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Synthesis, Characterization and LPG Gas Sensing Response of 5% TiO2 Doped Polypyrrole Nano Composite

Khaire ND[1], Hamjade PT[2], Motke SG[3]

[1]Ex. Associate Professor, Department of Physics, Phulsing Naik Mahavidyalaya, Pusad, Maharashtra, India
[2]Associate Professor, Department of Physics, Phulsing Naik Mahavidyalaya, Pusad, Maharashtra, India
[3]Ex. Principal, Phulsing Naik Mahavidyalaya, Pusad, Maharashtra, India

ABSTRACT

Polypyrrole and its nano-composite is synthesized using in-situ chemical oxidative polymerization technique using pyrrole monomer. Crystalline nano sized TiO2 particles are embedded in amorphous Polypyrrole. Ammonium Per Sulphate is used as oxidizing agent for polymerization. The structure of composite was confirmed by the characterization techniques XRD, FTIR and UV Visible spectra. V-I characteristics and gas sensing response to LPG at room temperature is investigated.

Average grain size and chain separation is determined from XRD. UV-Visible studies show that the composite exhibit absorption peak at 278 nm; which corresponds to band gap energy 4.4633 eV. V-I characteristic is plotted using two probe method, which indicate fairly linear or ohmic behavior of the material with very high resistivity. Gas sensing response to LPG is also observed.

Key words – Polypyrrole, nano-composites, band gap energy, LPG gas sensing, TiO2

I. INTRODUCTION

Conducting polymers have widely been studied in the last two decades due to their potential applications as chemical sensors, gas sensors, electrochemical super capacitors, electro-chromic devices, photo-voltaics, light-emitting diodes, optical computers, microwave absorbers and batteries [1- 5]. In recent years, extensive research has been performed on creating conducting polymer matrix composites with the aim of improving physical and structural properties of conducting polymers. One such class of these, attracting a special attention is to create composites which contain inorganic materials which are usually metals or metal oxides filled into the conducting polymers via various methods. Polypyrrole is one of the most extensively studied conducting polymers due to its high electrical conductivity and chemical stability and as well as its easy preparation through chemical and electrochemical oxidation of pyrrole in the organic solvents and in aqueous medium. [6-9]

Growing industrialization and increasing pollutants from vehicular exhaust have resulted into increased air pollution. The problems related to air quality monitoring are important issues of current research activity. At present, LPG gas is being used in the car, in the storage tank or service station in addition to cooking. But, due to some reasons the LPG gas might leak from the gas cylinders, this may cause severe accident leading to damage of property and live-stock.

To overcome the problem, an LPG gas sensor is required. [10 - 11]

In the present study, polypyrrole/titanium di-oxide 5 % by weight (PPy/TiO_2) nano-composite was prepared by chemical polymerization. TiO_2, which was used as a filler, also have important applications as gas sensors. Samples were characterized by using X-ray diffraction, Fourier transform infrared spectroscopy (FTIR) and ultraviolet-visible spectroscopy (UV-vis). V- I characteristic and gas sensing response to LPG are also investigated.

II. METHODS AND MATERIAL

Pyrrole was supplied by Shah Scientific Mumbai; ammonium peroxydisulphate ((NH4)2S2O8) by Fisher scientific, nitric acid (HNO3) by Loba; TiO2 nanopowder by Nanolab, Jamshedpur, Jharkhand. These materials were used without any preprocessing. In a 50 ml of de-ionized water 0.5 ml concentrated nitric acid, 3.4 ml pyrrole and titanium dioxide (5 % by wt.) were added. The mixture was kept for continuous stirring.

Aqueous solution of oxidant was made by adding 17 gm of ammonium per sulphate into 50 ml of de - ionized water. This solution was added slowly into a mixture of pyrrole-acid-TiO2 in about half an hour. Gradual change in color from light black to dark black indicated formation of PPy/TiO2 nano-composite. After addition of APS solution, the stirring was continued at room temperature for four hours. The mixture was then kept overnight to ensure complete polymerization. The precipitate obtained after polymerization was filtered and washed with de-ionized water several times. The black colored PPy/TiO2 powder so obtained was dried at 50°C for 24 hours [12]

III. RESULTS AND DISCUSSION

Characterization:

XRD

Samples obtained were powdery with black in colour. These were characterized by XRD. The 2θ scans were recorded using Cu - Kα radiation of wavelength 1.5418 A in a range of (5-80) degree as shown in fig. 1.

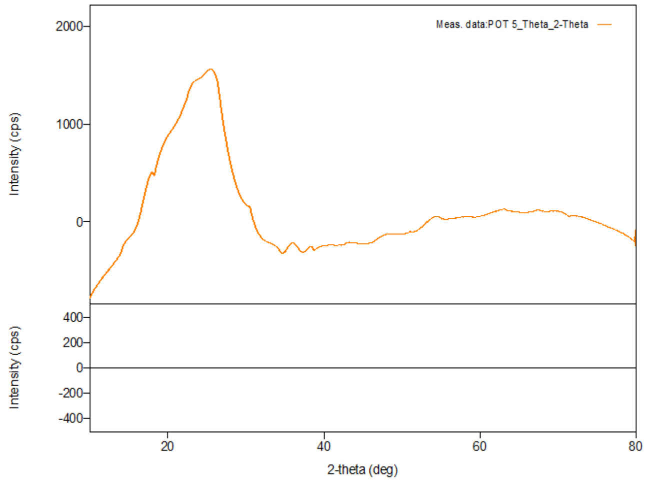

Fig. 1. X-ray diffraction of PPy/TiO_2 (5 %)

X-ray diffraction pattern shows that crystalline TiO_2 is embedded in amorphous poly-pyrrole. Broad peak is found at 2θ = (25.46) °. Average chain separation can be found from the maxima using the relation,

$$S = 5\lambda / 8 \sin\theta$$

Where S is polymer chain separation, λ is x- ray wavelength and θ is the diffraction angle at maximum intensity of amorphous halo. The average chain separation was found to be 4.37 A°.

The average crystallite size, can be estimated by using Scherrer's formula.

$$D = K \lambda / \beta \cos\theta$$

Where D is the crystallite size, K is the shape factor, which can be assigned a value of 0.89 if the shape is unknown, θ is the diffraction angle at maximum peak intensity and β is the full width at half maximum of diffraction angle in radians. When applied to sharp peaks, the equation leads to the average crystallite size of about 10 nm.

FTIR spectra

Infra-red spectroscopy (FTIR) was performed on Shimadzu FTIR 8201 spectrophotometer between 4000–400 cm^{-1} as shown in fig. 2.

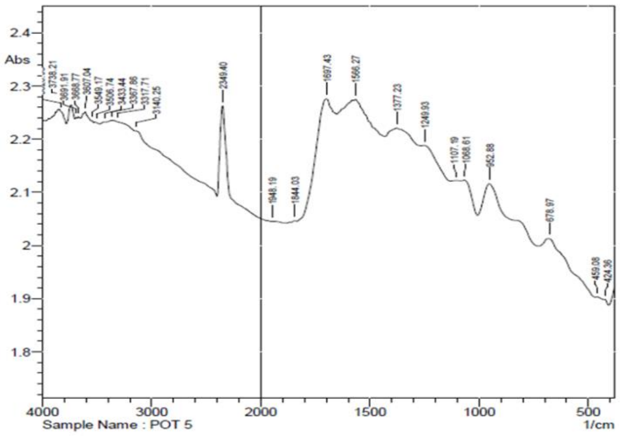

Fig. 2. FTIR spectra of PPy/TiO_2 (5%)

The peaks at around 3368 cm^{-1} indicates N-H stretching, 3318 cm^{-1} indicates C-H stretching, 1697 cm^{-1} indicates C= N bond, 1377 cm^{-1} is related to C-N asymmetric vibration, 1107 cm^{-1} is indicative of = C - H bond vibration, 679 cm^{-1} indicates C= C bending. The peaks at 459 cm^{-1} shows bending and stretching mode of Ti– O–Ti. [13]. The peaks observed in present work match well with literature values confirming formation of poly-pyrrole.

UV-Vis spectra

Ultra violet spectroscopy refers to absorption spectroscopy is made using Shimadzu UV-vis spectrometer between 200–1100 nm as shown in fig 3.

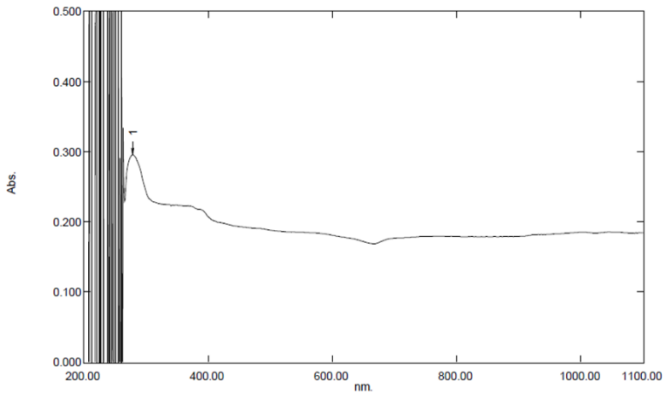

Fig.3. UV-Vis spectra of PPy/TiO_2(5%)

Using these spectra the band gap energy can be found out from the formula,

$$E_g = hc/e\lambda$$

Where h is Planck's constant, c is the speed of light, λ is the wave length and e is the charge of electron. For PPy / TiO_2 UV-Vis spectra exhibit absorption peak is at 278 nm, which corresponds to band gap energy 4.4633 eV.

DC Conductivity

The film of the composite is deposited using spin coating technique on pre-cleaned glass substrate using 8% polyvinyl acetate as a binder. For measurement of conductivity, juxtaposed copper electrodes, each of length 0.6 cm and 1 mm separation between them, are gently placed on the film with the help of loose spring. Pico- ammeter (DPM–111, SES, Roorky) and a dc power supply (Agronic - 93) are used for the purpose.

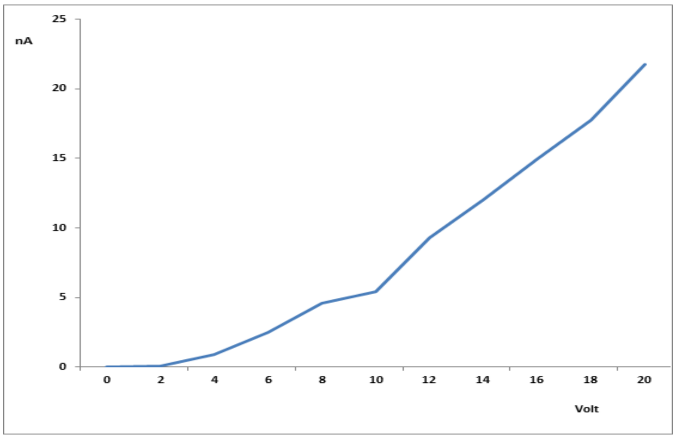

Fig.4. V-I Characteristic of PPy/TiO_2 (5%)

Fig. 4 represents variation of current through surface of sample with voltage. The graph is fairly linear indicating ohmic nature of the sample. As voltage increases the current increases. This may be due to transfer of electrons as charge carriers in the direction of applied field [14]. Surface resistivity is calculated from the formula -

$$\rho = \frac{E}{J} = \frac{V}{I} L$$

where, V is voltage applied, I is surface current and L represents length of the electrode. The surface resistivity, ρ is evaluated as 23.22 M Ω m.

Gas Sensing Response

Gas sensing response of the sensor is defined as change in conductance of a sample upon exposure to target gas to the original conductance in air. The figure shows gas response of PPy/TiO_2 thick film to LPG gas at room temperature.

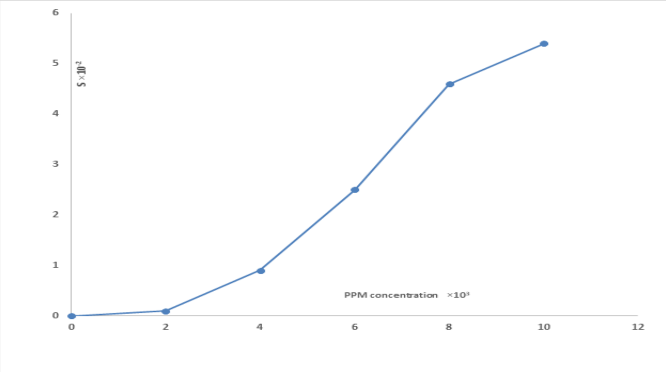

Fig.5. LPG gas response of PPy/TiO_2 (5%)

Fig. 5 shows that sensitivity increases fairly linearly with concentration of the gas. Percentage sensitivity per unit concentration is determined from the slope of linear range, is found to be 2.5866×10^{-4} % per ppm of LPG. The exact fundamental mechanisms to explain gas sensing response are controversial. But decrease in current due to increase in LPG concentration may be due to trapping of electrons in LPG molecule.

IV. CONCLUSION

PPy/TiO_2(5%) nano-composite was successfully synthesized by in situ chemical oxidative method. The characteristic peaks of PPy /TiO_2 nano-composite was observed in XRD, FTIR and UV-Vis spectra. X-ray diffraction pattern shows that crystalline TiO_2 is embedded in amorphous poly-pyrrole. Average chain separation and grain size are in the nanometer range. Band gap energy is also evaluated. DC electrical conductivity is investigated. Gas response to LPG is also determined.

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