes (200) of pure polypyrrole and PPy/NiO nanocomposites. It was clearly observed from the PXRD graph that pure PPy was in amorphous nature and as the NiO concentration increases from 30 wt% the amorphous nature partially disappear and crystallinity in the NCs were observed. It was evident that the NiO nanoparticles were completely interlocked between the polypyrrole chains. There were no additional impurity peaks were observed in XRD spectrum. The average crystallite size (D) was designed by using Scherrer's formula [5],

$$D=\frac{0.89\lambda}{\beta\cos\theta} \tag{1}$$

where D, the average crystallite size, λ; the wavelength of the X-rays (0.15405 nm). The deliberate average crystalline size (D) of PPy/NiO 30 wt % NCs were establish to be 28-36 nm.

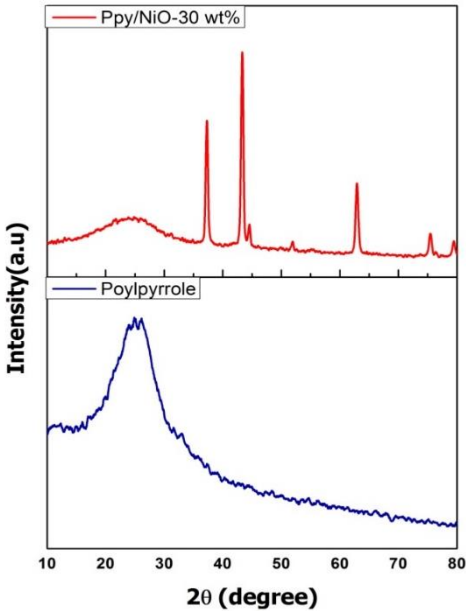

Fig. 1 XRD Pattern of (a) PPy (b)PPy/ NiO, (50 wt %)

3.2 Morphology Studies

The Fig. 2 shows surface morphology of the composites were studied by means of SEM and the results showed agglomeration of particles. From micrographs of TEM, particles size was around 25 nm. Since AFM images it is exposed that the nanocomposite is accompanied by much micrometric and nanometric irregularity [12-14].

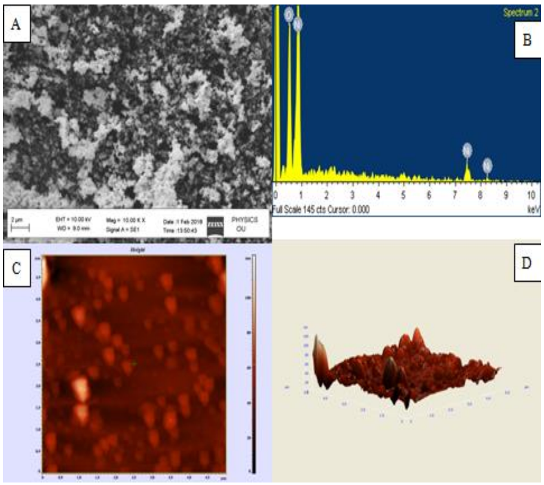

Fig. 2 SEM Micrographs of (a) PPy (b) NiO, (c&d) AFM images of Ppy: NiO (50 wt %)

3.3 Optical Properties

The diffuse reflectance spectra (DRS) of pure and PPy/NiO 30 wt % were shown in Fig. 3. The Kubelka–Munk (K-M) theory was utilized to determine the energy band gap (E_g) synthesized PPy/NiO from DR spectra. The Kubelka–Munk function F (R_∞) and photon energy (hυ) was calculated by following equations.

$$F(R_\infty)=\frac{(1-R_\infty)^2}{2R_\infty} \tag{2}$$

$$h\nu=\frac{1240}{\lambda} \tag{3}$$

where R_∞; reflection coefficient of the sample, λ; the absorption wavelength. The estimated Eg values 1.84 were mainly ascribed to degree of structural arrange and mess in the matrix as well as transform the distribution of energy levels within in the band gap.

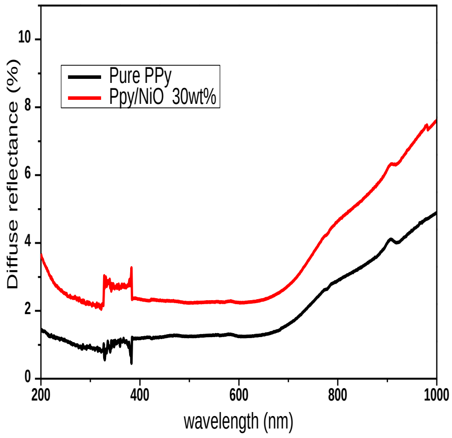

Fig. 3 DRS Pattern of (a) PPy (b)PPy/ NiO, (30 wt %)

4. Conclusion

Pure and PPy/NiO NCs were successfully prepared by in-situ polymerization technique. The nanocomposites showed high crystalline nature with no impurity peaks. The surface morphology of the composites were studied by means of SEM and the results showed agglomeration of particles. From micrographs of TEM, particles size was around 25 nm which was in good agreement with that of the particle size calculated from Scherer equation. All these results together reveal that the synthesized composites can be used in the field of optical applications.

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