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Studies on Conduction Mechanism, Magnetization and Electrochemical Properties of Polythiophene-Cobalt Nanocomposites

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ABSTRACT

Polythiophene (PTh) was prepared by chemical oxidation and cobalt nanoparticles (Co-nps) by modified polyol process. The nanocomposites were prepared by mixing mechanically the PTh and Co-nps in the weight percentage, $PTh_{100-x}Co_x$, x = 10, 20, 30, 40 and 50. XRD of these nanocomposites showed amorphous nature. DC conductivity variation with temperature in the range from 303 K to 473 K indicated semiconducting behavior. Conduction mechanism was found to be small polaron hopping at high temperature and 3D variable range hopping at low temperatures. Conductivity and activation energy values points out that addition of Co-nps to the PTh induces electrically insulating effect. The SQUID magnetometer measurements at 10 K and 300 K showed perfect ferromagnetic nature and magnetically softness of the composites. Cyclic voltammetry study has been carried out on all the composites and in that a composite having 30% of Co-nps measured Specific Capacitance (SC) value as high as 583.20 F/g. This can be the best candidate for capacitor applications. This is for the first time that PTh-Co nanocomposites have been thoroughly probed for structure, conduction mechanism, magnetization and supercapacitor applications.

1. Introduction

Polythiophene (PTh) is an intrinsically conducting polymer and it has been studied extensively for its electronic properties and applications like light emitting diode, supercapacitors, field effect transistor, DNA detection, etc., [1-5]. On the reinforcement of inorganic nano material in to the matrix induces changes in its properties [6]. For example, improvement in conductivity was observed in polythiophene-silver [7], polyaniline-silver [8] and polyaniline-gold [9]. The dispersion of ferromagnetic metal nanoparticles in a polymer matrix is of great interest due to the combined effect of the magnetic and electric properties of the individual elements. Nanocomposites prepared using conducting polymer and ferromagnetic metal nanoparticles such as polythiophene-nickel by electrochemical method exhibited good electrical conductive response [10]. Increase of saturation magnetization and decrease in electrical conductivity with respect to Fe content were noted for polyaniline-Fe nanocomposites [11]. Super paramagnetic behavior and microwave absorption application for the polyacrylonitrile/Ni/Co/Ni-Co nanocomposites were proposed in [12]. Thermal conductivity increased in nickel doped polypyrrole composites [13]. Incorporation of cobalt in polypyrrole exhibited resistive switching and magnetism [14] and showed utility in the application of electromagnetic wave absorption [15]. Of the three prominent ferromagnetic metals, cobalt has always been considered to be special as it has high saturation magnetization and Curie temperature [16]. Also, it is known for its allotropic forms of fcc, hcp, epsilon and bcc [17]. Many researchers have reported study on the magnetic properties of the cobalt nanoparticles synthesized via different methods [18–21]. Cobalt nanoparticles have shown some potential applications like microwave absorption [22] adsorption ability [23] and high energy product [24]. To the best of our knowledge there are no reports on thorough analysis of electrical properties and magnetization of polythiophene-cobalt (PTh-Co) nanocomposites. It is also interesting to study electrochemical behavior of such nano composites.

Keeping in view of this, we experimentally investigated XRD, electrical conductivity as a function of temperature, magnetization and voltammetry on PTh-Co nanocomposites and results obtained are presented in this paper.

2. Experimental Methods

Analytical grade chemicals were used to synthesize polythiophene and cobalt nanoparticles. Polythiophene (PTh) has been synthesized by chemical oxidation method using $FeCl_3$ as an oxidizing agent. The polymerization was carried out for 24 hours at the temperature of 275 K. Black precipitate of polythiophene obtained was filtered and washed several times with methanol and double distilled water and, the resulted powder was dried in an oven. The complete procedure of polythiophene preparation is described in reference [25].

To prepare cobalt nanoparticles, the modified polyol process was followed in which cobaltous chloride hexahydrate ($CoCl_2 . 6H_2O$) and sodium hydroxide (NaOH) were dissolved in 1,2-propandiol separately. Both solutions were stirred and mixed and, then treated with hydrazine hydrate ($N_2H_4 . H_2O$) 80%. The reduction was carried out in the temperature range from 328K to 333K. The dark grey colour cobalt particles formed were collected and washed several times with double distilled water and acetone. Finally, the powder was dried in an electric oven [26].

As prepared PTh and Co particles were mechanically mixed well in pestle mortar in the weight percentages defined as $PTh_{100-x}Co_x$, where, x = 10, 20, 30, 40 and 50 and the composites were labeled as PTh-CO1, PTh-CO2, PTh-CO3, PTh-CO4 and PTh-CO5 respectively. The powder composites were pelletized using hydraulic press by applying a pressure of 20 kg/cm^2 for conductivity measurement. The composites have been investigated for structure by means of XRD, dc conductivity measured by two probe method in the temperature range from 303 K to 473 K. A constant voltage, V has been applied across two side silver painted pellet. Current, I through the pellet has been measured. Resistivity, ρ has been calculated and conductivity, σ has been determined using the relation $\sigma = 1/\rho$. Magnetization of the powder nanocomposites was measured in the field range from -1T to +1T in a SQUID magnetometer (MPMS–XL, Quantum Design) at temperatures 10K and 300K. Cyclic voltammetry measurement of the PTh-CO nanocomposites were carried out using electrochemical analyzer (CH instruments model CHI 6112D) with

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standard 3 electrode system in which platinum wire was used as counter electrode, Ag/Agcl as reference electrode and PTh-CO composite as working electrode in a potential window from (-0.85) V to (+0.80) V.

3. Results and Discussion

3.1 X-Ray Diffraction Study

X-ray diffraction pattern of the sample PTh-CO1 is shown in Fig. 1. No sharp peaks are observed in the XRD pattern which indicates amorphous nature of the sample. The XRD patterns of PTh and cobalt nanoparticles separately also indicated amorphous nature [25, 26]. The remaining composites PTh-CO2, PTh-CO3, PTh-CO4 and PTh-CO5 also exhibited no peaks on their XRD patterns. This confirms that all the present composites are amorphous in nature. However the two small additional peaks are observed at 2θ values of 33.18° and 35.55°. These may be due to the residual $FeCl_3$ particles left in the polymerization process [25].

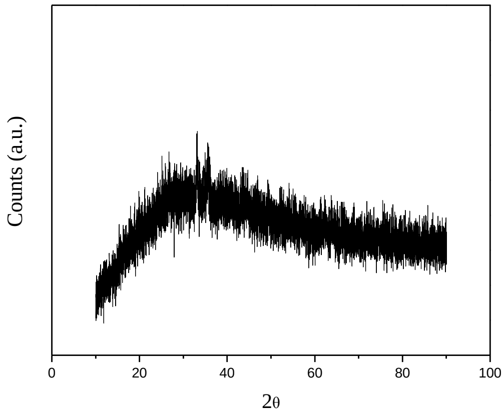

Fig. 1 XRD pattern of PTh-CO1 nanocomposite

3.2 Conductivity

Conductivity changes with temperature measured for PTh-CO1 composite is shown in Fig. 2. Conductivity increases with increasing temperature which reveals semiconducting type behavior. Similar nature of the conductivity behavior with temperature has been noted for the present remaining composites. The conductivity of all the present PTh-CO nanocomposites at 303 K are shown in Fig. 3. From the figure, decrease in conductivity with increase of weight percentage of cobalt-nps can be noted. This discloses that cobalt particles play an obstructive role to the polaronic motion. Similar results have been reported for PANI-Fe nanocomposites in reference [11]. We too reported similar observations for PTh-Ni nanocomposites in [25].

The temperature variation of electrical conductivity has been considered for analysis in terms of Mott's Small Polaron Hopping (SPH) model [27]. According to this model, conductivity is given by

$$\sigma = \frac{\sigma_o}{T} \exp\left(-\frac{E_a}{k_B T}\right) \quad (1)$$

where, σ_o is the pre exponential factor and E_a is the activation energy for small polaron hopping.

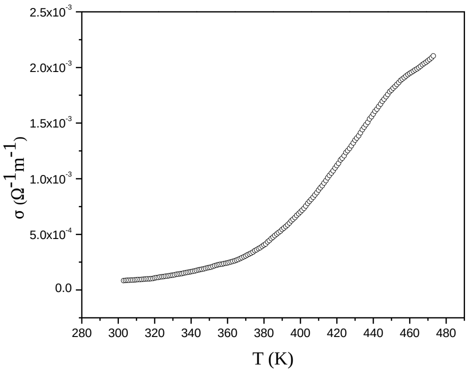

Fig. 2 Conductivity, σ variation with temperature, T for PTh-CO1 composite

In the Fig. 4, the plots of ln(σT) versus (1/T) for the present PTh-CO nanocomposites are shown. Data appeared linear in the high temperature region hence linear lines were fit to the data in that range of temperatures. The slope of these linear fits has been used to estimate activation energy (E_a) for conduction.

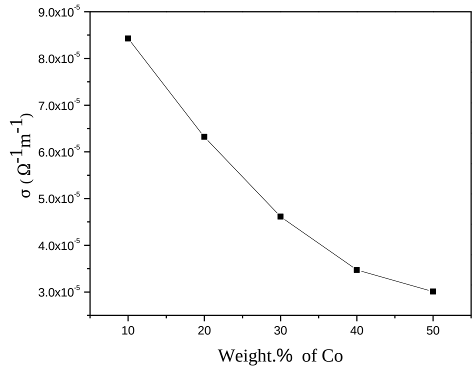

Fig. 3 Conductivity, σ (at 303 K) for the present PTh-CO nanocomposites

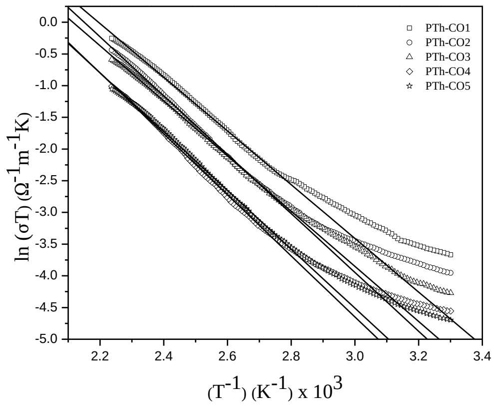

Fig. 4 The plots of ln(σT) versus (1/T) for PTh-CO nanocomposites

Table 1 Activation energy, E_a values observed from Mott's SPH model fits different weight percentage of cobalt in polythiophene

Samples→	PTh-Co1	PTh-Co2	PTh-Co3	PTh-Co4	PTh-Co5
E_a (meV)→	0.365	0.398	0.370	0.411	0.398

Activation energy, E_a values obtained from Mott's SPH fits to the data are tabulated in Table 1. Activation energy for all the composites is of the order of fraction of meV. Activation energy of the present composites is of the same order as reported for PTh-Ni composites and pure PTh particles [25, 28]. It can be observed that there is no ordered change in the E_a.

The data deviated from the SPH model has been fit to Mott's Variable Range Hopping (VRH) model According to this model, conductivity is given by [29],

$$\sigma = \sigma_o \exp\left\{-\left(\frac{T_o}{T}\right)^{\gamma}\right\} \quad (2)$$

where σ the conductivity, σ_o the high temperature limit of conductivity, T_o the Mott's characteristic temperature, T the temperature and exponent γ = 1/d+1, with d = 1, 2, 3 being dimensionality of the conduction process.

The slope, T_o of the plots of ln (σ) versus ($T^{-1/4}$) will be,

$$T_o = \left[\frac{18}{r_o^3 k N(E_F)}\right] \quad (3)$$

here, $N(E_F)$ refers to the density of states at Fermi level, *k* the Boltzmann's constant and r_o localization length ~1 nm [30].

The plots of ln(σ) versus ($T^{-1/2}$), ($T^{-1/3}$) and ($T^{-1/4}$) are shown in Fig. 5(a-c) respectively for 1D, 2D and 3D conduction processes. From the Fig. 5, it appears that linear fits corresponding to all 1D, 2D and 3D cases are good. But the regression coefficient, R extracted from these three different fits indicates that the 3D model fit is better than 1D and 2D model fits. Based on this, it can be argued that the 3D variable range hopping mechanism as envisaged by Mott is prevailing in the present nanocomposites. This deduction of result is in agreement with the reported result of reference [29, 31, 32]. Considering 3D VRH conduction mechanism to be operated at low temperatures, $N(E_F)$ values determined and are tabulated in Table 2. Results are in close agreement with the poly(2,5-dimethoxyaniline) and polythiophene films [33, 34]. However, the present N(EF) values are smaller than reported values for PTh-Ni, PPy-Cu nanocomposites [25, 27].

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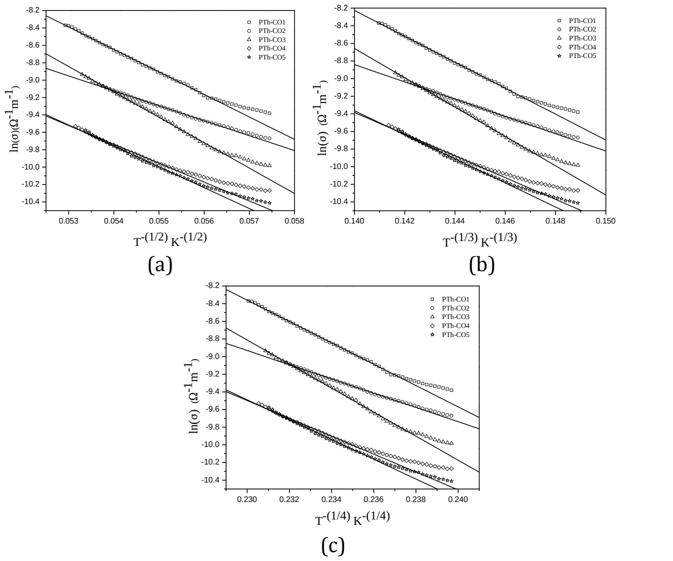

Fig. 5 The plots of ln(σ) versus $T^{-1/d+1}$ corresponding to (a) 1D, (b) 2D and (c) 3D conduction processes. Solid lines are linear fits to the data

Table 2 Estimated parameters from VRH model fits to the data at low temperatures

Composite	Regression Coefficient, R			T_o	$N(E_F)$
	1D	2D	3D	(K)	$eV^{-1}m^{-3}$
PTh-CO1	0.9993	0.9994	0.9994	$2.133X10^8$	$9.806X10^{23}$
PTh-CO2	0.9987	0.9988	0.9988	$4.231X10^7$	$4.941X10^{24}$
PTh-CO3	0.9990	0.9990	0.9990	$3.436X10^8$	$6.084X10^{23}$
PTh-CO4	0.9973	0.9974	0.9975	$1.047X10^8$	$1.998X10^{24}$
PTh-CO5	0.9974	0.9975	0.9976	$1.581X10^8$	$1.322X10^{24}$

3.3 Magnetic Properties

The magnetization of the PTh-CO nanocomposites has been investigated at low temperature, 10 K and room temperature, 300 K in a SQUID magnetometer as a function of an applied field in the range from 0 to 1 T. The results are displayed in Fig. 6. Areas of the loops are very small for all the five composites indicating that these samples are of loss less type. Other magnetic parameters obtained from the hysteresis loops are tabulated in Table 3. The saturation magnetization, M_s, remnant magnetization, M_r and coercive field, H_c are observed at 10 K and 300 K increases monotonously as the weight % of cobalt nanoparticles increases in the polythiophene. The increase of M_s, M_r and H_c with increase of Co-nps is in conformity with the expectation. It is because, the magnetization of these composites is solely due to Co-nps only and that increase of % of Co-nps in the PTh must have to increase the magnetization. It may be noted that the values of the present magnetic parameters are less than those reported values for pure Co-nps [26].

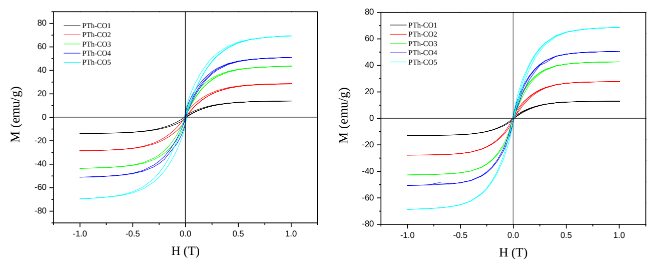

Fig. 6 Hysteresis loop of PTh-CO nanocomposites at (a) 10 K and (b) 300 K

Table 3 Magnetic parameters of the present PTh-Co nanocomposites

Composite	M_s ($emug^{-1}$)		M_r ($emug^{-1}$)		H_c (T) $X10^{-3}$		M_r / M_s	
	10K	300K	10K	300K	10K	300K	10K	300K
PTh-CO1	13.921	12.949	0.942	0.481	8.005	6.862	0.067	0.037
PTh-CO2	28.706	27.763	1.884	1.183	8.865	7.149	0.066	0.043
PTh-CO3	43.643	42.726	3.162	2.090	10.0092	7.232	0.072	0.049
PTh-CO4	51.099	50.618	4.123	2.371	11.7250	7.721	0.081	0.047
PTh-CO5	69.536	68.528	5.927	2.565	12.5801	8.005	0.085	0.037

3.4 Cyclic Voltammetry

To probe the utility of these composites for supercapacitors applications, the cyclic voltammetry (CV) measurements has been carried out in an Electrochemical Analyzer (CH instruments model CHI 6112D) in the potential window from (-0.85) V to (+0.80) V. The paste of the composites has been used as electrode material. These electrodes were prepared by mixing PTh-CO composites with PVA in 10:1 proportion and the same was coated on stainless steel strips of 304 grade by following doctor blade method.

The electrodes were electrochemically analyzed in 1 M Na_2SO_3 solution with standard 3 electrode system in which platinum wire was used as counter electrode, Ag/AgCl as reference electrode and PTh-CO composite as working electrode. The current that flows through the present 3-electrode system for different applied potential (voltage) were measured and plotted in Fig. 7(a-e). Experiments were conducted for different scan rates of voltage. The data collected for scan rates of 5 mVs^{-1}, 10 mVs^{-1}, 20 mVs^{-1}, 50 mVs^{-1}, 100 mVs^{-1} have been plotted in single graph so that they can be compared with ease.

In Figs. 7(a) and (b), no distinct peaks can be observed either for positive or for negative potential sweeps. The potential window was extended from -0.51 V to -0.62 V for PTh-CO1 and from -0.674 V to 0.8V for PTh-CO2. The areal current density increased with increase in scan rate in both of these samples. In Fig. 7(c), for scan rate of 100 mVs^{-1}, PTh-CO3 exhibited a sharp peak is observed on positive side around 0.1 V and a small peak on negative side around -0.1 V. Peaks for both positive and negative potential sweeps can be seen in Fig. 7(d) for PTh-CO4. The peaks become sharper and height of the peaks increases with increase in scan rate. This may be due to occurrence of more reactions at that potential and for those sweep rates by maintaining phase intact. The current integral increases with increase in scan rate. In Fig. 7(e), the broad peak at 0.4 V on positive side is observed. This peak becomes sharp as the scan rate is increased remarkably, the negative potential sweep does not show any peaks. No perfect rectangular shape of the voltammogram is seen for any of the samples. The peaks found in PTh-CO3, PTh-CO4 and PTh-CO5 nanocomposites exhibits pseudo capacitance behavior as was observed in reference [35].

Capacitance, C and specific capacitance, SC were estimated using the formulae [36],

$$C = \frac{\int_{V_1}^{V_2} I\,dv}{V\frac{dv}{dt}} \quad (4)$$

$$SC = \frac{\int_{V_1}^{V_2} I\,dv}{mV\frac{dv}{dt}} \quad (5)$$

where, I is the current, V = V_2 – V_1 the potential range, dv/dt is the scan rate, m = weight of the electrochemically active material.

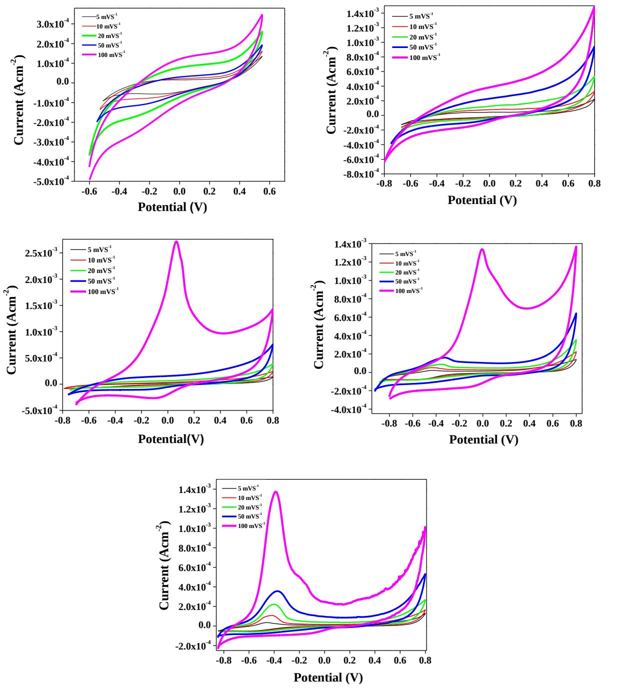

Fig. 7 Current, I versus potential, V for composites (a) PTh-CO1, (b) PTh-CO2, (c) PTh-CO3, (d) PTh-CO4 and (e) PTh-CO5

The SC values obtained for all the samples are listed in the Table 4. SC values are very low for PTh-CO1 and PTh-CO2 and moderate for PTh-CO4 and PTh-CO5. Whereas the composite PTh-CO3 exhibited appreciable size of SC. From these SC results, one may propose the PTh-CO3 composite, to be the best for use as electrode material in the capacitors. The decrease of

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SC value with increase of scan rate generally noted for all the present samples may be attributed to underutilization of electrode material at higher scan rates. Similar conclusion was drawn in references [35, 37, 38].

Table 4 Calculated Specific Capacitance, SC for PTh-CO nanocomposites

Scan rate	Specific capacitance (SC) in F/g for composite				
mVs^{-1}	PTh-CO1	PTh-CO2	PTh-CO3	PTh-CO4	PTh-CO5
5	1.500	1.195	583.20	12.75	14.04
10	0.950	0.966	475.20	8.40	11.50
20	0.630	0.804	77.22	6.12	10.11
50	0.460	0.552	39.60	4.56	9.80
100	0.360	0.463	34.74	3.75	8.50

4. Conclusion

Polythiophene and cobalt nanoparticles were synthesized. The nanocomposites were prepared by mechanically mixing PTh and Co nps in the weight percentages, $PTh_{100-x}Co_x$ where, x = 10, 20, 30, 40 and 50

(i) XRD studies revealed amorphous nature of the composites.

(ii) Small polaron hopping is found to be the conduction mechanism at high temperatures and Mott's 3D-VRH to be the mechanism at lower temperatures. Activation energy for conduction and density of states at Fermi level were estimated.

(iii) Magnetic properties indicate that these are lossless material to large extent. Hysteresis loops at 10K and 300K shows perfect ferromagnetic nature. Saturation magnetization, remnant magnetization and coercive field are found to increase with weight % of Co nps which is in agreement with general expectation. The values of these parameters also indicate that these composites are soft magnetic materials.

(iv) The cyclic voltammograms revealed that the nanocomposite having 30 wt % of Co nps is best suited for electrodes in capacitors as it has exhibited higher value of specific capacitance. It is for the first time that PTh-CO nanocomposites have been explored for structure, electrical conduction mechanism, magnetic properties and capacitor applications.

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