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Development and Characterization of Plasma Polymerized Polyaniline Nanocomposite Thin Films for H2S Gas Sensing Applications

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ABSTRACT: Several hazardous gases are prevailing in the environment. These are big challenge for environmental scientists regarding their detection and monitoring. Among these H2S gas is a big concern. Methods being used for its detection are indirect and time consuming and are effective at high temperatures only, hence limiting their applicability. For detection of such gases, nanocomposite polymers doped with Fe-Al and prepared by plasma polymerization are very suitable. With different substrates, these have wide application. Using this technique, the preparation and characterization of polymer thin films is discussed in this paper. Using conducting polymer based sensors, a new avenue for sensing H2S gas and dangerous chemicals prevailing in the environment, is the subject of investigation.

1. INTRODUCTION

Environmental gas monitoring and development of sensitive and suitable materials for their detection is well understood. Human activity directly or indirectly accounts for the majority of hazardous gases in the atmosphere. H_2S gas is one of those which is widely generated due to bacterial breakdown of organic matter and biomass decompositions, in manufacture of semiconductors, in medical wastes and in chemical industries [1,2], whose excessive concentrations cause health risk to people. There is a great need of detection of H_2S gas with selective, sensitive and precise sensing methods. Conducting polymers are very suitable materials and among these, polyaniline (PANI) is the most effective for sensing these gases. Semiconducting characteristics of PANI play an important role in chemical sensors as being sensitive for gas absorption and de-absorption [3]. Among various chemical sensors developed for detection of H_2S gas, electrochemical, optical and chemiresistive sensors have drawn greater attention and are classified as shown in fig.1. In chemiresistive type, the conductive polymer and metal oxide composition has greater advantage over other ones.

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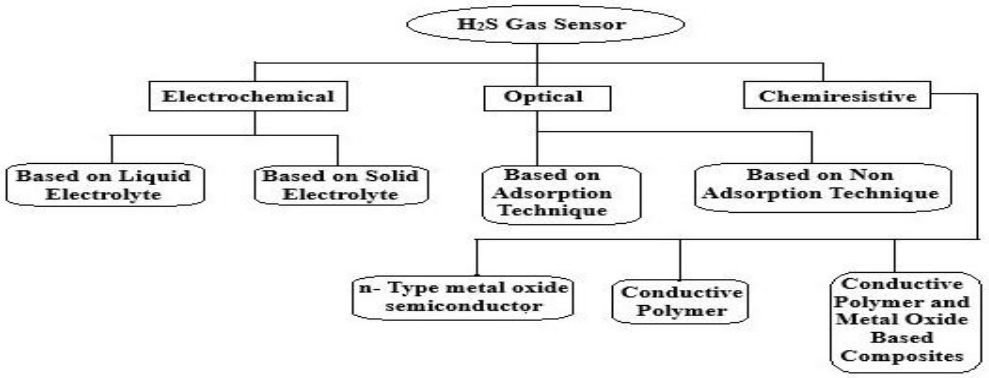

Fig.1. Types of H_2S gas sensors

Plasma polymerized nanocrystalline PANI thin films [4, 5, 6, 7, 8, 9] are very suitable for chemiresistive type conducting polymer sensors and are very sensitive to the presence of H_2S gas and can work in a wide range of temperatures. H_2S gas being hazardous, has harmful impact with exposure of 5ppm (parts per million). As it exists in gaseous as well as aqueous forms, its detection upto sub ppm levels is very much needed. It is harmful for heart, lungs and respiratory system. Plants are also badly affected by it.

With the bad impacts of H_2S gas, its detection with sensitive sensors is very essential. The techniques for detection of H_2S gas currently being used [10] are costly, time consuming and complex, as these are based on sample drawing and its analysis. Available solid state sensors work at high temperatures [11, 12, 13, 14]. Hence easy, quick and cost effective methods are needed for detection of H_2S gas. Electroactive semiconducting polymer thin films have recently been created for detection of H_2S gas [15]. Polymer based semiconducting devices are compatible, easy in processing and packaging and are cost saving. The conductive polymers with conjugate backbone with alternative single and double bonds consist of alternative $\sigma-$ bond and $\pi-$ bond . $\sigma-$ bond is responsible for mechanical strength of polymer while $\pi-$ bond is weaker and causes the movement of charges throughout the polymer chain and makes the polymer semiconducting or conducting. The bond structure is presented in diagram 2,

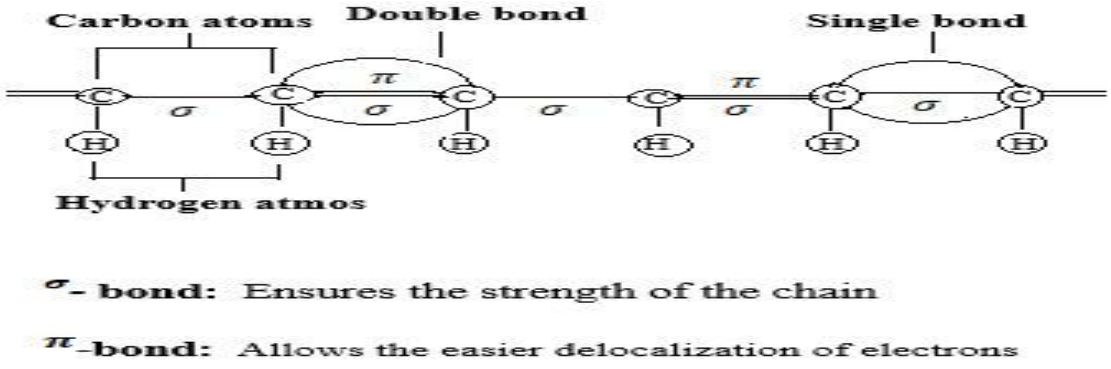

Fig.2. A schematic of conjugated backbone of conducting polymer

Polymer thin films fabricated with flexible substrates are very suitable over traditional sensors. Semiconducting polymers with altering doping level show altered conductivity and can vary from insulator to conductor [16, 17, 18, 19, 20, 21, 22]. H_2S gas has weak acidic nature, therefore pure PANI does not show excellent response with it. The response of PANI is fascinating for strong acids having high doping ability. Therefore composite of PANI are used to have fruitful response towards H_2S gas [23]. With addition of Iron and Aluminium oxides with aniline formaldehyde, quick response is exhibited by nano copolymer thin films with better sensitivity and selectivity to the exposure of H_2S gas. These features lead to make these as sensors. The sensitivity is an important property of sensor which is the ratio of its conductivities after and before gas exposure. With

suitable change in stoichiometric composition of the dopant, amazing sensitivity, selectivity and quick reaction time are obtained. These excellent features put forward the fabrication of plasma polymerized thin films and their application as suitable sensors for sensing H_2S gas.

2. EXPERIMENTAL

Instrumentation and materials:

An assembled plasma polymerization unit purcharged through Usha Chemicals Kanpur was used for fabrication of PANI thin film at 1200 volt DC power supply.

For proper and uniform deposition of thin film, a substrate is needed which should fulfill the desired qualities like electrical conductivity and thermal stability, compatible with the process of film deposition. Glass, metal oxide and silicon are commonly used materials which fulfill the above requirements. Nanocrystalline materials commonly used for detection of H_2S gas are polymers, metal oxides and semiconductors. Nanocrystalline material senses the existence of H_2S gas and indicates by signals.

Any of the gases like Styrene, Nitrogen and Vinyl acetate in the reactor produces plasma which deposits selected nanocrystalline material over the substrate. As per the quality of sensor and type of nanocrystalline material the gas is chosen in the process. As for metal oxide nanocrystalline material, nitrogen is normally utilized and we have also used it for polymer nanocrystalline material.

For more accurate and sensitive detection of H_2S gas, electrodes are added to thin films to enhance their electrical conductivity. Protective coatings are done to prevent the film form environmental influences. The materials suitable for synthesis of nanocrystalline thin films for detection of H_2S gas are mentioned below.

(2.1) Glass, Silicon, and metal oxides (e.g. Alumina, titania) are taken as substrates for film deposition. Glass is used as substrate in this process.

(2.2) Crystalline materials are commonly polymers (e.g. Polyaniline, Polypyrrole) and metal oxides (SO_2, ZnO, WO_3 etc). PANI is used in our process.

(2.3) Plasma polymerization gas: Nitrogen, Styrene and Vinyl acetate are used for plasma generation. We have used nitrogen in our process.

Analytical grade $AlCl_3$ and $FeCl_3$ are used with aniline distilled twice.

All chemicals and materials were purcharged through Usha Chemicals, Kanpur.

3. PREPARATION OF PANI THIN FILM

Many workers (18, 24) obtained nanocomposites of aniline formaldehyde with Fe-Al doping. $AlCl_3$ acts as a lewis acid dopant and enhances electrical conductivity by increasing charge carrier density. It improves chemical and thermal stability of polymer. It provides uniform and controlled doping for thin films and reduces moisture sensitivity of polymers. $FeCl_3$ is strong oxidizing dopant and improves redox activity of polymer and increases conductivity of conducting polymer. Therefore, doping PANI with $AlCl_3$ and $FeCl_3$ with required proportions makes its sensing performance fast responsive, reversible and long lasting. Plasma polymerization with inherent advantages is a versatile technique for deposition of PANI thin films. Electronic impacts ionize gaseous molecules which form radical cations leading to formation of the polymer. A transparent solution of 0.1M aniline, 1M HCl, 0.7g anhydrous $AlCl_3$, 9.3g anhydrous $FeCl_3$ is mixed with 100ml distilled water [25, 26]. Mixture is stirred with formaldehyde solution for proper mixing.

The plasma is created in plasma zone in vapour state. The solution injected above anode in reaction chamber reaches to the plasma zone. The material is deposited over the substrate in form of thin film [27, 28, 25, 29, 20, 21].

Thickness of the film is measured with quartz thickness meter in our process. We doped Fe-Al in the ratio 93:07 by weight. As copolymer decomposes and dissociates above 280°C, we kept deposition temperature below 270°C to preserve polymer structure. First the film was deposited for undoped polymer and then Fe-Al doped copolymer. Molecular weight of plasma was found to be 16.320 and it is because of heavier dopants of Fe-Al [29] and should be low.

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4. CHARACTERIZATION AND RESULTS

In continuation to our earlier work of formation of PANI thin films for its application in detection of SO_2 gas [30], we have explored the methods and modifications to apply the PANI thin films prepared by plasma polymerization for detection of H_2S gas, by changing doping levels to make them suitable for sensing H_2S gas. Being lewis acid, increased quantity of $AlCl_3$ enhances interaction of PANI with H_2S gas. PANI alone has weak interacting with H_2S gas, being weak acid. This (93:07) combination of Fe and Al makes thin films prominent for sensing H_2S gas.

I-V characteristics of the prepared film was studied with metal/polymer/metal sandwich with the exposure of H_2S gas. These film characteristics could be used for detection of H_2S gas in different environments .Study of Structural, and electrical characteristics have been presented in this paper. We studied the structure in XRD facility in UPTTI, Kanpur [30].

Figure 1(a) shows x-ray diffractogram of Fe-Al doped film and 1(b) that of undoped film. Peaks in the diagram 1(a) depicts high degree of crystallinity, which is expected due to Fe-Al doping, otherwise the film shows amorphous character. Fig. 1(b) shows comparatively flat curve and is for entirely amorphous PANI composite film.

Table 1 (a):

S. No.	2θ (Degree)	Lin (Counts)	Peak Observation
1.	10	5	Small Peak
2.	20	11	Peak
3.	33	8	Peak
4.	39	16	Highest Peak
5.	45	8	Peak
6.	54	3	Very Small Peak
7.	56	6	Small Peak
8.	59	7	Small Peak
9.	66	11	Peak
10.	77	6	Small Peak
11.	84	3	Very Small Peak
12.	90	2	Very Small Peak

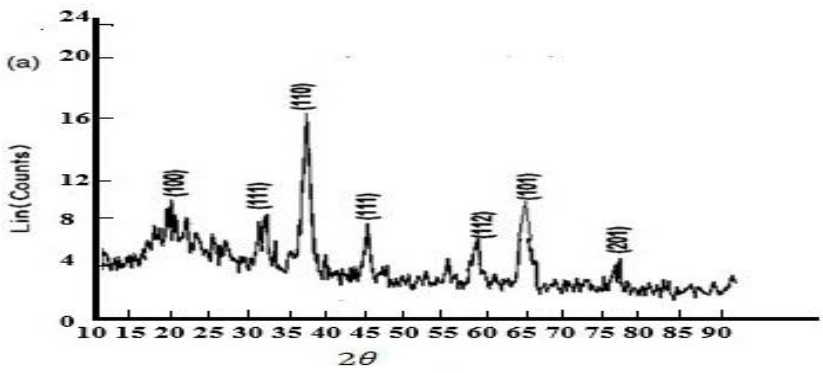

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Table 1 (b):

S. No.	2θ (Degree)	Lin (Counts)	Peak Observation
1.	10	4.6	Small Peak
2.	15	3.7	Small Peak
3.	20	1.8	Weak Peak
4.	24	3.0	Broad Peak
5.	27	3.8	Medium Peak
6.	30	4.0	Peak
7.	35	4.3	Peak
8.	40	3.9	Broad Peak
9.	44	2.9	Weak Peak
10.	50	2.0	Small Peak
11.	58	0.9	Very Weak Peak
12.	65	0.7	Very Weak Peak
13.	74	0.6	Very Weak Peak

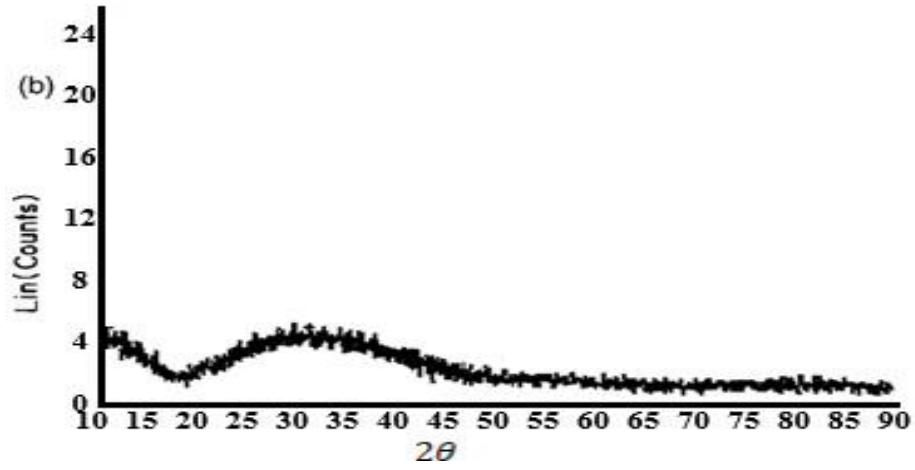


Fig.1. (a) and (b) intensity vs diffraction angle curves for X-ray diffraction of polymeric thin films

5. THIN FILMS IN SENSOR APPLICATIONS

Purpose of nanocrystalline thin films is to use these in the development of hazardous gas sensor. Glass substrate is properly cleaned with methanol and distilled water to deposit film of thickness around 1000A°. To enhance conductivity and sensitivity, the film deposited over the glass substrate is installed with electrodes covering entire glass surface.

6. DC CONDUCTIVITY, RESPONSE AND SENSITIVITY OF SENSOR

The I-V characteristics for nanocomposite thin film sensor were studied by four probe apparatus in our lab purchased from Omega Electronics Jaipur. The schematic diagram and drawing of four probe apparatus are shown in fig.2 (a) and (b). Current is passed through the outer probes and the voltage drop across the inner probes is measured for the PANI thin film

below the probes. The thin film sheet resistance (R_s) is calculated using the relation (31) given below and is expressed in ohm/Square.

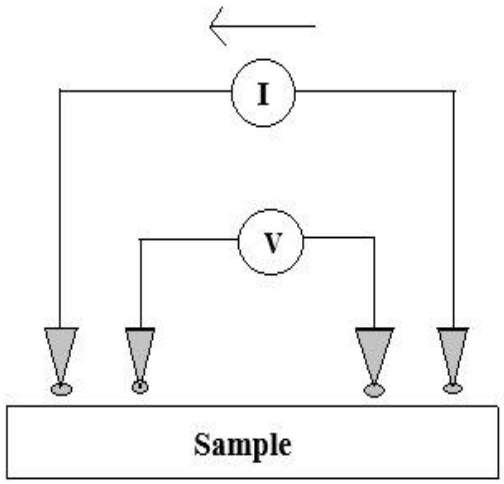
$$R_S = \frac{\pi(\Delta V)}{\ln(2)(I)} = 4.53236\frac{\Delta V}{I}$$

Where, $\pi/\ln(2)$ in above equation is a geometric correction factor and is dimensionless, ΔV is voltage drop across the inner probes and I is the current passing through the sample.

The resistivity of the material of film is calculated by

$$\rho = R_s \times t_f$$

Where, t_f is the thickness of thin film. Conductivity is the reciprocal of the resistivity.



2(a) **2(b)**

Fig.2. Four Probe Set up 2(a) and its drawing 2(b)

Conductivity changes occur when the H_2S gas is exposed to the sensor, which is the measure of the sensitivity and the response to detect the gas. Sensitivity of the sensor is the ratio of rates of conductivity after the exposure of H_2S gas (S_e) and before (S_o) and is known as sensitivity factor given as

$$S = S_e / S_o$$

I-V characteristics are drawn for different concentrations of H_2S gas in air ppm shown in fig. 3. The conductivity for variation of DC voltage from 0.5V to 6V for different series from 1 to 6 is studied. Graph shows highest selectivity for series 4 at 4V, which is because of lowering of intercrystalline grain barriers and significant current flows at this stage. This voltage is actual voltage for sensor. The quality of sensors is measured by sensitivity factor, which is as high as 800. With changing H_2S gas concentrations the reaction time of sensor changes.

Table 3: Conductivity of thin film at different concentrations of H_2S gas on certain voltages**:**

S.No.		**Series 1**	**Series 2**	**Series 3**	**Series 4**	**Series 5**	**Series 6**
	Conc of H_2S gas (ppm)	**0.5 V**	**1.0 V**	**1.5 V**	**2.5 V**	**4 V**	**6 V**
1.	0.01	5	11	20	30	100	100
2.	10.01	9	29	78	90	105	100
3.	20.01	13	30	90	105	110	100

4.	30.01	14	40	99	125	115	100
5.	40.01	15	40	100	135	140	100
6.	50.01	15	40	100	140	170	100
7.	70.01	15	40	100	155	200	100
8.	90.01	15	40	100	160	250	100
9.	100.01	15	40	100	170	310	100

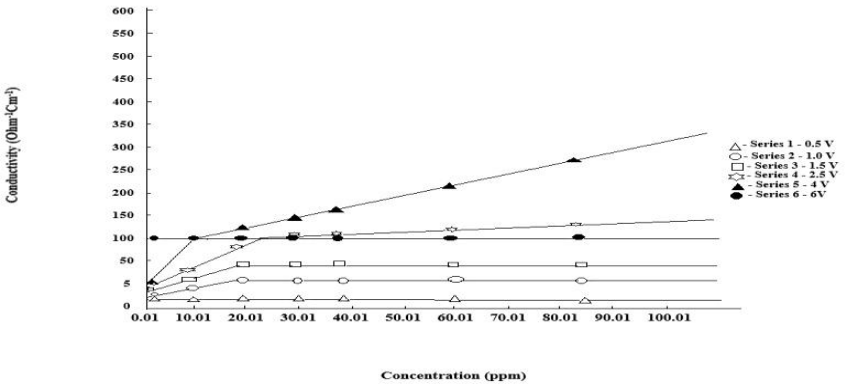


Fig.3. I-V characteristics of the sensor

For 0.2 to 20 ppm of H_2S gas the reaction time is shown in fig. 4. For air H_2S gas combination an initial surge is observed for 5 seconds before reaching saturation. With the interruption in gas flow, the sensor current drops exponentially in 8 to 10 seconds and with the increase in air H_2S gas flow current and reaction time increase exponentially while with the steady flow the reaction time does not rise. Fig. 5 (a) and 5 (b) show response and recovery time of sensor. Response time varies from 4 to 14 sec for increasing concentrations of H_2S gas while recovery time varies nearly from 3 to 12 sec for the same. Working of sensor is based on physical absorption of gas on polymeric film and when the gas flow is stopped, the sensor restores again. Hence the sensor can be reused many times over the years. Polymers are non biodegradable and have low environmental impact, hence these are stable. Plasma polymerized PANI copolymer behaves as p-type semiconductor. Polarons and bipolarons are mobilized by the action of electric field across crystallite boundaries, which acts as barrier [28, 29]. H_2S gas exposure decreases barrier height and increases charge flow across grain borders, increasing current output. Barrier height and sensor response is directly related to amount of gas absorbed.

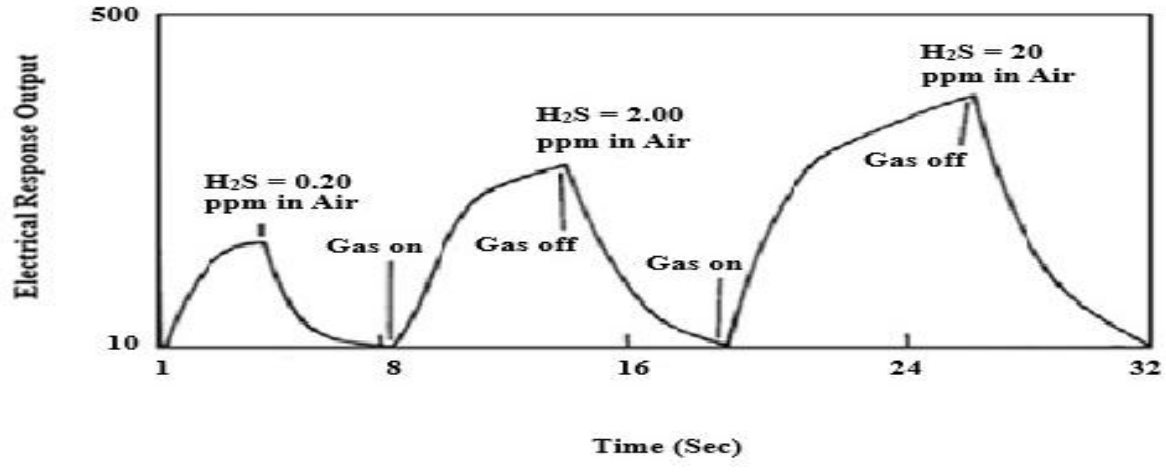
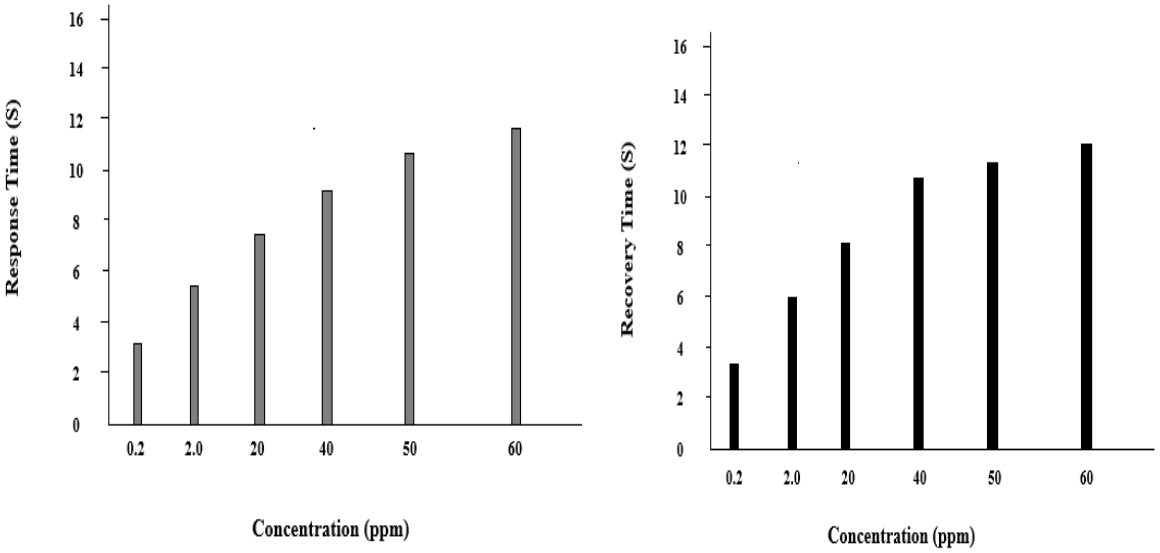


Fig.4. Repeating response of sensor up to 20ppm

Table 5: Response and recovery time of the sensor with different concentrations of H_2S gas

S. No.	Conc of H_2S gas (ppm)	Response time (S)	Recovery time (S)
1.	0.2	3	3.7
2.	1.7	4.4	5.6
3.	2.0	5.2	6.0
4.	14	6.4	7.0
5.	20	7.5	8.0
6.	35	8.8	9.0
7.	40	9.4	10.6
8.	46	10.2	11.0
9.	50	11.2	11.4
10.	54	11.6	11.8
11.	60	11.9	12.0



5(a) **5(b)**

Fig. 5. (a) response time and (b) recovery time of sensor for various concentrations of H_2S gas

Earlier workers as Meenakshi Kumawat et.al. [23] used chemical oxidation polymerization method for depositing PANI/WO_3/$CuCl_2$ composite film for sensing H_2S gas and could achieved a response time nearly 100 sec. Raju P Tandel et.al. [32] used polypyrole-Fe$(ClO_4)_2$-Ag Nanocomposite using, In situ chemical polymerization for detection of H_2S gas and could achieve a response time nearly 80 sec. We in our work could achieve a response time of nearly 10 sec with Fe-Al doped PANI nanocomposites with plasma polymerization technique and could have better achievement for H_2S gas sensing techniques.

CONCLUSION

In our methodology, the synthesis of PANI thin films results to the high quality, sensitivity and better response, useful for the detection of harmful gases as H_2S gas in sensors. The proper doping of PANI in the process of synthesis is major achievement regarding high sensitivity and selectivity. Fe-Al doping with appropriate composition in ratio (93:07) is used to enhance conductivity and is useful to fabricate sensors with reaction time around 10 sec for detection of H_2S gas. The inorganic sensors show response time from 1 to 2 minutes. The reuse and adaptation at room temperature increase sensor's life span. The continuous monitoring and detection of H_2S gas suits with the behaviour acceptability of plasma polymerized PANI thin films. Such sensors are useful for the detection of safety levels and limits of H_2S gas.

With varied composition of doping, these sensors are useful for the detection of other gases. These properties and their wide application and responses might be very useful for research workers, for scholars and industry.

Acknowledgments

The synthesis of PANI nanocomposite film by plasma polymerization technique has been carried out with the plasma unit in our lab, which has been sanctioned in project by Uttar Pradesh Higher Education Council. Vivek Dwivedi, Rajendra Kumar and Preeti Dwivedi have been involved in literature survey, literature collection and collection of sample and accessories in different centers as IIT Kanpur, HBTU, CSJMU etc. Kajal Kushwaha, Sunil Singh and Navnit misra have worked with the apparatus in experimental work for deposition of thin films. Measurements have been carried out in HBTU and UPTTI Kanpur labs and everyone has contributed as needed. We extend compliments to department of physics HBTU, Kanpur and UPTTI, Kanpur for their support . We also extend our gratitude to prinicipal, Brahmanand College, Kanpur for his valuable support.

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