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Development of Nanostructured Polypyrrole (PPy) Thin Film Sensor for NO_2 Detection

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Abstract: Polypyrrole (PPy) was synthesized by chemical oxidative polymerization technique using monomer pyrrole and ammonium persulphate as an oxidant in a ratio of 1:1. Polypyrrole was characterized by X ray diffraction (XRD), Scanning electron microscopy (SEM), Fourier transform infra red spectroscopy (FTIR), UV visible spectroscopy and electrical resistivity by four probe method. The XRD and SAED analysis showed that the polypyrrole is amorphous in nature. Field emission scanning electron microscopy showed formation of porous agglomerated granular structure with average grain size of 80-90nm. HRTEM image confirms formation of nanostructured PPy. The FTIR spectra shows that the presence of all characteristics absorption peaks of polypyrrole that is, 926 cm^{-1} (=C–H out-of-plane vibration), 1048 cm^{-1} (=C–H in-plane vibration), 1210 cm^{-1} (N–C stretch bending) and 1478 cm^{-1} (pyrrole ring vibration). UV visible study shows PPy films exhibit absorption peak at 446 nm (2.77 eV). These films were investigated for their sensing behavior towards NO_2, H_2S, NH_3, C_2H_5OH and CH_3OH gases at room temperature. It has been observed that these films are selective for NO_2 gas, and the maximum sensitivity of 14 % (100 ppm NO_2) with 78 % stability at room temperature was achieved. These studies show that polypyrrole films can be used as room temperature NO_2 sensors. *Copyright © 2012 IFSA.*

Keywords: Polypyrrole, Chemical polymerization, XRD, SEM, FTIR, NO_2 sensor.

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1. Introduction

NITROGEN oxides (NO*x*, NO and NO_2) are typical air pollutants exhausting from car engines, boilers, or any other combustion processes. In order to monitor and control the concentration of NO*x* in automotive exhausts, it is eagerly required to develop high-performance compact NO*x* sensor, which should detect NO*x* sensitively and selectively with fast response.

Nitrogen dioxide (NO_2) is important to be detected because it is colorless, flammable, and dangerous, even at very low concentration. Furthermore, the interaction of NO_2 and CO in sunlight tends to produce O_3, which is believed to be harmful to plants and the respiratory system of human beings and animals due to its strongly oxidizing behavior. Therefore, from the public health and environmental protection viewpoint, the sensitive detection of nitrogen dioxide is of great scientific importance. Up to now, the metal oxides films have been widely used for detecting NO_2 gas. However, most of those sensors, lack high sensitivity and operate at high temperature (300–500 ^{0}C) [1-4].

Here we firstly demonstrate that the sensor based on PPy nanoparticles simply prepared by the chemical polymerization has the advantages of sensitivity, spatial resolution, and rapid time response for NO_2 gas at room temperature in supporting our observation on structure, morphology, of polypyrrole thin films prepared using spin coating technique.

2. Experimental Techniques

Polypyrrole (PPy) was synthesized by chemical oxidative polymerization technique using monomer pyrrole. Analytical grade ammonium per-sulphate APS, (Sd fine) was used as oxidizing agent. The chemical polymerization was carried out in a beaker by mixing 0.1 M aqueous solution of pyrrole and 0.1 M of APS in 1:1 ratio by volume. The polymerization was carried out for a period of three hours. After termination of polymerization process, the precipitate obtained was filtered. The product was washed successively by methanol followed by distilled water. The films of polypyrrole were deposited using spin coating method on pre cleaned glass substrate (size 10 mm x 10 mm). X-ray diffraction (XRD) studies were carried out using a Philips powder X-ray diffractometer (Model: PW1710). The XRD patterns were recorded in the 2θ range of 20–80^o with step width 0.02° and step time 1.25 sec using CuKα radiation (λ =1.5406 Å). The morphology of the polypyrrole films was examined using Field emission scanning electron microscope (FESEM) (MIRA3 TESCAN) using acceleration voltage of 20kV.For this purpose thin layer of gold (50Å) was deposited on the film using physical vapour deposition. High resolution transmission electron microscopy (HRTEM) was obtained in order to investigate the morphology of PPy thin films using Hitachi Model H-800. Chemical structure of thin film was examined by FTIR technique (Model: Perkin Elmer 100) using uncoated glass as reference. UV-Vis spectra of the samples, which were dispersed in de-ionized water under ultrasonic action, were recorded on a Simandzu-100 UV-Vis spectrophotometer. The thickness of PPy film (110 nm) is measured using Dektac-150 surface profiler.

In order to measure the gas response, the resistance of the films was measured in air ambient and in gas atmosphere. For resistance measurement, two silver electrodes, separated by 1mm, were deposited on PPy film and silver wires were attached using silver paint. The resistance was measured using Keithley 6514 system Electrometer. For monitoring the response of the films to various gases, the films were mounted in 250 cc air tight container and the known gas (NO_2, NH_3, H_2S, C_2H_5OH and CH_3OH) of particular concentration was injected through a syringe. All the gas sensitivity measurements were carried out at room temperature. Fig. 1 shows photograph of experimental setup for gas sensor measurement

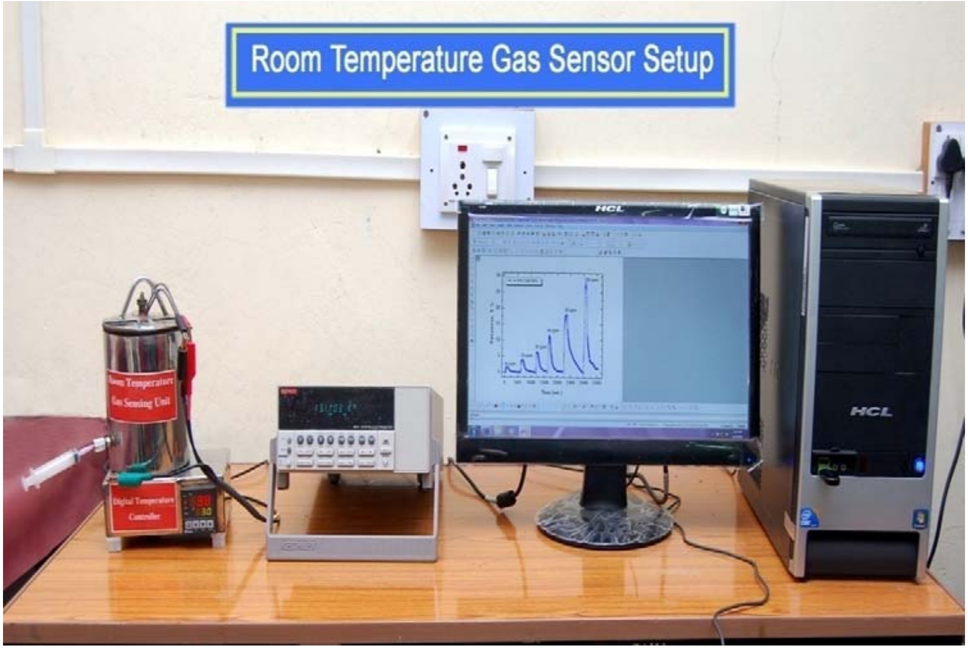


Fig. 1. Photograph of experimental setup for gas sensor measurement.

3. Results and Discussion

3.1. FTIR Analysis

Fig. 2 shows the FTIR spectra of PPy powder. The peaks at 794 cm^{-1}, 926 cm^{-1} are attributed to C-H wagging [5, 6]. The characteristic peaks at 1478 cm^{-1} and 1558.4 cm^{-1} correspond to the C=C stretching, whereas peaks at 1705 cm^{-1} and 1292 cm^{-1} represent to respectively, C=N and C-N bonds [7]. The occurrence of small peaks at 3115 cm^{-1} is assigned to presence of N-H stretching vibrations. The peaks observed in the present work match well with the ones available in the literature [6-7] confirming the formation of polypyrrole.

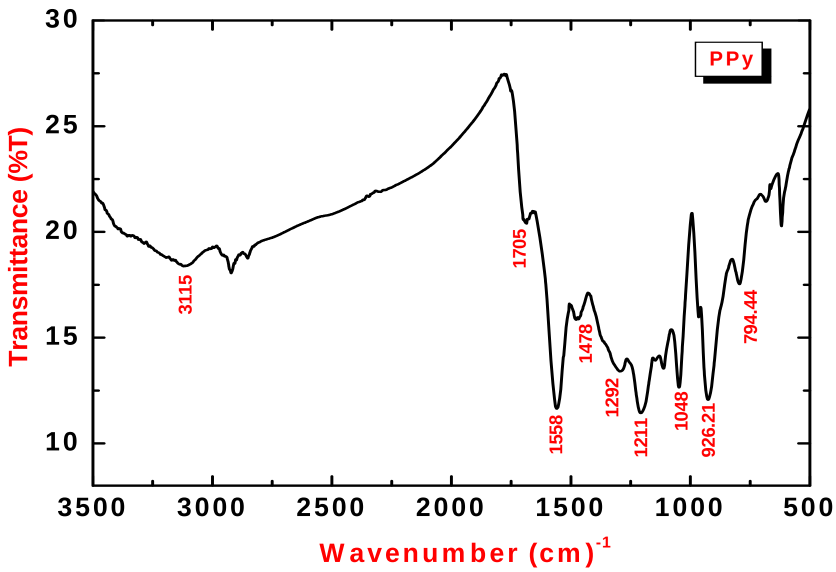


Fig. 2. FTIR spectra of polypyrrole (PPy) film.

3.2. XRD Analysis

X- ray diffraction studies show that the PPy is amorphous in nature, as shown in Fig. 3. In Fig. 4 broad peak was observed at about $2\theta = 24^o$. The broad peak is characteristic of amorphous PPy [8] and is due to the scattering from PPy chains at the interplanar spacing [9].

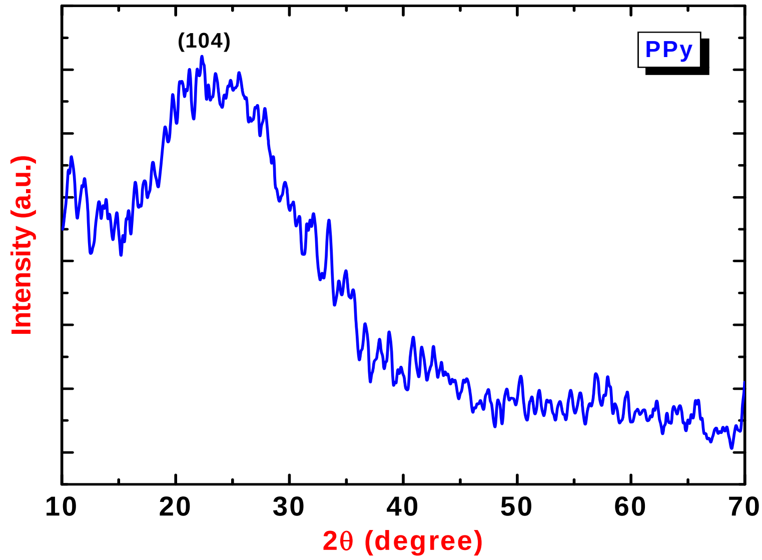


Fig. 3. X-ray diffraction pattern of Ppy.

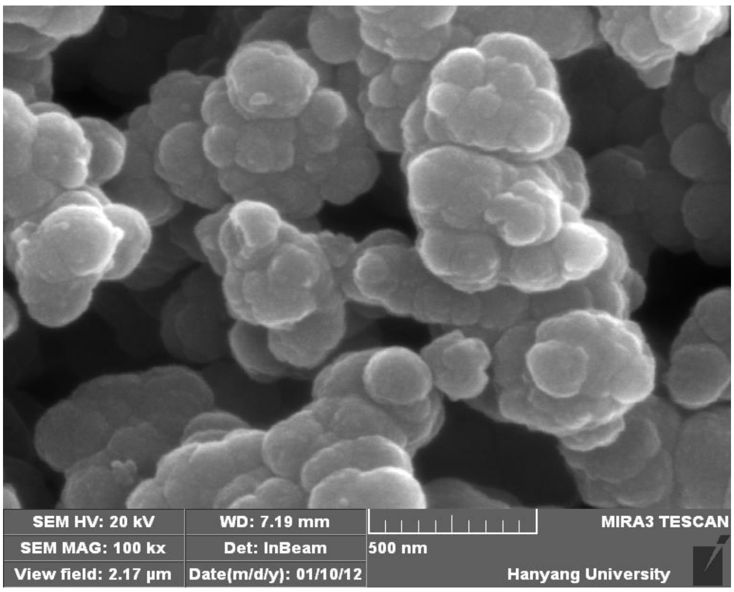


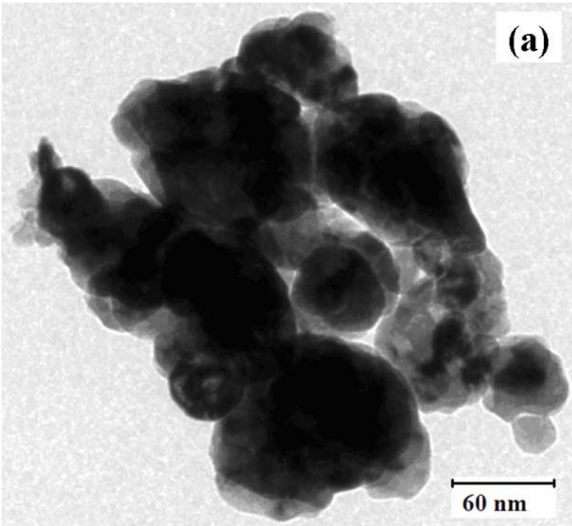
Fig. 4. Microstructure of a polypyrrole thin film.

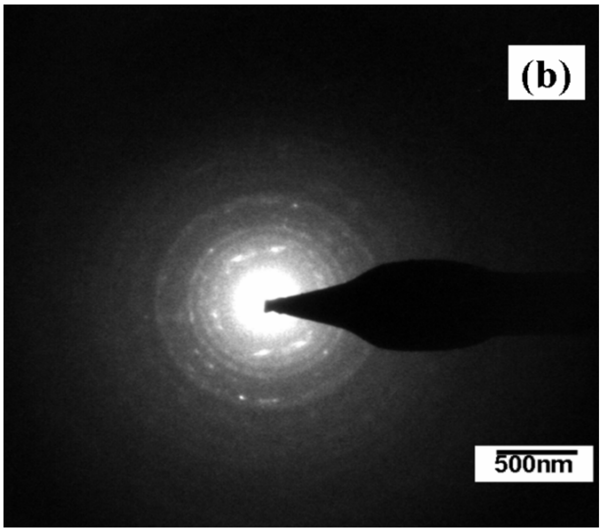
3.3. FESEM Analysis

The microstructure of a polypyrrole thin film is shown in Fig. 4. The film has porous agglomerated granular morphology with average grain size is 80-90 nm. The porous agglomerated granular morphology is preferred for gas sensing applications as it promotes adsorption of gas molecules throughout the film surface [10, 11].

3.4. TEM Analysis

The morphology of strongly interconnected grains of PPy was further studied with high resolution transmission electron micrograph (HRTEM) as shown in Fig. 5 (a). It shows that the film composed of interconnected nanograins of average diameter around 50 nm, which is consistent with grain size determined from SEM studies. Fig. 5 (b) shows corresponding selected area electron diffraction (SAED) pattern of polypyrrole nanograin. The blurred bright electron diffraction rings show that the polypyrrole film is amorphous or poorly crystalline, supporting to X-ray diffraction results (Fig. 3).





(a) (b)

Fig. 5. (a) TEM image of polypyrrole nanograins;
(b) SAED pattern of corresponding polypyrrole nanograin.

3.5. UV-Vis Analysis

Fig. 6 shows UV-Vis spectra of PPy film. PPy films exhibit absorption peak at 446 nm (2.77 eV) which are in agreement with literature values [12, 13].

3.6. Gas Sensing Properties of PPy Thin Film

The gas sensitivity of a film is usually measured as the percentage change in film resistance on gas exposure, or may be defined as the ratio of its resistance in air to its steady state value in the presence of a gas, or vice versa. Irrespective of the definition one uses, it is important to monitor the (change in) electrical resistance of a sensor film. Here, we describe the operation of PPy sensor operating at room temperature.

For measurement of change in resistance on exposure to gases, the sensor head was put in an air-tight chamber of volume 250 ml, a pre-defined concentration of gas taken from calibrated canister of 20-100 ppm was introduced in this chamber by syringe (Fig. 7).

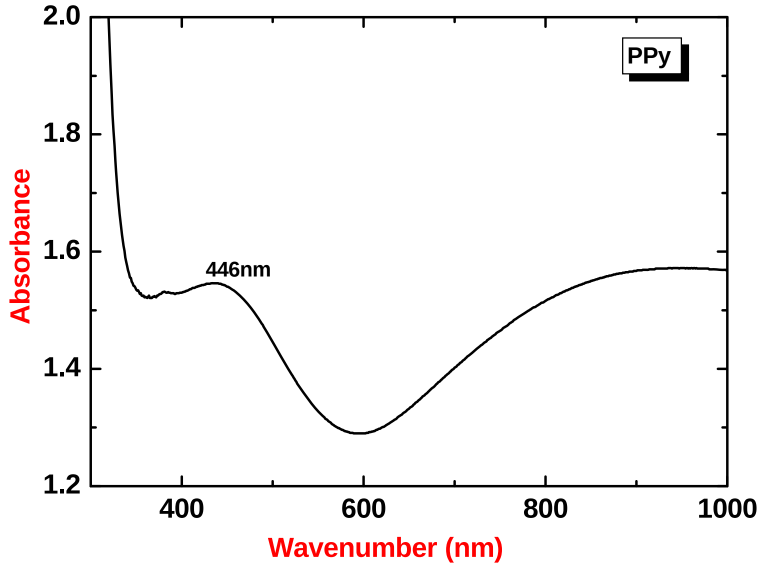


Fig. 6. UV-Vis spectra of PPy film.

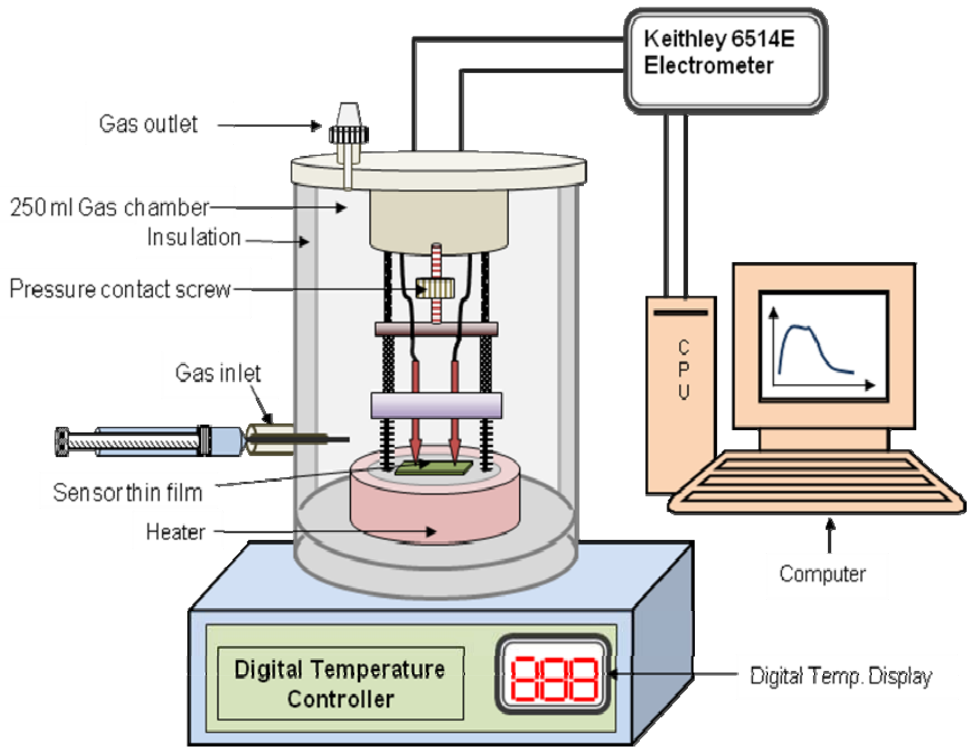


Fig. 7. Schematic of custom designed Gas Sensing set up.

The gas sensing properties of PPy film was carried out for NH_3, CH_3OH, C_2H_5OH, H_2S and NO_2 gases. All the measurements were carried out at room temperature. To determine response and recovery times, a film was exposed to 100 ppm concentrations of NO_2, CH_3OH, C_2H_5OH, H_2S, and NH_3 and its resistance was recorded as a function of time. The response time has been defined as the time taken to attain 90 % of final value, and the recovery time as the time taken to regain 10% of the base value [10, 14]. It was observed that the resistance of the PPy films increases on exposure to H_2S, NH_3CH_3OH andC_2H_5OH gas where as it decreases on exposure to and NO_2 gas.

An attempt was made to study selectivity of PPy films for 100 ppm concentration of NO_2 as compared to the sensitivities of CH_3-OH, C_2H_5-OH, NH_3 and H_2S.

The ability of a sensor to respond to a certain gas in the presence of other gases is known as selectivity. The selectivity of a sensor in relation to a definite gas is closely associated with its operating temperature. Here, the selectivity measured in terms of selectivity coefficient/factor of a target gas to another gas is defined as $K=S_A/S_B$, where S_A and S_B are the responses of a sensor to a target gas A and an interference gas B, respectively.

Fig. 8 shows the bar diagram of gas response to different gases. The PPy sensor offered maximum response towards NO_2 (14 %), NH_3 (12 %), H_2S (1%), CH_3OH (0.3 %), and C_2H_5OH (0.2 %) at room temperature. This may be due to the different gases have different energies for reaction to occur on the surface of PPy. Thus PPy sensor is highly selective to NO_2 over C_2H_5OH compared to NH_3, H_2S, CH_3OH [(S_{NO2}/S_{C2H5OH}=70), (S_{NO2}/S_{CH3OH}=46), (S_{NO2}/S_{H2S}=14), and (S_{NO2}/S_{NH3}=1.17)] at room temperature. Therefore, further dependence of NO_2 response is studied for various concentration of NO_2 at room temperature.

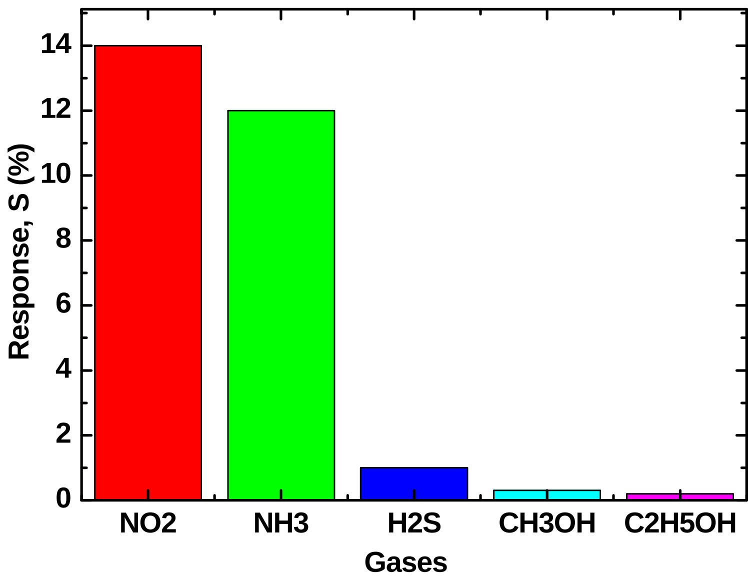


Fig. 8. Gas responses of PPy sensor film to 100 ppm of NO_2, NH_3, H_2S, CH_3-OH and C_2H_5-OH.

Typical gas response curves of PPy films for 20-100 ppm concentrations of NO_2, is shown in Fig. 9.

The gas response is defined as:

$$\text{Response (S) (\%)} = \left| (R_{gas} - R_{air})/R_{air} \right| \times 100\ \%$$

It is observed that PPy films can sense NO_2 with higher sensitivity value as compared to other gases. It is observed that PPy film is sensitive as well as fast in responding to NO_2. The response for NO_2 is better by a factor of ~ 14 and also faster than other gases. A high sensitivity for NO_2 indicates that the PPy films are selective for this gas. The higher sensitivity towards NO_2 than NH_3, CH_3OH, C_2H_5OH, and H_2S, can be explained on the basis of different interactions between sensing film and adsorbed gas. PPy is p-type materials and when it interacts with NO_2 (oxidizing gas) there is increase in charge carrier density. This results in increasing the conductivity of material and film resistance decreases. In case of reducing gases like NH_3, CH_3OH, C_2H_5OH and H_2S there is a decrease in charge carrier concentration and thereby decreasing conductivity. The interaction of H_2S, CH_3OH, C_2H_5OH and $NH_3$with PPy film is very less as compared to NO_2; hence it shows very slow response and less sensitivity.

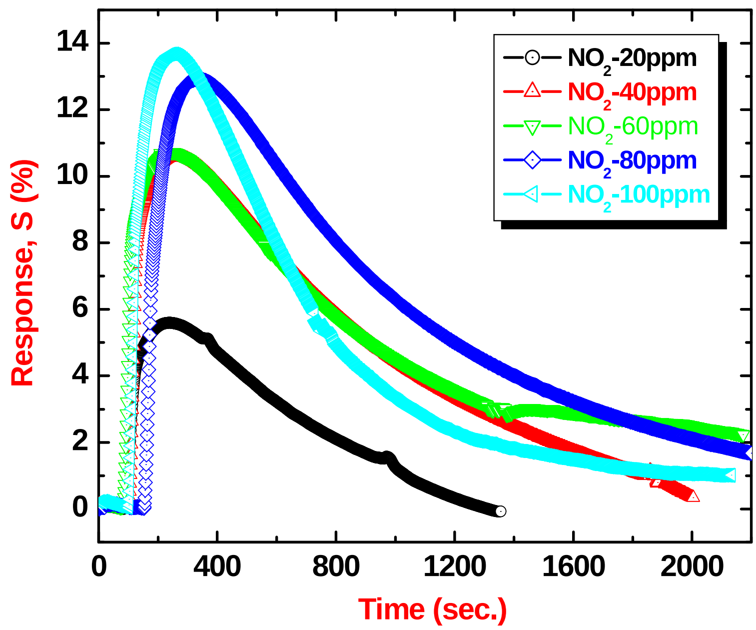


Fig. 9. Gas response curve of PPy films for 20-100 ppm NO_2

The response values of PPy sensor film is plotted as a function of NO_2concentration in Fig. 10. It is observed that the response slows down at higher concentration; this may be due to less availability of surface area with possible reaction sites on surface of the film.

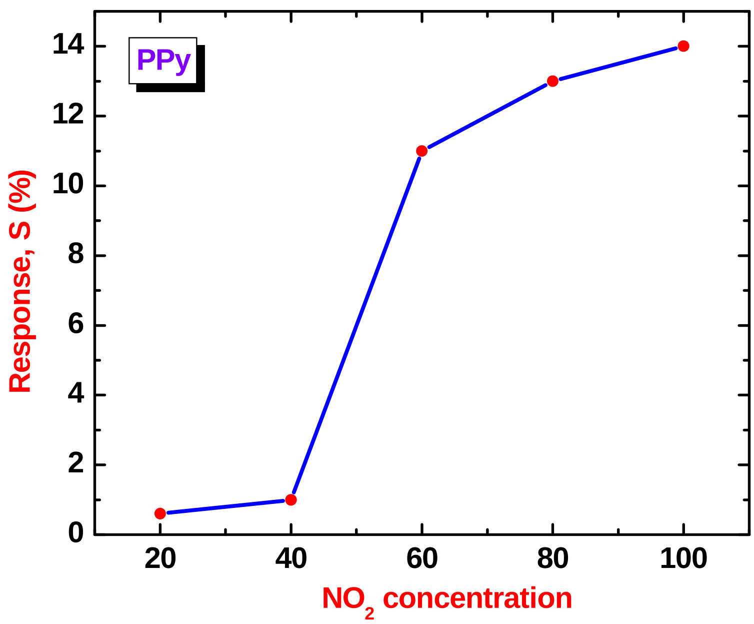


Fig. 10. Gas response of PPy thin film sensor to NO_2 (20- 100 ppm).

The response and recovery time is an important parameter used for characterizing a sensor. Fig. 11 shows response and recovery curve of PPy sensor for different concentration of NO_2 (20-100 ppm). It is revealed that the response time decreased from 132 to 122 sec when NO_2 concentration increased from 20 to 100 ppm. This may be due to the sufficient gas molecules on the surface of PPy for reaction to occur. From Fig. 11 it is also seen that for higher concentration of NO_2, the recovery time was long. This may probably due to the heavier nature of NO_2 and the reaction products are not leaving from the surface immediately after the reaction [10, 14].

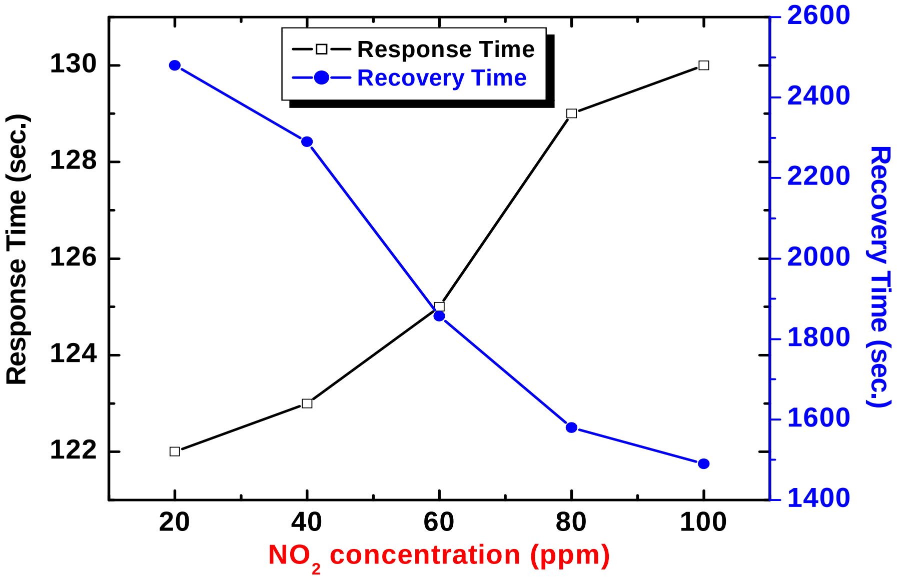


Fig. 11. Response and recovery curve of PPy sensor for 100 ppm NO_2.

In order to check the stability of PPy sensor, the change in resistance is studied at room temperature upon exposer of fixed concentration (100 ppm) of NO_2 for 30 days at an interval of 5 days, after the first measurement and the results of gas response are shown in Fig. 12. Initially PPy sensor showed relatively high response, however it dropped from 14 to 11 % and stable response obtained after 15 days (78.57 % stability). This is because in the initial stage PPy sensor may undergo interface modification during operation and then reaches to steady state indicating the stability of the PPy sensor operating at room temperature.

4. Conclusion

The Polypyrrole thin films have been deposited on glass substrate by spin coating technique. The polypyrrole synthesized using chemical polymerization process. The films had a uniform granular morphology. The structure of the films is confirmed by FTIR and XRD techniques. UV-Vis studies showed that the PPy films exhibit absorption peak at 446 nm (2.77 eV). The changes in sensitivity of the PPy sensor elements in response to NO_2 gas were evaluated and compared with NH_3, H_2S, CH_3OH, C_2H_5OH .A good sensitivity and response time was found towards NO_2 gas in the concentration range of 20–100 ppm at room temperature. The variations of the sensitivity of the developed sensors have been found to be rather due to the microstructure of the PPy thin films. The spin coating processed PPy thin layers have the potential to be further investigated and used as novel NO_2 gas sensor stable elements at room temperature due to their good sensing properties for compelling safety and environmental needs for low-cost, mass market gas sensors.

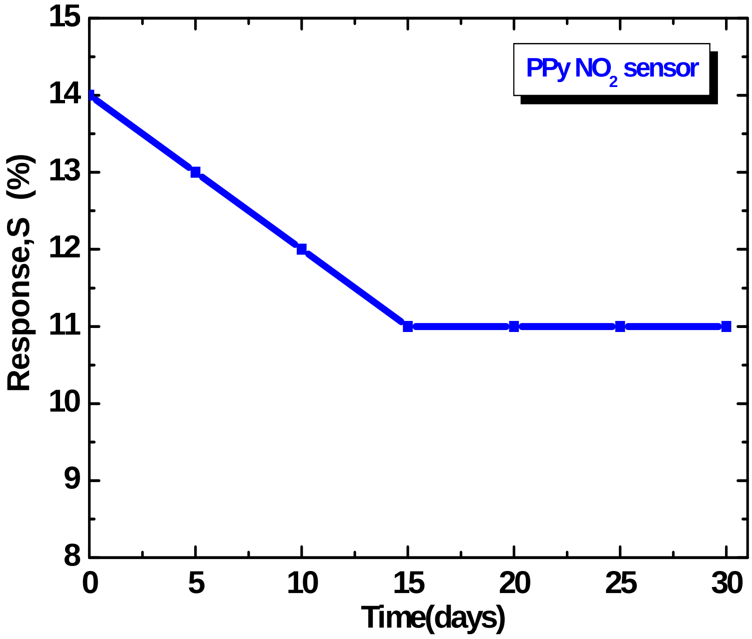


Fig. 12. Stability curve of PPy sensor operating at room temperature.

Acknowledgement

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Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726-5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because of it is a peer reviewed international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per year by International Frequency Sensor Association (IFSA). In additional, some special sponsored and conference issues published annually. *Sensors & Transducers Journal* is indexed and abstracted very quickly by Chemical Abstracts, IndexCopernicus Journals Master List, Open J-Gate, Google Scholar, etc. Since 2011 the journal is covered and indexed (including a Scopus, Embase, Engineering Village and Reaxys) in Elsevier products.

Topics Covered

Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

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