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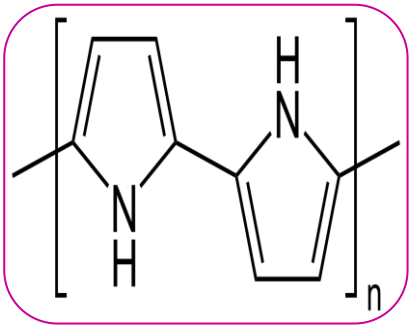
LOADING EFFECT OF HEMATITEON MORPHOLOGY ANDCONDUCTIVITY OF PPY/ A-FE_2O_3NANOCOMPOSITES



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ABSTRACT

We prepared the conducting polymer (CP) polypyrrole (PPY) and its composites with α - Fe_2O_3 by in situ chemical oxidation polymerization using anhydrous $FeCl_3$ as oxidant and HCl as dopant by keeping different molar percentage of Fe_2O_3 with pyrrole monomer. The prepared PPY and PPY/α-Fe_2O_3 composites were characterized by FTIR and UV-Vis spectroscopy and, their morphology examined by SEM and TEM techniques. The d.c. electrical conductivity (σ_{dc}) of PPY and nanocomposites (NCs) PPY/α-Fe_2O_3 against temperature ($32^0c – 100^0c$) has been measure by four probe method.

KEYWORDS : *Polypyrrole, Nanocomposites,FT-IR, UV-Vis, SEM, d.c. electrical conductivity .*

I. INTRODUCTION:

There has been explosive growth of present era research in the field of CPs. With remarkable physical, thermal, electrical and magnetic properties, CPs have their potential application in various fields such as solar cell, light emitting diodes, sensors, molecular electronic devices, light weight batteries, biosensors, protecting agent and flexible displays [1].

So many efforts have been taken by researchers to change structure of CPs so that their applications should highly improve by using organic / inorganic dopants and oxidants [2]. Many investigated CPs are available but among these the PPY have high room temperature electrical conductivity, appreciable environmental stability, and easy synthesis, and easily doped with various dopants. Lot of potential application of PPY such as sensors, solid electrolytes and electrodes, for capacitor and solid state batteries have been reported [3].

Valuable ingredient Fe_2O_3 used in PPY/Fe_2O_3 composite which has been attractive physical and chemical properties such as low cost, non- toxicity, and, thermal and chemical stability. It has been identify as an important material used to modify biological active material, storage devices, magnetic sensors and catalysts [4].

The conducting polymer composite (CPC), PPY/Fe_2O_3 plays an important role due to physical and thermal stability, easy synthesis, facial processing, bio compatibility, reversibility and switching capacity and non-explosive. It is used in biosensors, chromatography, batteries and photovoltaic devices [5].Wanchun Jiang et al (2016) reported the enhanced electromagnetic absorbing (EA) performance of α-Fe_2O_3/PPy (prepared by a facile one-step method) composite. Potential application for microwave absorption of this composite also has been reported [6].

In the present study, we report the preparation of PPY and PPY/ α-Fe_2O_3 composites via in situ chemical oxidation polymerization [7]. FTIR, UV-Visible and SEM techniques are used to investigate their characteristics, morphology and structural information. In this study we report the effect of different molar concentration of Fe_2O_3 in composites on the morphology, optical property and dc conductivity.

II. EXPERIMENTAL

A. Materials

Pyrrole monomer (AR grade Sigma-Aldrich) was purified by distillation under reduce pressure and store at about 10 degree Celsius before use. The anhydrous $FeCl_3$ (AR grade), Hematite (α-Fe_2O_3) (AR grade) and HCl, all of MERCK make, are used in the present study. Deionized waterwas used as a solvent for all solutions.

B. Preparation of PPY

The PPY is synthesized by in-situ chemical oxidation method at room temperature (32^0c). First oxidant solution of 16.2gm (1M) anhydrous $FeCL_3$ in 100 ml deionized water has stirred for 1 hrs. The second aqueous solution containing 3.45 ml (0.5M) pyrrole in 50 ml deionized water has stirred for 1hrs. Then after the first solution has been added drop wise in second aqueous solution of PPY and stirred for 6 hrs [8]. The prepared greenish black colored solution was filtered,then washed by distilled water and methanol till getting colorless filtrate. Resulting greenish black precipitate was dried at 60^0C up to 8 hrs. On the basis of characterization resulting precipitate was identified as PPY [9,10].

C. Preparation of PPY/Fe_2O_3 composites

The first 50ml aqueous solution containing 3.45ml (0.5M) pyrrole stirred for 1hr. The second 100 ml aqueous solution containingdifferent weight percent of Fe_2O_3 (15% to 30 %) with monomer(0.075M, 0.1M, 0.125M, 0.15M) [11] and 1.225 ml (0.4M) HCl stirred for 1 hr. Third 100ml aqueous solution of 1M FeCl3 (16.2 gm) and 1M H_2SO_4 (5.389 ml) stirred for 1 hr. Second and third solutions were mixed and stirred for 20 minutes. Then first solution added drop wise in it and stirred for 6 hrs. The resulting precipitates (changing color from brown to greenish black) [12,13] were filtered and washed with deionized water and methanol till getting colorless filtrate. Resulting precipitates dried at 60^0C for to 8 hrs. [14].

D. Pellets designing

The samples of PPY and PPY/ α-Fe_2O_3 so obtained were crushed and finelygrinded using mortal pestle to make fine powder. The pellets of thickness 3 to 4 mm were designed in die-punch (13 mm diameter) using hydraulic press at the pressure of 15 tons.

E. Measurement and characterization techniques

All samples were characterized by FTIR and UV-vis spectroscopy. Their morphological and structural study has done by SEM technique. Four probe set up (DFP-RM -SES Roorkee - India) was used to examine dc electrical conductivity of all sample pallets. The discussion in detail regarding the results of FTIR, UV-vis spectroscopy (IIIA, IIIB), SEM (III C) and dc conductivity (III D) has been given in the following part III.

III. RESULT and DISCUSSION

A. FTIR spectra

FTIR spectra (Fig.1) of PPY and PPY/ α-Fe_2O_3 composites are enriched with absorption peaks attributes the typical characteristics of PPY which are in the range of 4000-500 cm^{-1}consistent with literature.Figure 1(a) shows a typical FTIR spectrum of PPY granules. The peaks at 3434.34 cm^{-1} attribute to free N-H stretching vibration mode of secondary amines having aromatic structure. The peaks at 1543.31 $cm^{-1}$and 1452.30 cm^{-1} corresponds to the stretching vibration of C=C and the C-C of PPY respectively. The band of C-N and C-H in plane deformation vibration are located at 1174.42 cm^{-1} while the band of =C-H out of plane deformation vibration indicating polymerization of pyrroleat 910.74 cm^{-1}. The strong absorption bands in the region 805–1591cm^{-1}arecorresponds to the characteristics of PPY[15].

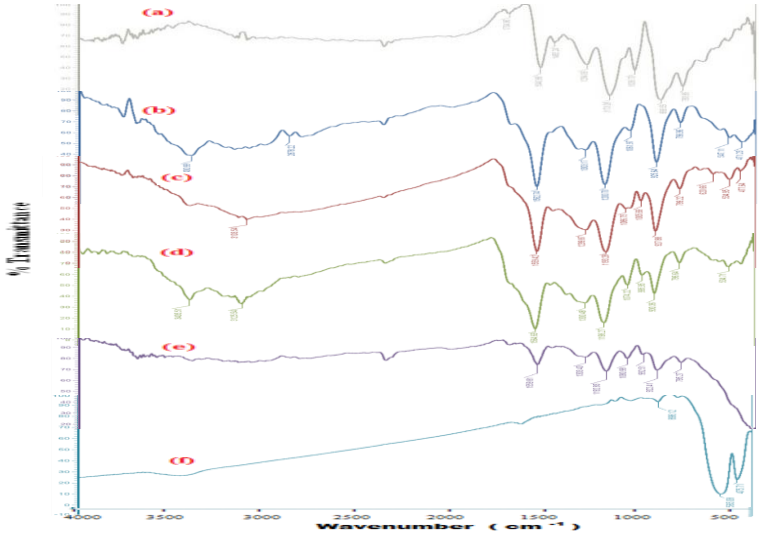

Figure 1:FTIR spectra of (a) Pure PPY, (b) PPY/15% α-Fe_2O_3, (c) PPY/20% α-Fe_2O_3, (d) PPY/25% α-Fe_2O_3 (e) PPY/30%Fe_2O_3 & (f) α-Fe_2O_3

Figure 1(f) shows IR spectra of α-Fe_2O_3 with a characteristic IR peak being observed at 565.08 cm^{-1}could assigned to M-O lattice vibration. The band at 898.12 $cm^{-1}$is assigned the Fe-O-Fe bending vibration[16].

Figure (1b-1e) shows FTIR spectra of PPY/ α-Fe_2O_3composites. In case of PPY/ 20%α-Fe_2O_3, the peak at 3405.21$cm^{-1}$and 3125.54cm^{-1} attribute to N-H stretching vibration mode of secondary amines and =CH-H alkene respectively. The peak at 1300.48 cm^{-1} is due to fundamental vibration of PPY ring. The peak at 1564.69 cm^{-1} attribute to C=C stretching. The peak at 1074.22 cm^{-1} refers C-H in plane bend and peak at 1198.27cm^{-1} corresponds to ring breathing. In addition to these peaks two peaks are noticed at 930.36cm^{-1} and 796.64 cm^{-1} in nanocomposite (NC) which assigned the O-H bend and C-H out of plane deformation[17]. The peaks match well with the previous studies. It is clear from FTIR spectra that NCs have almost identical peaks those associated with pure PPY.With α-Fe_2O_3 loading in NCs, the identity peaks of PPY for bonds C=C, C-N, C-H and =C-H shows the alternate red and blue shifts with increasing wt% of Fe_2O_3 in the NCs. Identity peak of C-C bond on PPY IR spectra suppressed in the NCs with the loading of Fe_2O_3. Thus we ensure the better intercalation of Fe_2O_3 with PPY in the NCs.

B. UV- Visible spectra:

Each of spectra reveals the presence of two absorption band one in visible region and other in UV region. For pure PPY (Figure 2 a) the band in UV region at 357 nm having absorption at 1.501 corresponds to the π- π^* transition and the band in visible region at 533nm having absorption at 1.4349 correspond to the n - π^* transition. The band in the visible region corresponds to their ring charge transfer associate with benzonoid to quinoid (interchain) moieties for PPY[18].

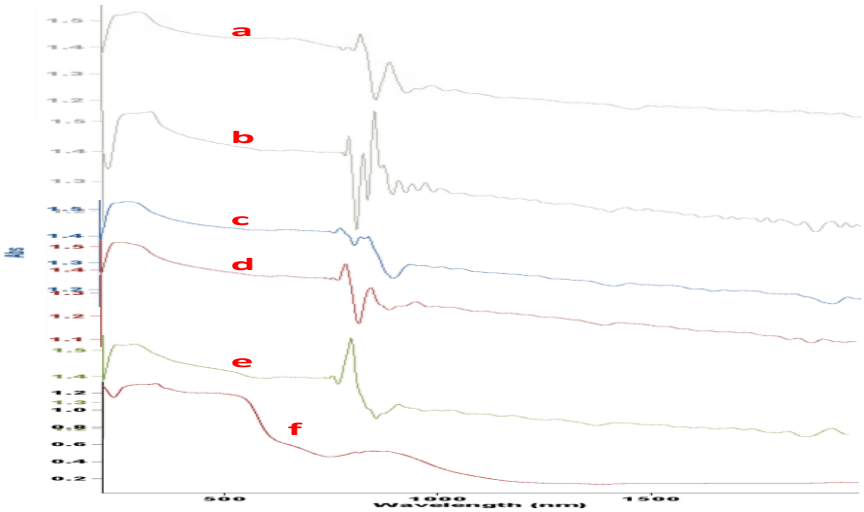

Figure 2:UV-Vis spectra of (a) Pure PPY, (b) PPY/15% α-Fe_2O_3, (c) PPY/20% α-Fe_2O_3,(d) PPY/25% α-Fe_2O_3 (e) PPY/30%Fe_2O_3 & (f) α-Fe_2O_3

Figure 2 (f), UV-vis spectra of α-Fe_2O_3, shows the absorption peaks at 363 nm (UV region) and 747nm (visible region) having 1.3241 and 0.5273 absorption respectively. The purchased Fe_2O_3 is of hematite (α-nature) has been supported by reported data [19,20]

Figure 2 (b-e), spectra for PPY/ α-Fe_2O_3 NCs, shows thepeaks at336 nm and 787nm for PPY/ 15% α-Fe_2O_3, assigns π -π* transition (intrachain transitionsin benzonoid ring) and n - π*transition (interchain hopping from benzonoid to quinoid – HOMO to LUMO or exciton absorption in quinoid rings -) with absorptions 1.533 and 1.451 respectively. It shows good agreement for conjugated polymer chain in PPY/ α-Fe_2O_3 NCs.Shift in λ_{max} value for most of composite samples due to their structural changes arises due to loading of Fe_2O_3[21,22]. With increasing wt% of Fe_2O_3 in the NCs, the absorption peaks in the UV region shows blue shift up to 25% loading of Fe_2O_3 and again shows red shift for further loading up to 30%. Alternate blue and red shifts are observed in the visible region for increasing weight percent of Fe_2O_3 in NCs. It reveals the better agreement for the agglomeration between PPY and Fe_2O_3.UV-vis absorbance data and band energy (eV) of PPY, α-Fe_2O_3 and PPY/ α-Fe_2O_3 NCsare given in table 1. The calculated values of transition band energy and exciton band energies of PPY are 3.801eV and 1.612 eV respectively. The band energies of NCs are found to be lowered than PPY for 15% wt of Fe_2O_3 in NCS. With rise in wt% of Fe_2O_3 in NCs, the transition band energy has found to be increases up to 25% wt of Fe_2O_3 and decreases for further addition of Fe_2O_3 in NCs. With rise in wt% of Fe_2O_3 in NCs, the exciton band energy has found to be increased.

Table: 1 – UV-Vis absorbance data and band energy of PPY, α-Fe_2O_3 and PPY/ α-Fe_2O_3 NCs

Samples	λ_{max}(nm)		Absorbance		Band energy (eV)	
	π - π* Transition Band	n- π* Exciton Band	π - π* Transition Band	n- π* Exciton Band	π - π* Transition Band	n- π* Exciton Band
PPY	327	771	1.534	1.401	3.801	1.612
α-Fe_2O_3	340	849	1.31	0.525	3.656	1.464
PPY/ 15%α-Fe_2O_3	336	787	1.533	1.451	3.699	1.579
PPY/ 20%α-	309	778	1.528	1.43	4.023	1.597

Fe_2O_3						
PPY/ 25%α-Fe_2O_3	282	800	1.518	1.426	4.408	1.553
PPY/ 30%α-Fe_2O_3	320	773	1.524	1.403	3.884	1.608

SCANNING ELECTRON MICROSCOPE (SEM) ANALYSIS

SEM technology has used toanalyze the size and shape of present CP, additive and NCs. Figure 3 (a), SEM image of PPYshows the granule clusters having spherical morphology with 211 nm in average diameter. Formation of smooth and homogeneous PPY granules can be clearly seen [23].

Figure 3 (b-e),SEM images of PPY/ α-Fe_2O_3 NCs shows the Fe_2O_3 nanoparticles are covered by spherical granules of PPY and forms multiple aggregation because of weak interparticle interaction. It shows the presence of spherical particle cluster of polymer in the composite which revels the presence of Fe_2O_3in PPY. The average size of PPY/ α-Fe_2O_3 NCs is about 107 nm and gives small decrease in granule size by adding the wt% of Fe_2O_3[24].

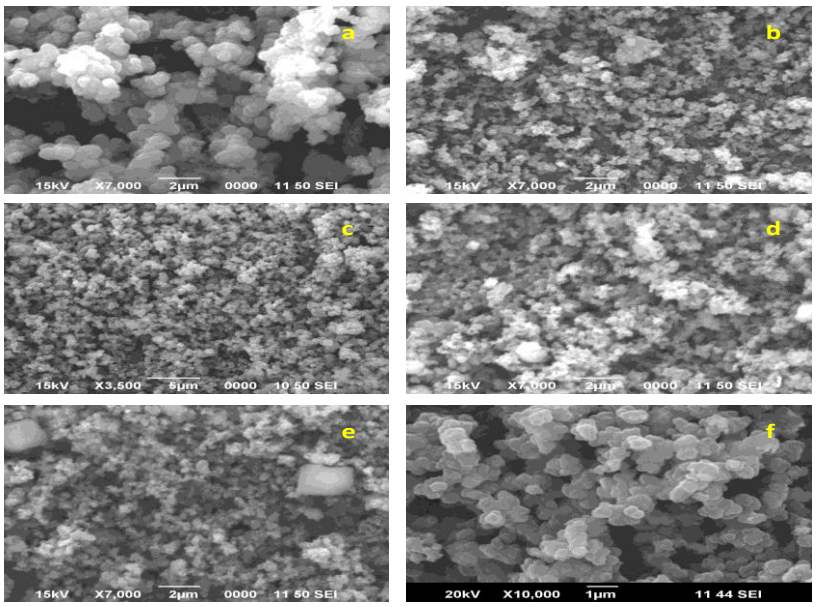

Figure 3: UV-vis spectra of (a) Pure PPY, (b) PPY/15% α-Fe_2O_3, (c) PPY/20% α-Fe_2O_3, (d) PPY/25% α-Fe_2O_3 (e) PPY/30%Fe_2O_3 & (f) α-Fe_2O_3

M.M. Rahman Khan et al (2012) reported the loading effect of CuO on the morphology and diameter of PANI/CuOnanofibers (NFs). Smallest diameter of NFs has been reported for the lowest loading of CuO in NFs. The loading of appropriate amount of metal oxides can be directly control the particle size of CPCs [25].Thus, the addition of CuO always gave NFs with regular and uniform surface morphology whether the loadings of CuO is lower or higher.

Continuous increase in the volume of the core shell in PANI/TiO_2composites with increase in percentage of nanocrystalline TiO_2 in polyaniline matrix has reported by AmeenaParveen et al (2013) [26]. The composite particles are clustered, homogeneous and circular in shape and have almost similarin size.

Similarsize control of CPC by the loading of metal oxide has been observed in the present study, but the smallest particle size of CPC is for lowest wt% (15%) and highest wt% (30%) of metal oxide Fe_2O_3in NCs. Overall, no much change in particle size has observed with loading of Fe_2O_3 but the composite particles are clustered, homogeneous and nearly spherical in shape and have almost similar in size.

C. ELECTRICAL DC CONDUCTIVITY

Fig. 4 displays the temperature dependence of the DC conductivity of samples of PPy and NCs. The dc surface conductivity measurements of the pure PPY and PPY/ α-Fe_2O_3NCs were observed with decreasing temperature from 368 K to 298 K. It was observed that the electrical conductivity of the sample increases with increase in temperature and the values were in the order of $10^{-1}$to 10^{0} S cm^{-1} for PPYcomposites whereas 10^{0} Scm^{-1} for PPY/α-Fe_2O_3 which lies in the semi-conductor region[27] and they followed the Arrhenius equation similar to other semiconductors. Conductivity of NCs increases with increasing molar concentration of Fe_2O_3 up to 25% wt. mole may be due to a high polarization rate [28]. For further increase in molar weight of Fe_2O_3 up to 30 % the dc conductivity of NCs decreases. The decrease in conductivity at 30 %wt of metal oxide is may be due to low polarization rate. The change in dc conductivity of CPCs with metal oxides additive wt% has been reported in our previous study [29].

Conductivity of PPY and its NCsincreases with increase in temperature. It may be due to a hopping mechanism between coordinating sides, local structural relaxations, segmental motionsand thus overall "thermal activated behavior", following (VRH model) [30, 31, 32] Mott's equation

$$\sigma = \sigma_0 \exp [- (T_0 / T)^{1/n+1}], n=1,2,3 \quad \text{---------} \quad (1)$$

Where, σ is the conductivity of the sample at temperature T (K), σ_0 is the pre-exponential factor (S/cm), T_0 is the characteristic Mott temperature (K)and n = 3, 2,1 have to use for three, two and one dimensional nanosystemsrespectively. In our present study, as the samples are of three dimensional structure, we have to use n =3.

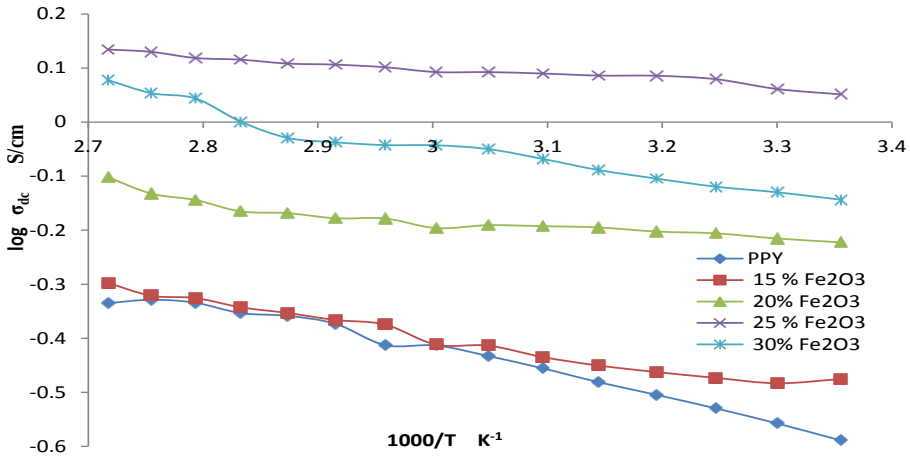

Figure 4:Electrical conductivity $\sigma_{dc}$of (a) Pure PPY, (b-e) PPY/15% α-Fe_2O_3

IV. CONCLUSION

The PPY and composites PPY/α-Fe_2O_3 have been successfully synthesized using Chemical Oxidation Method. The FTIR spectra peaks of PPY and PPY/ α-Fe_2O_3composite with different molar percent of α-Fe_2O_3shows formation of CP and NCs using pyrrole monomer. FTIR of composites shows presence of metal oxide and it involve chemically in PPY. UV-Vis spectra show formation of absorption band in both UV and Visible region, and exhibited a good support with the conductivity result. SEM image shows granular surface morphology of PPY. SEM images of PPY/ α-Fe_2O_3 NCs also shows its homogeneous granular distribution (nearly spherical) of decreasing particle size with increasing molar percent of α-Fe_2O_3. Electrical conductivity σ (dc)decreases with decreasing temperature. The average surface dc conductivity of PPY at room

temperature is 0.4128 S/cm and dc conductivity of NCs increases with increase in molar concentration of Fe_2O_3in composites.

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