Magnetic and transport properties of conducting polyaniline/nickel oxide nanocomposites

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ABSTRACT

Nickel oxide (NiO) nanoparticles were prepared by the simple approach of co-precipitation method using nickel carbonate as precursor. Novel electrically conducting composite materials consisting of nickel oxide nanoparticles dispersed in a polyaniline (PANI) are prepared by an in-situ polymerization method in the presence of different weight percentage of NiO (5, 10, 15 and 20%) at room temperature using ammonium persulphate $(NH4)2S2O8$ as an oxidant in acidic medium. The synthesized PANI/NiO nanocomposites have been characterized by means of XRD, FTIR, UV-VIS, TEM and VSM for structural and magnetic investigation. NiO has single phase cubic structure with average crystallite size of 23nm and is intercalated to form a core shell of PANI due to which nanocomposites show the peaks of NiO as well as PANI. PANI/NiO nanocomposites showed semiconducting as well as ferromagnetic nature. It was also observed that the conductivity of the PANI/NiO nanocomposites decreased and the magnetization increased with the increase in weight percentage of NiO in PANI. We studied first time the effect of NiO on transport properties of PANI/NiO nanocomposites in terms of transport parameters such as electrical conductivity (σ), charge localization length (α-1), most probable hopping distance (R) and charge hopping energy (w) using variable range hoping (VRH) of charge model as described by Ziller to conducting polymers. Copyright © 2013 VBRI press.

Keywords: conducting polymers; polyaniline; nanostructure nickel oxide; nanocomposites; transport parameters; VSM.

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Introduction

Organic-inorganic composites have attracted considerable attention as they can combine the advantages of both components and may offer special properties through reinforcing or modifying each other. In conducting polymer nanocomposites filler in nanoscale plays the major role in strengthening the properties of the nanocomposites. Uniform dispersion of nanoscopically sized filler particles produces ultra large interfacial area per volume between the filler and the host polymer. Thus, nanostructure filler into polymer matrix provide opportunities to enhance the properties associated with conventional composite materials [1]. The conducting polyaniline (PANI) is one of the promising conducting polymers due to its high conductivity, ease preparation, good environmental stability and large variety of applications [2-4] which make this polymer suitable for gas sensor [5], functional hybrid [6], as pH swchiting electrical conducting biopolymer hybrid for sensor applications [7], as an electrically active redox biomaterial for sensor applications [8], as a matrix for preparation of conducting polymer nanocomposites [9,10]. Therefore, there has been increasing interest of the researchers for the preparation of nanocomposites based on PANI. The literature review on conducting polymer nanocomposites show that PANI has been successfully utilized for the preparation of nanocomposites [11, 12]. When nanostructure filler particles are magnetic in polymer matrix, then such nanocomposites show enhance magnetic as well as transport properties of the polymer [13]. Magnetism of nanoscopic systems draws much attention due to their unique magnetic properties as well as their technological applications. Besides the structural aspects and magnetic properties of nanomaterials to be incorporated into polymer matrix, transition metal oxide nanoparticles are of particular interest.

Nickel (II) oxide is a notable and well-studied material among various transition metal oxides because of its unique advantage in terms of properties and applications such as p-type transparent conducting film, electrochromic devices, gas sensors, spin valve devices, cathode in alkaline batteries etc [14]. Some reports suggested that NiO nanoparticles exhibit weak ferromagnetism or superparamagnetism for the fine particles [15]. Recently, Karthik et al [16] have studied that the particle size has strong influence on the magnetic properties of NiO nanoparticles. Smaller particles have large interface area between the ferromagnetic phase and antiferromagnetic matrix and the structural disorder and exchange coupling increases with interface area. NiO is an antiferromagnetic transition metal oxide which is considered to be a semiconductor with p-type conductivity and band gap 3.51 eV [17]. NiO nanoparticles also have excellent thermal stability and capable of improving the thermal stability of PANI. Song and co-workers have prepared PANI/NiO nanoparticle, nanobelt and nanotubes in the presence of sodium dodecylbenzenesulphonate which showed improved conductivity and thermal stability [18-20]. Xu-Yuan Peng et al studied electrochemical co-deposition of NiO and PANI and showed the enhanced catalytic

activities of the composite material [21]. Yanni Qi et al studied the thermal stability and glass transition temperature of PANI/NiO composites increased with NiO content increasing in PANI [22]. NiO nanoparticles can also be prepared by simple route such as homogeneous precipitation [23], sol-gel route [24], chemical precipitation [25], chemical spry pyrolysis [26], solid state decomposition route [27], electrochemical deposition [28] etc. With this background, we have prepared NiO nanoparticles by simplest sol-gel route and the novel PANI/NiO nanocomposites by encapsulating the NiO nanoparticles with PANI matrix by an in-situ polymerization. To the best of our knowledge, first time we have reported in this paper about the transport properties in terms of transport parameters of chemically synthesized PANI/NiO nanocomposites by applying a variable range hopping model so as to find efficient, functionalized and electronic nanocomposites for multifunctional applications.

Experimental

Chemicals

Aniline (S. D. Fine-Chem. Ltd., 99.5%) was vacuum distilled prior to use. All other chemicals like nickel carbonate (99%), hydrochloric acid (35.4%), ammonium peroxydisulfate (APS, 98%), ethanol (99.9%), sulphuric acid (98%), N, N-dimethyl formamide (DMF) were bought from S. D. Fine-Chem. Ltd. and used as received without further purification. All chemicals were of analytical grade. Solutions were prepared in deionized water.

Synthesis of NiO nanoparticles

Nickel oxide (NiO) nanoparticles were prepared by the simple approach of sol–gel process in which, 0.1M nickel carbonate was added in 100ml starch solution and the mixture was stirred for half an hour. Then ammonia was added drop wise in the solution under constant stirring. After complete addition of ammonia, the solution was allowed to settle for overnight and then filtered using membrane filtration assembly, washed using deionised water and ethanol to remove the impurities and then dried at 80oC in hot air oven. Dried sample was treated at different temperatures in order to maintain the stability of compound. The colour of the sample was changed from green to faint gray at 100°C to 750oC. This nickel oxide was used to prepare PANI/NiO nanocomposites.

Synthesis of PANI/ NiO nanocomposites

The as-synthesized NiO nanoparticles were incarporated in PANI matrix by an in-situ chemical oxidative polymerization [29]. In a typical procedure, 0.1M of aniline was dissolved in 100 ml of 1M H_2SO_4 solution and stirred for 1 hour, then mixed with 10 ml of sonicated NiO nanoparticles by further sonication for 30 min. 100 ml of 1M H_2SO_4 solution containing 0.1M $(NH_4)_2S_2O_8$ (APS) was then slowly added drop wise to well dispersed suspension mixture for 2 hour with a continuous stirring. After 3 hour, a good degree of polymerization was

achieved as observed by the change in colour from blue to blackish green and the solution was kept overnight. The precipitate produced in the reaction was removed by filtration, washed repeatedly with 1M H2SO4 and dried under vacuum for 24 hour. The conductive emeraldine salt (ES) form of PANI/NiO nanocomposite powder was obtained. A schematic presentation of PANI/NiO nanocomposite preparation in the form of flow chart is described in **Scheme 1**. PANI/NiO nanocomposites with different weight percentage were synthesized using 5, 10, 15 and 20 weight % of NiO with respect to aniline monomer and these were abbreviated as PN-5%, PN-10%, PN-15% and PN-20% respectively.
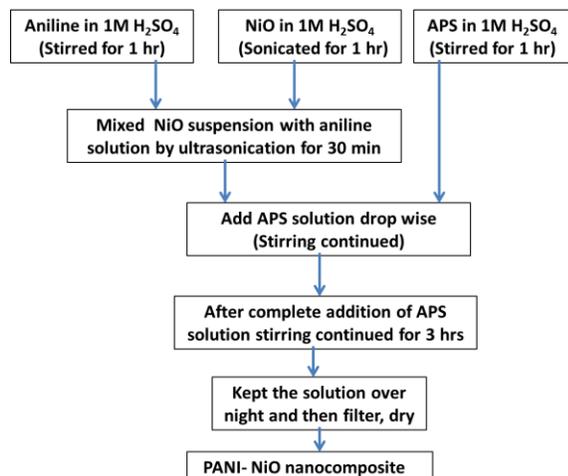
Scheme 1. Schematic presentation of PANI/NiO nanocomposite preparation

Characterization

X-ray diffraction (XRD) analysis was carried on Philips PW1710 automatic X-ray diffractometer with Cu-Kα radiation (λ=1.5404Å), with a scanning speed of 10° min−1. FTIR spectra were performed on Shimatzu FTIR-8101A Spectrophotometer in the wavelength range of 400–4000cm−1. Transmission electron microscopy images were obtained using PHILIPS-CM200 TEM with resolution 2.4Å. The optical absorption spectra were carried out using UV-1800 double beam UV-VIS spectrophotometer. The electrical conductivity of the compressed pellets of PANI/NiO nanocomposites were determined by four probe resistivity technique. The pellets were prepared with the help of hydraulic press (Kimaya Engineers, India) by applying a pressure of 5000 kg/cm2. The magnetization measurements were carried out using vibrating sample magnetometer (VSM) model Oxford Maglab 14T.

Results and discussion

Fig. 1 shows the FTIR spectra of NiO, PANI and PANI/NiO nanocomposites. FTIR spectra of PANI show the main characteristic peaks at 3454, 1578, 1487, 1307, 1251, 1147 and 824 cm[-1]. The peak at 3454 cm[-1] could be attributed to N-H stretching mode. The bands at 1578 and 1487 cm[-1] are attributed to stretching vibrations of N=Q=N ring and N-B-N ring respectively (where B refers to benzenic-type rings and Q refers to quinonic-type rings). The bands at 1307 and 1251 cm[-1] correspond to N-H bending and the symmetric component of the C-C stretching modes. The peak at 1147 cm[-1] assigned to C-N stretching of secondary aromatic amine. The peak at 824 cm[-1] can be attributed to the out-of-plane bending vibration of C-H on the 1,4-disubstituted aromatic rings **[30]**.
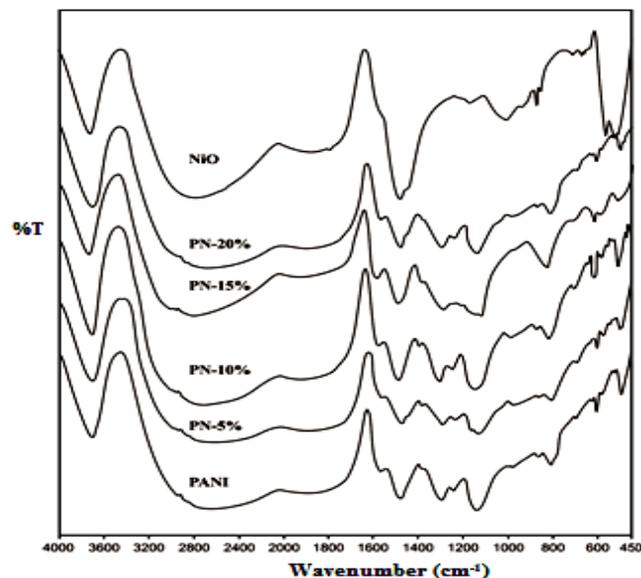
Fig. 1. FTIR spectra of PANI, NiO and PANI/NiO nanocomposites.
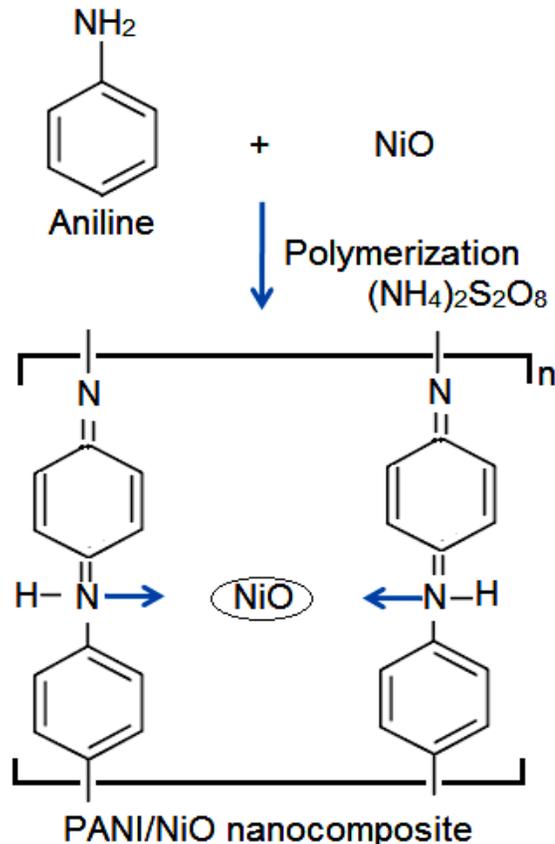
Scheme 2. Structure of PANI/NiO nanocomposite.

The characteristic peak at 713 cm^{-1} in the spectra of NiO ascribed to the bending vibrations of the NiO crystal lattice was also observed in PANI/NiO nanocomposites. The absorption bands due to stretching vibrations of quinoid and benzoid rings in PANI/NiO nanocomposites indicate the emeraldine form of oxidation state confirmed the polymerization of aniline in the presence of NiO. The interaction between PANI and NiO nanoparticles was confirmed as these bands were found to be shifted to lower wavelengths as the content of NiO increases from 5 to 20% in PANI-NiO nanocomposites. **Scheme 2** shows the structure of PANI/NiO nanocomposites indicating NiO embedded polyaniline.
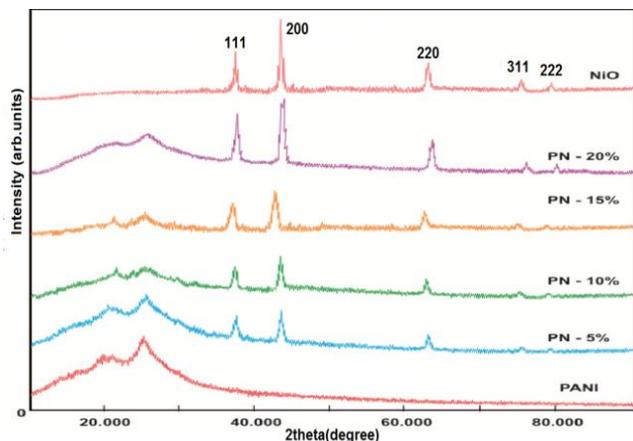
Fig. 2. XRD of PANI, NiO and PANI/NiO nanocomposites.

X-ray diffraction patterns of NiO, PANI and PANI/NiO nanocomposites are shown in **Fig. 2**. The crystalline peaks at 2θ = 37·53°, 43·60°, 63·09°, 75·67°, 79·58° which have been identified as peaks of single phase cubic structure of NiO with diffracting planes (111), (200), (220), (311), (222) respectively [31] matched with JCPDS file 73-1523. The crystallite size of the NiO was found to be 23nm determined by Scherer's formula. The XRD pattern of PANI shows that PANI has partly crystalline structure [2], and the two broad peaks are observed at 2θ = 20.38° and 25.72°. XRD patterns of PANI/NiO nanocomposites show the crystalline peaks of NiO and PANI. This indicates that PANI undergoes interfacial interactions with NiO crystallites and the presence of NiO in PANI matrix strongly affects the crystalline behavior of the formed PANI. Nanocomposites showed more crystalline than that of pure PANI indicating the advantage of both NiO and PANI to form an advanced material. **Fig. 3(A), (B)** and **(C)** shows TEM images of NiO, PANI/NiO (15%) nanocomposite and PANI respectively. TEM image (A) indicated that the prepared NiO nanoparticles have spherical morphology and average particle size of 25nm. TEM image (B) of PANI/NiO elucidated that NiO nanoparticles exist spherically in the PANI matrix of morphological wirelike or tubular as shown in (C) indicating the coating of PANI on the NiO nanoparticles. It might be a result of aniline polymerization and growth encircled the NiO

nanoparticles [22], representing the increased in particle size of the nanocomposite.
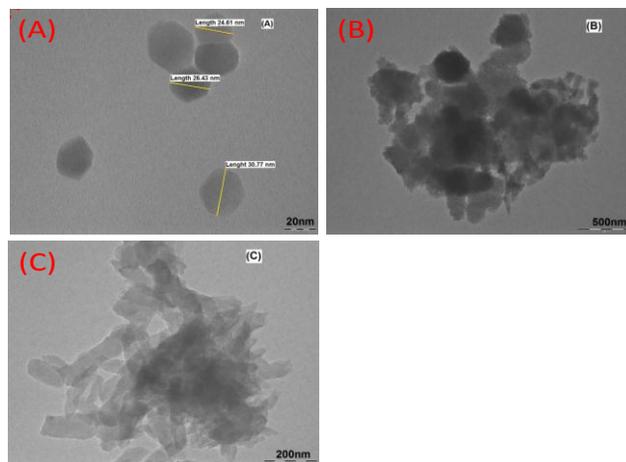
Fig. 3. TEM image of (A) NiO, (B) PANI-NiO (15%) nanocomposites, and (C) PANI.
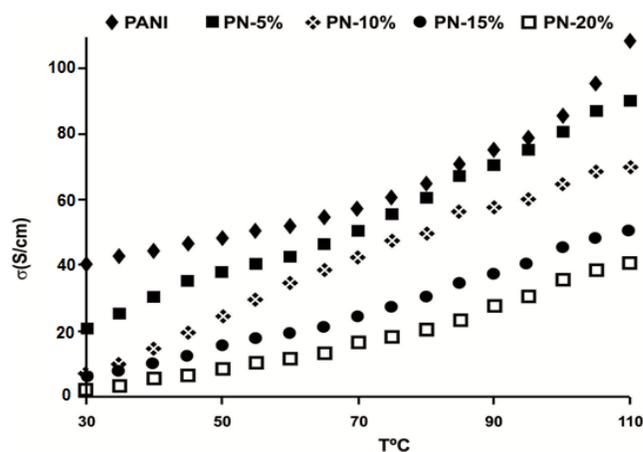
Fig. 4. Electrical conductivity of PANI and PANI-NiO nanocomposites.

Thermal dependence electrical conductivity of PANI and PANI-NiO nanocomposites is shown in **Fig. 4**. The increase in conductivity of PANI-NiO nanocomposites with the increase in temperature is the characteristics of thermal activated behavior following quasi-1D the variable range hopping conductivity model as that of pure PANI. Change in electrical conductivity of the conducting polymer nanocomposites depends on the conductivity of the filler particles into conducting polymer matrix [32].

The room temperature conductivity of PANI-NiO nanocomposites was found to be slightly lower than that of pure PANI but much higher than that of NiO (0.1x10^{-7}S/cm). This may be due to the partial blockage of the conductive paths by NiO nanoparticles embedded in the PANI matrix for the charge carriers which do not have sufficient energy to hop between favorable sites [30]. With the increase of NiO content in composite the decrease in electrical conductivity was observed. The decrease in the conductivity of the nanocomposites was attributed to the increased charge carrier scattering between doped PANI and NiO. As the NiO nanoparticles embedded in the PANI

matrix which interrupted the doping process, interactions between NiO and PANI increased the localization of charge carriers, and thus decreased the conductivity of the composites. A similar decrease in electrical conductivity was also observed in various kinds of conducting polymer nanocomposites **[33,34].** The room temperature conductivity of the nanocomposites along with transport parameters are reported in **Table 1**. To find transport parameters of the conducting polymer nanocomposites, Ziller equation **[35]** has been used.

$$\sigma(T) = \sigma(o).e^{\left(-\frac{To}{T}\right)^{1/2}}$$

The plot of *log σ(T)* versus $T^{-1/2}$ was found to be linear for pure PANI and PANI-NiO nanocomposites, hence the characteristic temperature 'To' was determined from the slope of the line. Then transport parameters such as charge localization length (α^{-1}), most probable hopping distance (R) and charge hopping energy (*w*) were determined using the following relations,

$$\alpha^{-1} = \frac{8\,N(Ef)\,Zk}{To}$$

$$R = \left(\frac{To}{T}\right)^{1/2}\left(\frac{\alpha^{-1}}{4}\right)$$

$$w = Zk\,\frac{To}{16}$$

In these relations, *Z* is the number of nearest neighbouring chains (~4), *k* is Boltzmann constant and $N(E_f)$ is the density of states per electron volt (2–ring unit suggested for polyaniline) **[36].** Although nanocomposites have room temperature (303K) conductivity less as compared to pure PANI but conductivity of nanocomposites is due the extended chain length of polyaniline where polarons possess sufficient energy to hop between favorable sites. The electrical conductivity of PANI-NiO nanocomposites shows semiconducting behavior as that of conducting polyaniline with filler concentration dependant variation of transport parameters. From the **Table 1**, it is seen that the transport parameters of PANI/NiO nanocomposites are depending on the concentration of the filler. As the concentration of the filler in PANI matrix increases, the charge localization length and most probable hoping distance decreases whereas hoping energy and the characteristic temperature increases which may be due to increase of blockage or decrease of path way for the carriers. From the study of transport parameters, the nanocomposites under this study shows synergistic effect of good conductivity with moderate charge localization length, most probable hopping distance, charge hopping energy for the charge carriers to hop from / to polaronic states lies in the band gap of materials as that of PANI and nano dimension from NiO nanoparticles. Such nanocomposites materials can be useful for electronic devices.
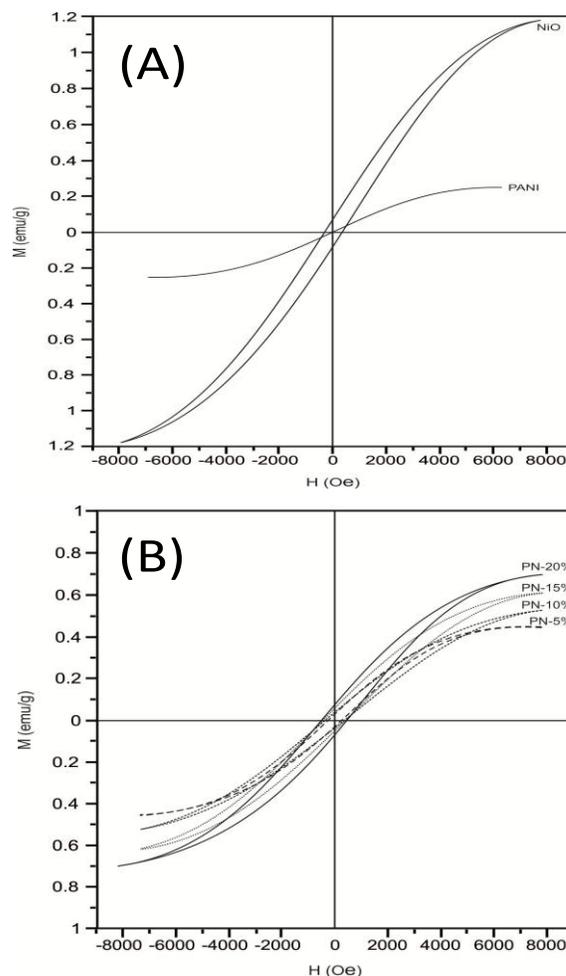
Fig. 5. Magnetic behavior of the (A) PANI and NiO, and (B) PANI-NiO nanocomposites.

Fig. 5(A) shows a typical hysteresis loop obtained at room temperature for PANI and NiO. The saturation magnetization (*Ms*) and retentivity (*Mr*) of the PANI were found to be 0.25 emu/g and 5.7 x 10^{-3} emu/g respectively exhibited paramagnetic nature. The saturation magnetization, coercive field (*Hc*) and retentivity (*Mr*) of NiO were found to be 1.19 emu/g, 452.66 Oe and 98.4 x 10^{-3} emu/g respectively exhibited ferromagnetic nature. The squareness ratio (SQR = *Mr/Ms*) is an important assessment of the quality of the magnetic materials. The SQR for NiO was found to be 82.68X10^{-3} indicating that the pure NiO do not have the properties of a recording medium **[37]. Fig. 5(B)** shows a typical hysteresis loop obtained at room temperature for PANI-NiO nanocomposites. The hysteresis loop area of the nanocomposites was found to be dependent on concentration of NiO in PANI matrix. The saturation magnetization (*Ms*), coercive field (*Hc*) and retentivity (*Mr*) of PANI-NiO nanocomposites were found to be increased with increasing the concentration of NiO due to high dispersivity of the NiO in polyaniline matrix. The SQR along with *Ms, Mr* and *Hc* for nanocomposites are reported in **Table 2**, which shows that nanocomposites also do not have the properties of a recording medium as like pure NiO but are good conductors as compared to

NiO. Such materials can be used as soft electro-magnetic materials in constructing transformers and cathode materials in batteries.

Table 1. Transport parameters of PANI and PANI/NiO nanocomposites

Polymer	σ(S/cm) at 303K	T_o (K)	α^{-1} (nm)	R (nm)	W (eV)
PANI	40.4	14085	0.862	2.15	0.30
PN-5%	21.2	23285	0.409	1.42	0.56
PN-10%	8.80	37168	0.303	1.26	0.71
PN-15%	6.38	42271	0.238	1.12	0.85
PN-20%	1.12	47102	0.214	1.05	0.92

Table 2. Magnetic parameters of PANI, NiO and PANI/NiO nanocomposites

Material	Ms (emu/g)	Mr (emu/g) $\times 10^{-3}$	Hc (Oe)	SQR $\times 10^{-3}$
NiO	1.19	98.4	452.66	82.68
PANI	0.25	5.7	----	22.8
PN-5	0.45	39.5	238.48	87.7
PN-10	0.53	45.4	296.46	85.6
PN-15	0.61	56.3	312.12	92.2
PN-20	0.70	68.2	352.16	97.4

Conclusion

NiO nanoparticles and PANI-NiO nanocomposites have been successfully prepared. FTIR spectra, XRD patterns and TEM images of PANI-NiO nanocomposites indicate the interactions exist between PANI and NiO nanoparticles. The incorporation of NiO into PANI matrix affects the transport and magnetic properties of nanocomposites. PANI-NiO nanocomposites show ferromagnetic as that of NiO but high electrical conductivity forming good conductive magnetic materials. Due to this synergistic effect of PANI and nano dimension of NiO such nanocomposites can be useful as soft electro-magnetic materials in constructing transformers, cathode materials in batteries and for electronic devices.

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