

# Development of Room Temperature Operating NH<sub>3</sub> Gas Sensor Based on Free Standing PPy-PVA Composite Films

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**Abstract:** In the present investigation, room temperature operating ammonia (NH<sub>3</sub>) gas sensor based on free-standing Polypyrrole-Polyvinyl alcohol (PPy-PVA) composite films has been fabricated. The PPy-PVA composite films were synthesized by an in situ chemical polymerization of Pyrrole in the presence of PVA. The synthesized films were characterized by Scanning Electron Microscopy (SEM), Fourier Transforms Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD). The DC electrical conductivity ( $\sigma$ ) of the composite films has been investigated by two probe method and was found to be  $2.16 \times 10^{-3}$  S/cm at room temperature. Ammonia gas sensing behaviour of films was studied. Gas sensing tests demonstrated that the composite films exhibited a fast response, rapid recovery and high sensitivity at room temperature with a detection of low concentration (25 to 300 ppm) of ammonia gas. This work demonstrates the potential of using PPy-PVA composite films as sensing material in the fabrication of room temperature operating ammonia sensor.

**Keywords:** Polypyrrole, Polyvinyl alcohol, SEM, FTIR, XRD, Room temperature ammonia sensor.

## 1. Introduction

Ammonia is a colourless gas which is characterized by its pungent and suffocating odour. The major part of ammonia produced by the industries is used in agriculture as fertilizer. Ammonia is also utilized as a refrigerant gas, for purification of water supply and waste water treatment, in the manufacture of : plastics, pulp and paper industry, explosives, textiles, pesticides, dyes, food processing, medical diagnosis, fire power plants, etc. [1]. Although easily available in nature and widely used, ammonia is harmful as well as toxic in its concentrated form. Ammonia is amongst a group of toxic gases having permissible exposure limit (PEL) as 50 ppm, threshold limit value (TLV-8 hours): 25 ppm and short-term exposure (STEL -15 minutes): 35 ppm [2].

A leak in the ammonia production system can cause serious health hazards. Exposure to high concentration of ammonia irritates the respiratory tract and can cause chronic lung diseases, bronchiolar or alveolar edema, etc. Ammonia is one of the causes of air pollution, which is a global issue. In electronic industries, ammonia is widely used in the preparation of silicon nitride (Si<sub>3</sub>N<sub>4</sub>) and gallium nitride (GaN) by chemical vapour deposition leading to air pollution [3]. Therefore, all industries working with regards to ammonia should have a detection system which would warn when the concentration of ammonia would reach to dangerous limits. As per the points discussed above, it is necessary to be alerted even if a small quantity of ammonia leaks. Therefore, it is necessary to monitor the gas and hence develop ammonia detecting sensors that would function at room temperature.

In recent years, the conducting polymer- polypyrrole has been studied as a sensing material essentially due to its operation at room temperature [2, 4-8]. PPy can be easily synthesized by chemical or electrochemical oxidation of the

pyrrole monomer. However, its poor mechanical strength is the main limitation. This limitation can be overcome by preparing the conductive composites which possess the electrical properties of PPy and the mechanical properties of the insulating host matrix. PVA is one of the important polymers and is available in powder as well as fiber form. The PPy-PVA composite films combine the advantageous mechanical properties of the host polymer with the electrical properties of PPy [9]. These composite films have not been explored much for ammonia gas sensing. Thus, it is worthwhile to modify the brittle PPy with PVA for improving its gas-sensing properties as well as its stability.

In the present investigation, efforts have been made to develop ammonia sensor using PPy-PVA composite films synthesized by an in situ chemical polymerization of Pyrrole in the presence of PVA using FeCl<sub>3</sub> as an oxidant. The structure and surface morphology of the PPy-PVA composite films have been analyzed by SEM, FTIR and XRD respectively. We have also measured dc conductivity behaviour as a function of temperature using two probe method. PPy-PVA composite films have shown good responses to low concentrations (25–300 ppm) of NH<sub>3</sub>, with operating at room temperature. We have determined the response time, the recovery time and repeatability of the response. The experimental results obtained, demonstrate that the PPy-PVA composite films can be used for fabrication of NH<sub>3</sub> sensor operating at room temperature.

## 2. Experimental Technique

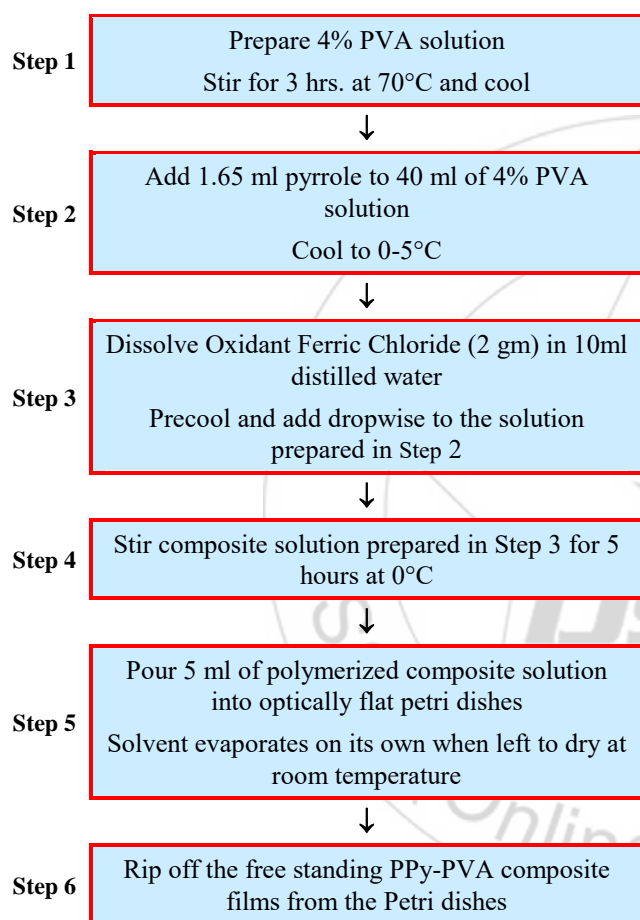
### 2.1 Materials

The monomer pyrrole was procured from Spectrochem Pvt. Ltd. Mumbai (India). Pyrrole was double distilled at 131°C and stored at 4°C prior to use for synthesis. Polyvinyl alcohol (PVA) was obtained from S. D. Fine Chem. Ltd., Mumbai.

The degree of hydrolysis of this powder is 86-89 % and M.W. 85000 – 124000. It was used as host polymer during the preparation of composites of polypyrrole. Ferric Chloride anhydrous (Iron (III) chloride) was also obtained from S. D. Fine Chem. Ltd., Mumbai. It was used as an oxidizing agent during polymerization. The purity of all chemicals was of analytical grade and used as-received, without further purification. All solutions were prepared using distilled water. Ammonia gas canister was purchased from Chemtron Science Lab. Pvt. Ltd. Mumbai

## 2.2 Synthesis of PPy-PVA composite films

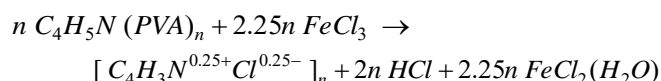
Fig.1 shows the synthesis scheme of PPy-PVA composite films.



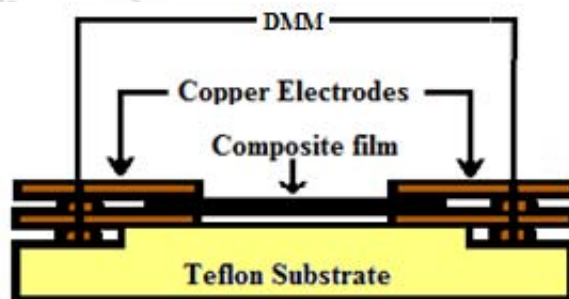
**Figure 1:** Synthesis scheme of PPy-PVA composite films

As discussed in our earlier publication [10], a 4% weight to volume polyvinyl alcohol solution was prepared by dissolving PVA powder in distilled water. This was then stirred vigorously at 70°C for 3 hours on a magnetic stirrer with hot plate so as to obtain clear PVA solution, which was then left to cool to room temperature. Next, to this PVA solution, double distilled pyrrole was added in different volume to weight proportions with respect to PVA and stirred vigorously for about 30 minutes. The solution was then cooled to 0-5°C. Polymerization of pyrrole was carried out using iron (III) chloride (FeCl<sub>3</sub>) as the oxidizing agent. The oxidizing agent (in terms of different proportions) was first dissolved in 10 ml of distilled water and then added dropwise to the PVA + pyrrole mixture. This composite

solution was gently stirred for 5 hours. A homogeneous black coloured solution was obtained. Films of this solution were prepared by pouring a certain small portion on to a flat glass petri-dish or polypropylene surface. The thickness of the film was controlled by the volume of the solution added. Every time 5 ml of the stock solution was poured on to the Petri dish so that the film thickness of approximately 35 to 40 micrometers could be produced. The solvent got evaporated on its own when left to dry at room temperature. Black coloured composite films were thus obtained. All the films were stored in a vacuum desiccator for further investigation. The polymerization of pyrrole occurs on the PVA-transition-metal salt film according to the reaction (proposed by Benseddik et al.) [11, 12]



## 2.3 Fabrication of NH<sub>3</sub> sensor



**Fig. 2:** Structure of fabricated gas sensor

The sensor device was in the form of two probes consisting of two small copper plates fixed onto a Teflon holder as shown in Fig. 2. The composite films were fixed between the two copper electrodes to which electrical contacts were made to connect to Programmable DMM (Scientific SM5051).

## 2.4 Instruments and analysis

The morphological examination of composite films was carried out using scanning electron microscope (SEM) (JEOL, JSM-5400) at accelerating voltage 10kV. Chemical structure was examined by FTIR spectroscopy (Perkin-Elmer Ltd. system 2000), in the 400–4000 cm<sup>-1</sup> range. The PPy-PVA composite films was characterized by XRD using Panalytical X'Pert (Philips). The XRD patterns were recorded between 2θ = 5° to 40°. The electrical conductivity was measured by two probe method, in the temperature range of 303K to 343K.

## 2.5 Gas sensing measurements

For measuring the gas response, specially prepared gas chamber (1000 cm<sup>3</sup>) was used. The fabricated sensor was fixed inside the gas chamber. The schematic diagram of a typical gas sensing unit is shown in Fig. 3. Measured quantity of NH<sub>3</sub> gas was injected through syringe so as to yield desired gas concentration in the chamber. The resistance was measured by using Scientific Programmable Digital

Multimeter (SM5015). Once steady state was achieved, recovery of the sensor was recorded by exposing the sensor to fresh air. This was done by opening both the valves  $V_1$  and  $V_2$  of the chamber simultaneously. At the same time, the vacuum pump was switched on. Valve  $V_1$  allowed fresh air to enter the chamber while valve  $V_2$  allowed the gas to exit the chamber. The vacuum pump accelerated the process of degassing. All the gas sensitivity measurements were carried out at room temperature [13].



Figure 3: Schematic sketch of gas sensor

### 3. Results and Discussion

#### 3.1 SEM analysis

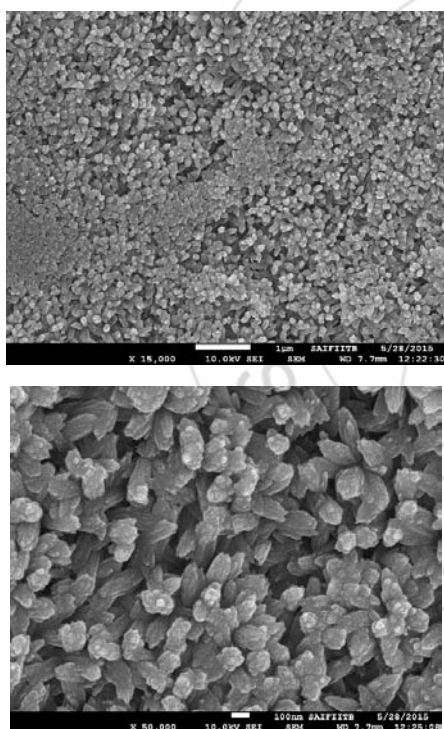


Figure 4: Electron micrographs of PPY-PVA composite films

Surface morphology is one of the important parameters for conducting polymers. Electron micrographs of the composite films are depicted in Fig.4. SEM study shows uniform, granular and porous surface morphology. This is suitable for gas sensing application which enhances both the adsorption as well as desorption of gas molecules [14, 15].

#### 3.2 FTIR analysis

Figure 5: FTIR spectra of control PVA-PPy composite film

Fig. 5 shows the FTIR spectra of PPY-PVA composite films. The bands appear in the region  $3300 - 3400 \text{ cm}^{-1}$  due O-H stretching of PVA and N-H stretching of pyrrole ring [16-21], absorption peaks at  $2944.1 \text{ cm}^{-1}$  due to CH stretching and  $\text{CH}_2$  stretching [19], the peak around  $1630$  are due C=C stretching of pyrrole [16]. The band at  $1632, 3 \text{ cm}^{-1}$  is due to C=C of pyrrole and deformation vibration of the absorbed water molecules [18, 20], the shoulder at  $1544.62 \text{ cm}^{-1}$  C=C stretching of pyrrole [16], the small band at  $1444.6 \text{ cm}^{-1}$  is due to in plane deformation O-H and C-H of PVA and stretching C-N of pyrrole, the peak around  $1371.2 \text{ cm}^{-1}$  correspondence to  $\text{CH}_2$  wagging of PVA[18], due to O-H in plane deformation with CH wag of PVA and C-C stretching of pyrrole peak is occurring at  $1312.2 \text{ cm}^{-1}$ [19], the band at  $1236.2 \text{ cm}^{-1}$  is due to C-O-H in plane deformation of pyrrole [21], the band at  $1178 \text{ cm}^{-1}$  is due to C-C stretching of pyrrole [16], the band at  $1094 \text{ cm}^{-1}$  is due to C-O stretching of PVA and C-H stretching of pyrrole [19]. A new strong band at  $1035.6 \text{ cm}^{-1}$  is observed in the composite film compared to the PVA and PPY which is evidence for the formation of the PPY-PVA composite film [20, 21]. The band at  $914.1 \text{ cm}^{-1}$  is due to  $\text{CH}_2$  rocking [20] and the strong characteristic band at  $852.6 \text{ cm}^{-1}$  is due to PVA skeletal band and C-H out-of-plane deformation of pyrrole [16, 17]. The band at  $779.8 \text{ cm}^{-1}$  is due to C-H out-of-plane deformation of pyrrole [16, 18] while the band at  $674.9 \text{ cm}^{-1}$  is due to O-H twisting of PVA. The C-H out-of-plane deformation of pyrrole and Fe-O stretching band [19, 20]. The assignment of these peaks and all these observations support that the synthesized product is a PPY-PVA composite.

#### 3.3 XRD analysis

Control PVA exhibits a typical peak at  $19.45^\circ$ , which indicates the semi-crystalline nature of PVA [22, 23]. Pure PPY displays a broad peak at  $25.35^\circ$  in its diffraction pattern. The broad peak is a characteristic of its amorphous nature [15, 24-25]. Fig. 6 shows the X-ray diffraction pattern of PPY-PVA composite films. A broad peak found at  $26.35^\circ$  due to PPY was slightly shifted to a higher angle [26]. Three low intensity peaks are observed at  $7.98^\circ$ ,  $12^\circ$ , and  $35.1^\circ$  which can be related to cross-linking between PVA and PPY. This gives the indication that the PPY crystals are present in the PPY-PVA composite film.



where  $R_{gas}$  and  $R_{air}$  are the electrical resistances of the sensor in  $NH_3$  gas and air respectively. Response time is the time taken for the sensor to attain 90% of the maximum change in resistance on exposure to the gas. Recovery time is the time taken by the sensor to get back 90% of the original resistance. The gas sensing studies were carried out for  $NH_3$  gas at room temperature [13].

### 3.5.1 Sensitivity of PPy-PVA composite films sensor to $NH_3$ gas

**Figure 6:** XRD pattern of PPy-PVA composite film

### 3.4 Measurement of Conductivity

**Figure 7:** I-V Characteristics of PPy-PVA composite films at different temperatures

In order to determine the current conduction mechanism of PPy-PVA composite films, the temperature dependence of electrical conductivity was studied in the temperature range of 303K to 343K. Plots of I vs. V for PPy-PVA composite films are as shown in Fig. 7. Nearly linear relationship of the I-V curve was found. The electrical conductivity of PPy-PVA composite films was found to be  $2.16 \times 10^{-3}$  S/cm at room temperature (303K). It was observed that if the temperature is increased gradually, the electrical conductivity also increases. It indicated the semiconducting behaviour of films. The mechanism of conduction was supported by polarons and bipolarons formation due to the dopant molecules [15]. The electrical conductivity behaviour can be explained using Arrhenius Model and Mott's Variable Range Hopping (VRH) Model [10]

### 3.5 Gas Sensing Performance

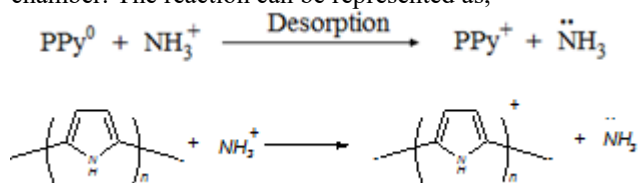
The response of the sensor (S) is given by:

$$S\% = \frac{R_{air} - R_{gas}}{R_{air}} \times 100\% = \frac{\Delta R}{R_{air}} \times 100\%$$

**Figure 8:** Response of PPy-PVA composite film sensor for various concentrations (ppm) of  $NH_3$  gas.

The response of the PPy-PVA composite film sensor for various concentration of the  $NH_3$  gas is shown in Fig. 8. The sensitivity of the PPy-PVA sensor to the concentration of the  $NH_3$  gas at 25 ppm, 50 ppm, 100 ppm, 200 ppm and 300 ppm was found to be 4.26 %, 10.44 %, 14.62 %, 17.73 % and 20.42 % respectively. This behaviour was related to the adsorption of the  $NH_3$  gas molecules on the surface of the PPy-PVA film. As the concentration of  $NH_3$  gas increased, the number of active sites for adsorption increased, leading to an increase in the sensor's response. The enhanced gas sensing performance of the PPy-PVA sensor was due to the porous microstructure of the sensing material which offers more chemical reactions to occur at the interface [27]. PPy-PVA sensor gives a good response from 25 ppm concentration of  $NH_3$  gas (4.26%). Also, the linearity of response in the range from 25 to 300 ppm of  $NH_3$  gas concentration suggests that the PPy sensor can be reliably used to monitor the concentration of  $NH_3$  gas over this range. Fig.9 presents the sensitivity of PPy-PVA films as a function of various concentrations (25-300 ppm) of  $NH_3$  gas. Fig. 9 reveals that the response of PPy-PVA film increases from 4.26 % to 20.42 % with increasing the concentration of  $NH_3$  from 25 to 300 ppm. It can be observed that the sensitivity increases with the increase in the concentration of ammonia (up to 300 ppm) and tend to saturate subsequently. The PPy-PVA film gives excellent sensitivity to ammonia gas, which may be due to the uniform and porous surface morphology of the films. The response increases almost linearly with the gas concentration, suggesting its suitability to be used as an ammonia sensor.

The original resistance value is achieved when the NH<sub>3</sub> gas has been turned off and fresh air is introduced into the test chamber. The reaction can be represented as,

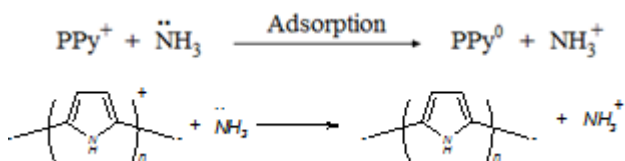


### 3.5.3 Reproducibility of PPy-PVA composite film sensor

**Figure 9:** Sensor response as a function of concentration (ppm) of NH<sub>3</sub> gas

### 3.5.2 Sensing mechanism of PPy-PVA composite film sensor

The change in the resistance of the sensing element (PPy-PVA composite film) as a function of time was recorded at room temperature and displayed in Fig.10. Polypyrrole is a 'p' type semiconductor i.e. the predominant current carriers are holes (positive charges). In ammonia molecule, the nitrogen atom has an unshared pair of electrons. Therefore on adsorption of this gas the holes i.e. the positive charge on the polypyrrole gets partially neutralized causing a decrease in carrier density of polypyrrole and intern decreasing the conductance. The reaction can be represented as follows [28],



**Figure 10:** Change in resistance of PPy- PVA composite film with respect to time on the exposure of NH<sub>3</sub> gas.

**Figure 11:** Repetitive response of PPy-PVA film sensor to 100 ppm of NH<sub>3</sub> gas

The reproducibility is the important parameter that is to be considered. The process of passing ammonia gas and subsequent pumping out was repeated to see the reproducibility and repeatability of the performance. The reproducibility of the PPy-PVA sensor was measured by repeating the response measurement test three times. Fig. 11 shows the gas response of PPy-PVA sensor upon periodic exposure to 100 ppm of NH<sub>3</sub> gas operating at room temperature. Interaction of ammonia with PPy-PVA increases the resistivity and removal of ammonia decreases the resistivity of the film steadily to a minimum value but a drift from its original value was observed. This results from the increasing diffusion of ammonia gas with every subsequent exposure. From Fig.11, it is concluded that PPy-PVA sensor has excellent reproducibility. This result indicates that the PPy-PVA sensor can be used as a reusable sensing material for the detection of NH<sub>3</sub> gas operating at room temperature

### 3.5.4 Response and recovery time of PPy-PVA sensor

An important parameter used for characterizing a sensor is the response and recovery time. The response and recovery time depend on the rate of adsorption and desorption respectively. The response and recovery times were obtained from the electrical response curve (Fig. 8) of PPy-PVA sensor to various concentrations of NH<sub>3</sub>. Response and recovery curve of PPy-PVA sensor for different concentration of NH<sub>3</sub> is as shown in Fig. 12.

at room temperature with a detection limit of even a ppm level. This work demonstrates that PPy-PVA composite films can be used for fabrication of high performance NH<sub>3</sub> sensors operating at room temperature.

## 5. Acknowledgments

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**Figure 12:** Response and recovery times of PPy-PVA composite film sensor for various concentrations of NH<sub>3</sub> gas

It was found that response time increased from 120s to 266s as the concentration of NH<sub>3</sub> increased from 25 ppm to 300 ppm. Such an increase in response time when concentration of NH<sub>3</sub> increases may be due to large availability of vacant sites on films for gas adsorption as evidenced from microstructural study. While recovery time increase from 64s to 572s as the concentration of NH<sub>3</sub> increased from 25 ppm to 300 ppm. This may be due to gas reaction species which left behind after the gas interaction results in decrease in desorption rate and hence increases the recovery times with increase in the concentration of NH<sub>3</sub>.

## 4. Conclusion

The successful synthesis of flexible and free-standing PPy-PVA composite films was achieved. The composite films showed good film forming ability on a flat glass petri-dish or polypropylene surface and have the best storage ability. SEM study shows uniform, granular and porous surface morphology which is suitable for gas sensing application. The structure of PPy-PVA composite films was confirmed by using FTIR and XRD techniques. The DC electrical conductivity ( $\sigma$ ) of the composite polymer films has been investigated by two probe method and was found to be  $2.16 \times 10^{-3}$  S/cm at room temperature

The flexible and free-standing PPy-PVA composite films have been investigated for room temperature operating ammonia gas sensor. The response of the films increased from 4.26 % to 20.42 % with an increase in the concentration of NH<sub>3</sub> from 25 to 300 ppm. The prepared sensor could detect concentration as low as 25 ppm of NH<sub>3</sub> with reasonable response of 4.26 %. The sensor based on PPy-PVA composite films enhanced both the adsorption as well as the desorption process. It was found that the response time increased from 120s to 266s as the concentration of NH<sub>3</sub> increased from 25 ppm to 300 ppm while the recovery time increased from 64s to 572s. The response increases almost linearly with the increasing gas concentration. The gas sensing tests performed show that the composite films exhibited a fast response, rapid recovery and high sensitivity

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