



# Synthesis, Characterisation and Conductivity Studies of Polypyrrole- $\text{Nb}_2\text{O}_5$ Composites

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**Abstract:** Conducting Polypyrrole-Niobium Pentoxide (PPy- $\text{Nb}_2\text{O}_5$ ) composites were synthesized by in situ deposition technique by placing different weight percentages of  $\text{Nb}_2\text{O}_5$  powder (10, 20, 30, 40 and 50%) during the polymerisation of pyrrole. The PPy- $\text{Nb}_2\text{O}_5$  composites were later characterised by Fourier Transform Infrared spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). FTIR studies confirm the presence of PPy in the composite. The SEM studies exhibit an aggregated granular morphology of the synthesized composite. The dc conductivity was studied in the temperature range 45-160 °C. It was observed that the conductivity remains nearly constant up to 100 °C and thereafter increases exponentially. Such behaviour is the characteristic of amorphous materials.

**Key words:** Conducting polymer, Polypyrrole, Composite, Conductivity.

## 1. INTRODUCTION:

During the last decade, interest in the development of conducting polymers such as polyaniline, polypyrrole, polythiophene, polyphenylene, etc has increased tremendously because of their electrochromic properties and many commercial applications such as batteries, electronic devices, functional electrodes, electro chromic devices, optical switching devices, sensors and so on [1-5]. As these polymers can substitute for conductors and semiconductors in a variety of electric and electronic devices, they are paid a considerable attention in the recent past. Preparation of these polymer composites with some suitable compositions of one or more insulating materials led to desirable properties [6].

Among the conducting polymers, Polypyrrole is the most representative one because of some special electrical properties. These properties originate from the fact that polypyrrole is an intrinsically conducting polymer and can be synthesized to have conductivities up to  $10^3 \text{ Scm}^{-1}$ , which approaches the conductivity of metals [7]. Polypyrrole has a good environmental stability and facile synthesis [8,9]. Polypyrrole has many commercial applications. They are used in solar cells, electromagnetic shielding, rechargeable batteries, super capacitors, corrosion protection [10]. Apart from these, polypyrrole is widely used in sensor applications because they provide stable and porous matrix for the gas component and also facilitates the e-transfer process [11].

Polypyrrole is synthesised either by oxidative chemical polymerisation [12, 13] or by electrochemical oxidation in aqueous or organic solutions [14].

Transition metal oxides constitute the most fascinating class of materials exhibiting various structures and properties [15]. Composites of electrically conducting polymers and metal nanoparticles are important in modern science and technology due to their potential applications and important physical properties [16].

A great number of nanocomposites between conducting polymers and nanoparticles of different oxides as  $\text{Fe}_2\text{O}_3$ ,  $\text{CuO}$ ,  $\text{SnO}_2$  and  $\text{CeO}_2$  have been reported [17-25].

The present study deals with the synthesis, characterisation and dc conductivity of PPy-  $\text{Nb}_2\text{O}_5$  composites. The samples were characterized by IR and SEM analysis techniques.

## 2. MATERIAL AND METHODS:

### 2.1 Materials:

Analytical-reagent-grade Pyrrole, Niobium Pentoxide and anhydrous Iron (III) Chloride (AR-grade) were obtained and used in the present study. Pyrrole monomer was purified by distillation under reduced pressure and stored in dark at 4 °C.

### 2.2 Synthesis of PPy and PPy- $\text{Nb}_2\text{O}_5$ composites:

For chemical polymerisation of pyrrole,  $\text{FeCl}_3$  was used as an oxidant. Anhydrous ethanol was used as solvent for the PPy synthesis.

3.4 ml of pyrrole was dissolved in 25 ml of ethanol and stirred for 10 min. 25 ml of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  was added drop wise to the ethanol solution of pyrrole. Niobium Pentoxide was varied in weight percentages (10, 20, 30, 40 and 50) and added to the PPy solution. This reaction mixture was stirred for 3 hr with magnetic stirrer in order to disperse  $\text{Nb}_2\text{O}_5$  in the polymer solution. The obtained product was filtered and washed thoroughly with distilled water. To remove last traces of unreacted pyrrole, it was then washed with ethanol. The samples were vacuum dried for 1 hr at 60-70 °C. The powders of PPy and PPy- $\text{Nb}_2\text{O}_5$  composites obtained were crushed and finely ground in agate mortar.

The composites so obtained were pressed in the form of circular pellets of 1 cm diameter and thickness 2.5 to 3 mm. The pellets were coated with silver paste on either side.

## 2.3 Measurements:

The FTIR spectra of all the samples were recorded on Perkin Elmer (model 783) IR spectrometer in KBr medium at room temperature in the region  $4600\text{--}400\text{ cm}^{-1}$ . The powder morphology of PPy and PPy-  $\text{Nb}_2\text{O}_5$  composites was investigated by using Philips XL-30 ESEM scanning electron microscope. The dc conductivity of the components was studied by varying the temperature from  $45\text{--}160^\circ\text{C}$  using a Keithley 6514 electrometer.

## 3. RESULTS AND DISCUSSIONS:

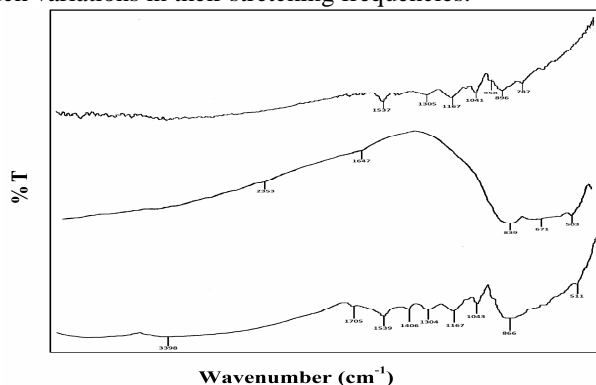
### 3.1 FTIR spectra:

The infra-red spectrum of pure PPy is shown in Fig. 1a. The absorption peak at  $1537\text{ cm}^{-1}$  is due to intra ring C=C and inter ring C-C vibration. The bands at  $1305$  and  $1167\text{ cm}^{-1}$  may correspond to =C-H in plane vibration. The band located at  $1041\text{ cm}^{-1}$  is for the in-plane deformation of C-H bond of pyrrole ring, while the peaks at  $896$  and  $787\text{ cm}^{-1}$  are attributable to =C-H out-of-plane vibrations. These bands are also called as bipolaron bands.

Fig. 1b indicates the IR spectrum of pure  $\text{Nb}_2\text{O}_5$  which shows several significant absorption peaks. The band at  $2353\text{ cm}^{-1}$  originates due to symmetric  $\text{CH}_2$  stretching. The peak at  $1647\text{ cm}^{-1}$  occurs due to C=N stretching originating from amine vibrations within the pyrrole ring.

Fig. 1c shows the IR spectrum of PPy -  $\text{Nb}_2\text{O}_5$  (50 wt %) composite which exhibits absorption peaks at  $1539$ ,  $1406$ ,  $1304$ ,  $1167$  and  $1043\text{ cm}^{-1}$ . The peaks at  $1539\text{ cm}^{-1}$  and  $1043\text{ cm}^{-1}$  are comparatively lower than the peaks  $1537\text{ cm}^{-1}$  and  $1041\text{ cm}^{-1}$  in pure PPy. This is due to the shifting of bands towards a lower wavelength after composite formation. The peak at  $1043\text{ cm}^{-1}$  may be attributed to the C-H vibration of 2, 5-substituted pyrrole which indicates the presence of polymerized (or oxidized) pyrrole in the composite. The band at  $511\text{ cm}^{-1}$  may be due to the metal- oxygen stretching which in turn implies the presence of a metal oxygen bond. The peak at  $503\text{ cm}^{-1}$  in pure  $\text{Nb}_2\text{O}_5$  is shifted to  $511\text{ cm}^{-1}$  in the composite indicating the Van der Waals interaction between polymeric chain and  $\text{Nb}_2\text{O}_5$ .

The IR spectra of other composites (PPy with 10, 20, 30 and 40% of  $\text{Nb}_2\text{O}_5$ ) show similar absorption peaks without much variations in their stretching frequencies.

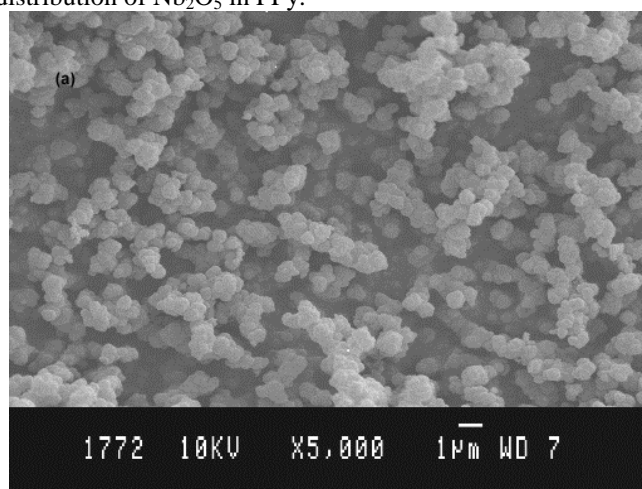


**Fig. 1** FTIR spectra of (a) pure PPy (b) pure  $\text{Nb}_2\text{O}_5$  and (c) PPy- $\text{Nb}_2\text{O}_5$  (50%).

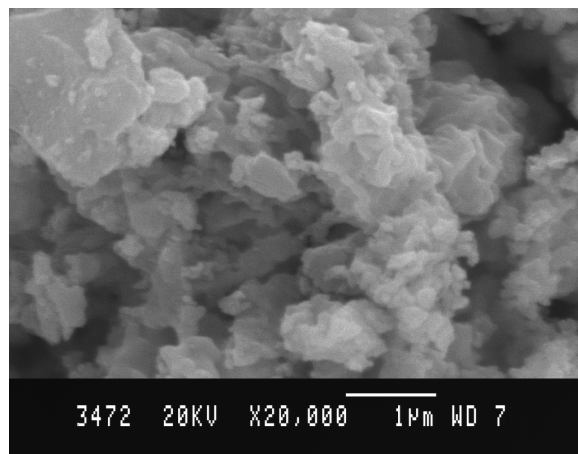
### 3.2 Scanning electron microscopy:

Fig.2a and Fig.2b show the scanning electron micrographs of the pure PPy and PPy-  $\text{Nb}_2\text{O}_5$  (50%) composite respectively. It is seen from Fig. 2b that the cluster and granular structure of PPy is maintained even after the addition of  $\text{Nb}_2\text{O}_5$  in PPy. Hence a network of  $\text{Nb}_2\text{O}_5$  and granular PPy has been formed in case of composites.

A very high magnification reveals the transformation of highly branched PPy to granular structure where  $\text{Nb}_2\text{O}_5$  particle is highly agglomerated with the PPy. The increase of  $\text{Nb}_2\text{O}_5$  in PPy (10, 20, 30 and 40 wt %), increases the granular size and decreases the porosity and confirms the homogeneous distribution of  $\text{Nb}_2\text{O}_5$  in PPy.



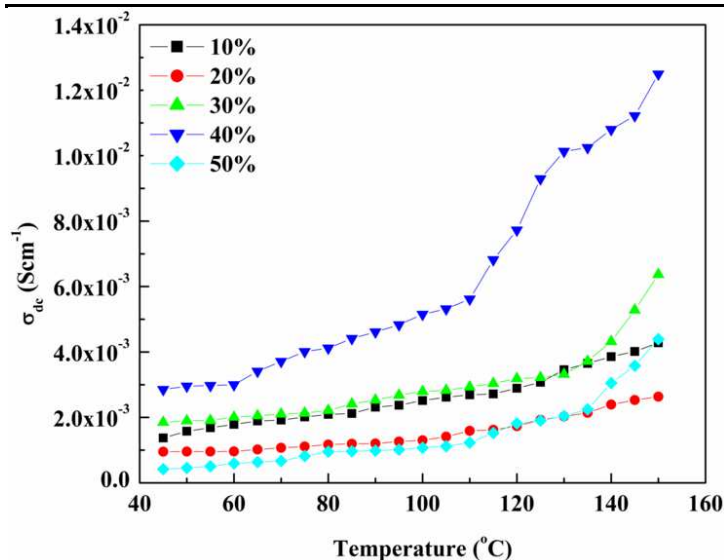
**Fig. 2a** SEM micrograph of Pure PPy



**Fig. 2b** SEM micrograph of PPy- $\text{Nb}_2\text{O}_5$  (50%)

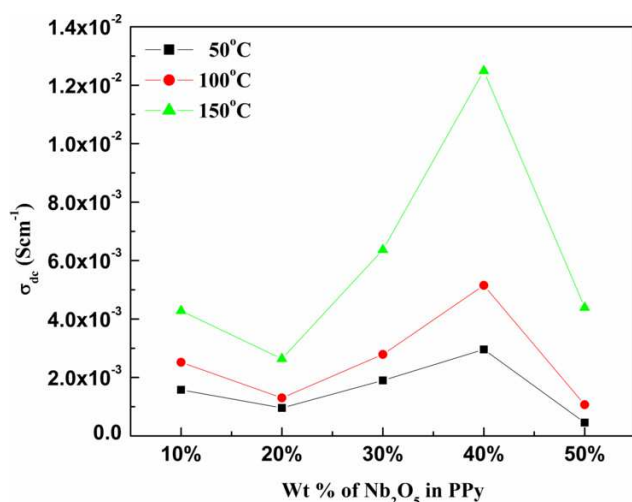
### 3.3 DC Conductivity:

Fig. 3 shows the variation of dc conductivity as a function of temperature for  $\text{Nb}_2\text{O}_5$  in PPy. It is observed that the value of dc conductivity of these composites increases with temperature. It is found to remain nearly constant up to  $100^\circ\text{C}$  and thereafter it increases exponentially. The conductivity reaches maximum for 40 wt% of  $\text{Nb}_2\text{O}_5$ .



**Fig. 3** Variation of dc conductivity ( $\sigma_{dc}$ ) as a function of temperature for PPy-Nb<sub>2</sub>O<sub>5</sub> composites

Fig. 4 shows the variation of dc conductivity as a function of wt% of Nb<sub>2</sub>O<sub>5</sub> in polypyrrole at three fixed temperatures – 50, 100 and 150°C. From the graph, it is observed that the values of dc conductivity of these composites are almost constant up to 30 wt% of Nb<sub>2</sub>O<sub>5</sub> and increases thereafter reaching a maximum at 40 wt% of Nb<sub>2</sub>O<sub>5</sub> and decreases further.



**Fig. 4** Variation of dc conductivity ( $\sigma_{dc}$ ) as a function of wt% for PPy-Nb<sub>2</sub>O<sub>5</sub> composites at different temperatures

At 40 wt% of Nb<sub>2</sub>O<sub>5</sub>, the conductivity is high because of the formation of excess charge carriers (polarons). In this particular weight percentage, polarization due to hopping conduction dominates. The decrease in the values of conductivity may be attributed to the presence of Nb<sub>2</sub>O<sub>5</sub> particles of larger dimensions, which makes the hopping of charge carriers between the favourable sites more difficult.

## 4. CONCLUSION:

PPy- Nb<sub>2</sub>O<sub>5</sub> composites are synthesized by dispersing different amounts of Nb<sub>2</sub>O<sub>5</sub> particles in polypyrrole matrix to study their electrical behaviour. A detailed characterization of the composites has been carried out with IR and SEM techniques. FTIR studies confirm the presence of PPy in the composite. The SEM images of PPy-Nb<sub>2</sub>O<sub>5</sub> show aggregation of particles as well as particle agglomeration. The dc conductivity was studied in the temperature range 45-160 °C. It was observed that the conductivity remains nearly constant up to 100 °C and thereafter increases exponentially. The dc conductivity studies on the composites show the presence of polarons as charge carriers and confirm the extended chain length of polypyrrole.

## 5. REFERENCES:

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