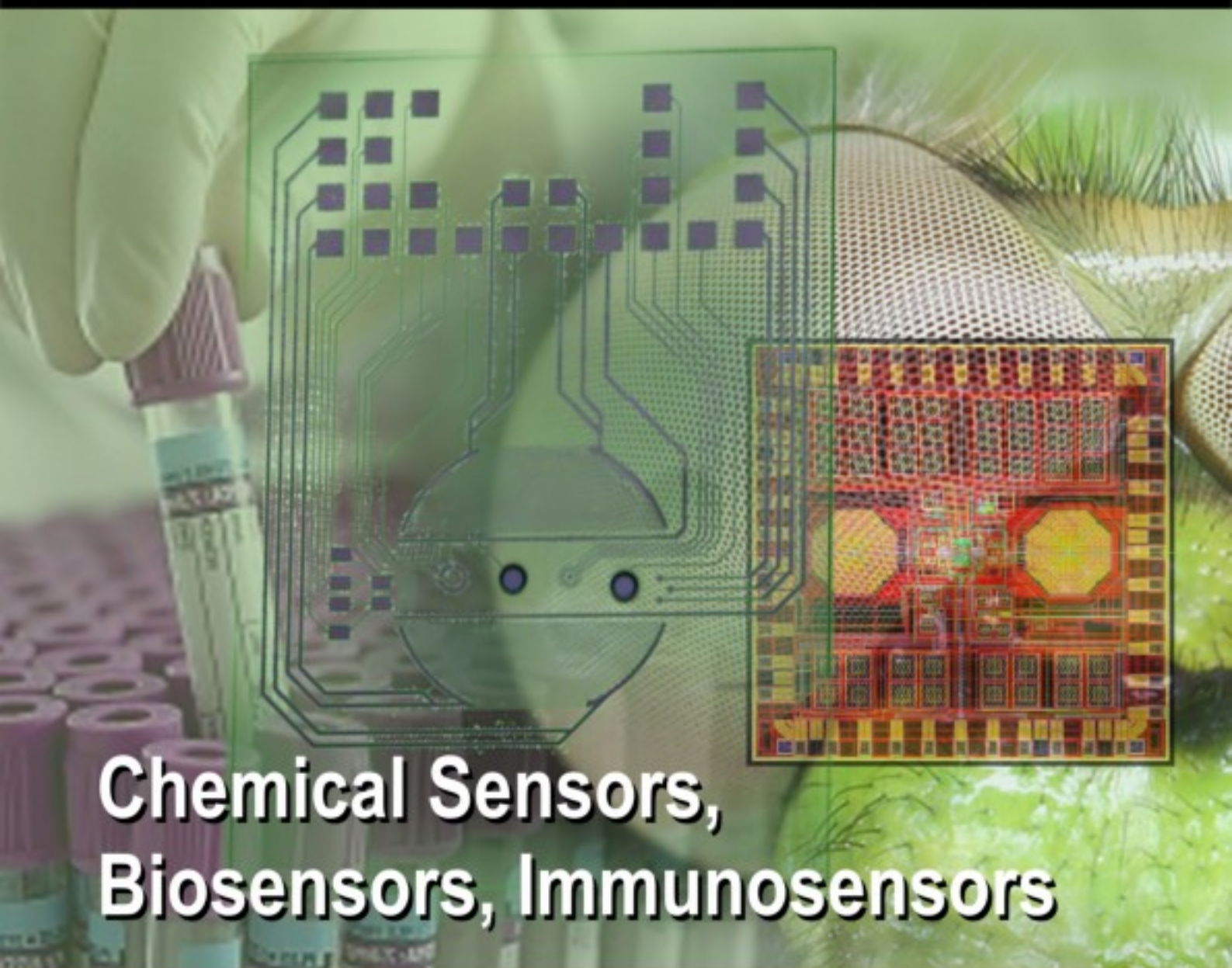


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Synthesis of WO₃-Polyaniline Composites and Their Gas Sensing Properties

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Abstract: Polyaniline (PANI) and WO₃-PANI composites were synthesized by a chemical polymerization method using ammonium persulphate (APS) as an oxidizing agent. This is a single step polymerization process to synthesize the conducting polymer. Thick films of PANI and WO₃-PANI were fabricated by screen-printing followed by firing at 300 °C for 30 min. WO₃-PANI (50 wt%) thick films resulted in LPG gas detector. Upon exposure to 100 ppm LPG gas, the barrier height between the grains of WO₃ and PANI decreases mark-ably leading to a drastic decrease in resistance. An exceptional sensitivity was found to LPG gas at 325 °C and no cross sensitivity was observed to other hazardous and polluting gases even at higher concentration (1000 ppm). The instant response (~ 14 s) and fast recovery (~ 38 s) are the main features of this sensor. XRD, SEM, E-DAX, FTIR and UV-Vis spectroscopy, characterized the synthesized polymers. The effects of microstructure and dopant concentrations on the gas response, selectivity, response time and recovery time of the sensor in the presence of LPG gas were studied and discussed. *Copyright © 2010 IFSA.*

Keywords: Polyaniline, Tungsten oxide, WO₃-PANI composites, LPG sensor, Chemical polymerization

1. Introduction

Conducting polymers have been attracted significant attention because of their potential applications in the fabrication of solid-state devices [1–4]. Various conducting polymers such as Polyaniline, Polypyrrole, and Polythiophene etc. have been widely investigated as effective materials for chemical sensors [5-9]. Among these polymers, polyaniline (PANI) is frequently used because of it's ease of

synthesis, flexibility, high electrical conductivity and good environmental stability [10-12]. As a result, it has been studied extensively and has emerged as the most promising candidate for commercial applications. However, a major problem with conducting polymer is its poor mechanical strength and processibility and poor chemical stability. There is wide scope for the enhancement by combining organic materials with inorganic counterparts to form composites [13-15]. PANI has a wide range of applications in the field of gas sensing [16-24]. However, very few reports of PANI and compositions of PANI are available on liquefied petroleum gas (LPG) sensors. Energy is the driving force [25] of all activities in the universe. Nothing moves, changes, grows or decays without consumption of energy. One of the simplest ways to withdraw the energy is from the burning of (LPG) like fuels. LPG is highly inflammable gas. It is explosively utilized in industrial and domestic fields as fuel. It is referred as town or cooking gas. Cooking gas consists chiefly of butane (55-vol %) [26], a colorless and odorless gas. It is usually mixed with compounds of sulfur (methyl mercaptan and ethyl mercaptan) having foul smell, so that its leakage can be noticed easily. This gas is potentially hazardous because explosion accidents might be caused when it leak out by mistake. It has been reported that, at the concentration up to noticeable leakage, it is very much more than the lower explosive limit LEL of the gas in air. Numbers of researchers are working for LPG detection, but they could not reached up to the depth demand by society. So there is a great demand and emerged challenges for monitoring LPG for the purpose of control and safety applications in domestic and industrial fields. The aim of the present work is to develop the sensor by modifying PANI and its compositions, which could be able to detect the LPG gas at trace level. Among the various metal oxide additives tested, WO_3 in PANI is outstanding in promoting the sensing properties to LPG in air.

2. Experimental

2.1. Synthesis of Material Powders

PANI/ WO_3 composite was synthesized by chemical polymerization route, which involves the freshly double distilled aniline as monomer with ammonium per sulphate (APS) as oxidizing agent and aqueous solution of hydrochloric acid as protonic acid medium. Monomer solution was prepared by adding 0.1 M aniline in 1M HCl followed by constant stirring. For various samples of PANI/ WO_3 composite with various concentration of WO_3 (0.025M, 0.05M, 0.075M) was added to this solution under constant stirring. The solution was allowed to cool in the ice bath till the temperature reaches in the range 0 to 5°C and this condition was maintained for two hours. 0.1 M APS solution in the same acidic medium was added drop wise. By maintaining the solution at such optimized condition for 24 h, polymerization was achieved. The solution was filtered and washed with distilled water repeatedly to remove the impurities and finally washed with 1 M HCl solution to obtain green precipitate, which was allowed to dry under dynamic vacuum (60°C) for 10 h. Pure PANI sample was synthesized by following same procedure in absence of WO_3 .

2.2. Materials Characterizations

The structural characterization of synthesized samples was analyzed with X-ray diffractogram (RIGAKU DMAX 2500) using Cu $K\alpha$ radiation with a wavelength 1.5418 Å in a 2θ range from 20 to 80°. The surface morphological study of PANI and WO_3 -PANI composites was carried out using scanning electron microscopy (JOEL JED 2300) coupled with an energy dispersive spectrometer (6360 LA). UV-Visible spectra were obtained by using U-2000 (Hitachi, Jpn) model. FT-IR spectra were obtained by using Perkin Elmer Spectrophotometer-1600 II (U. S. A).

The semi-conducting natures of PANI and WO₃-PANI compositions were observed from the measurements of conductivity with reciprocal of temperature. LPG Gas response of PANI and WO₃-PANI samples was determined by measuring electrical current in air and in presence of gas.

2.3. Thick Film Fabrication

Synthesized dark green powders of PANI and WO₃-PANI composites were ball milled to ensure sufficiently fine particle size. The fine powders were calcined at 300 °C for 1 h in air and reground. Thick films of, so obtained powders, were prepared by adopting the procedure explained elsewhere [25, 26].

3. Results

3.1 Structural Studies

The structure of composite sample was analyzed with X-ray diffractogram (RIGAKU DMAX 2500) using CuK radiation with a wavelength 1.5418 Å. Fig. 1 shows the X-ray diffractogram of the WO₃-PANI composite thick films doped with 50 wt % WO₃. The observed peaks are matching well with ASTM reported data of WO₃. The sharp peaks of the XRD patterns corresponds to WO₃ material and are observed to be triclinic and polycrystalline in nature.

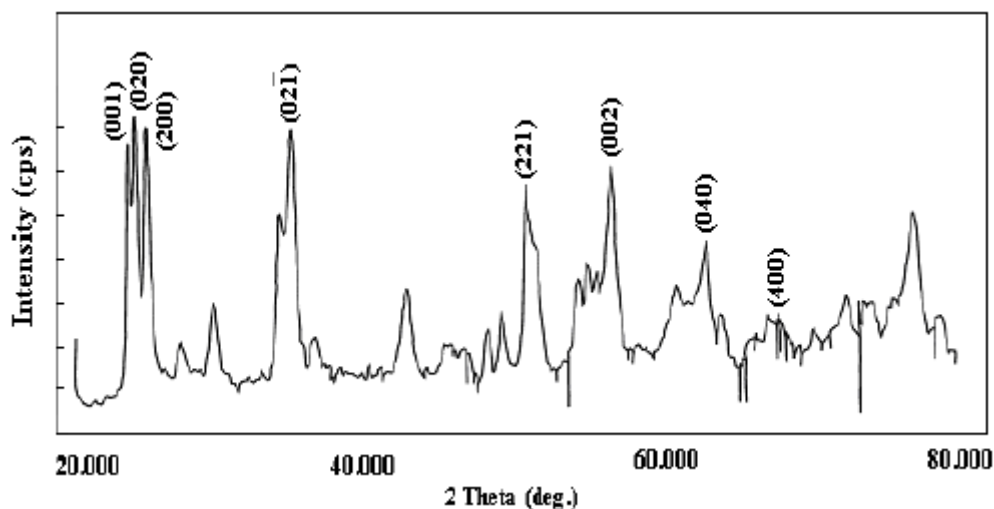


Fig. 1. XRD spectra of WO₃-PANI composites (50 wt%).

3.2. Microstructure-SEM

The microstructure and chemical composition of the films were analyzed using a scanning electron microscope (JOEL JED 2300) coupled with an energy dispersive spectrometer (6360 LA). Figs. 2(a-c) depict the microstructures of unmodified PANI and. Fig. 2(a) consists of randomly distributed chains of PANI. Fig. 2 (b) consists of WO₃ grains with smaller size and shape associated with the chains of PANI. Incorporation of inorganic material particles (WO₃) in PANI would cause to form compositions of WO₃ and PANI. Such compositions would construct heterojunctions. It is clear from the figure that the chains of compositions of WO₃ and PANI show the optimum porosity, which would help to reach the gas in the interstitial sites on exposure. The grains of WO₃ may reside in the intergranular regions

of PANI. Thus the effective surface area was expected to increase crucially. Fig. 2(c) consists of large number of WO_3 particles agglomeration. The effective surface area of the film may reduce due to high agglomeration of WO_3 with PANI, which resists reaching the gas interstitial sites of the film.

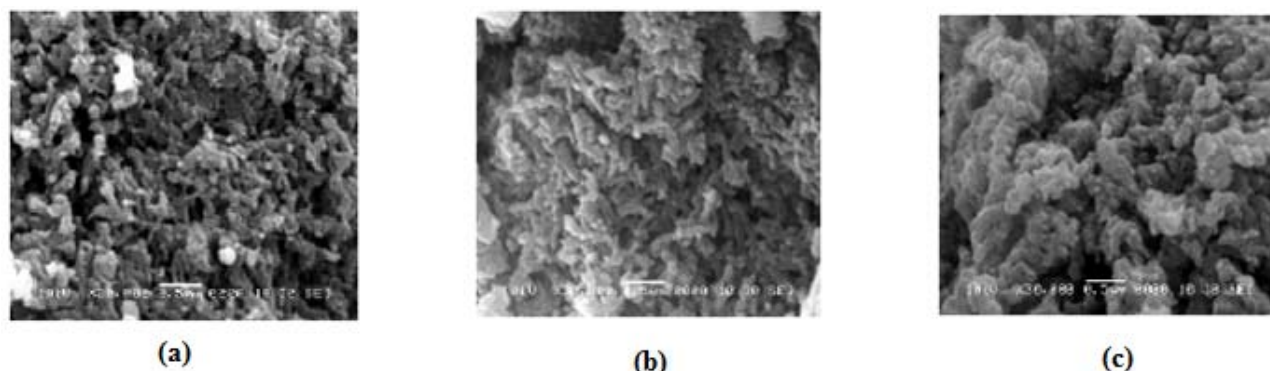


Fig. 2. SEM of (a) Pure PANI, (b) WO_3 -PANI (50-wt%) and (c) WO_3 -PANI (75 wt%).

3.3. Thickness and Thermoelectric Power Measurements

Thickness measurements were carried out using a Taylor-Hobson (Talystep, UK) system. The thickness of the films was observed to be in the range from 20 to 25 μm . The reproducibility of the film thickness was achieved by maintaining the proper rheology and thixotropy of the paste. The p- or n-type semi-conductivity of thick films of WO_3 and PANI were confirmed by measuring thermo electromotive force of thick film samples. The WO_3 was observed to be n-type and PANI, the p-type material.

3.4. UV-Visible Spectroscopic Study

Fig. 3 shows UV-Visible spectra of PANI and WO_3 -PANI powder sample dissolve in Dimethylsulphoxide. A peak at 350 nm (Fig. 3 a) can be assigned to π - π^* transition absorption band due to the excitation of benzene ring, peak at 450 nm can be assigned to polaron absorption band attributed to the formation of radical cation and a broad peak in visible region (~ 850 nm) can be assigned to dication absorption band, which is attributed to bipolaron formation on quionide segment in the polyemeraldine chain. It is observed that on addition of tungsten oxide, a broad peak at around 850 nm is removed totally (Figs. 3 b and c), and shifted at 650 nm. This blue shift is attributed to charge transfer from the benzoid of polyaniline to the quinoid units [27]. The charge compensation of the positive charge on WO_3 takes place at hetero-junctions. During polymerization of aniline in acidic medium with the help of $\text{NH}_4(\text{SO}_3)_8$ as oxidizing agent leads to the formation of $[\text{ANI}]^+$ radical cation which interact with another $[\text{ANI}]^+$ and then propagate as usual. Surface of WO_3 must be positively charged in acidic polymerization bath. Therefore adsorption of large amount of anions (Cl^-) seems likely to occur and compensate the positive charges on WO_3 surface. Beside this charge compensation, specific adsorption of anion (Cl^-) on the oxide surface may take place. These specifically adsorbed anions would work as the charge compensator for positively charged Nitrogen (N^+) atom in polyaniline macromolecule. Such charge compensation at n- WO_3 /p-polyaniline hetero-junction leads to nonconducting emeraldine base form of polyaniline.

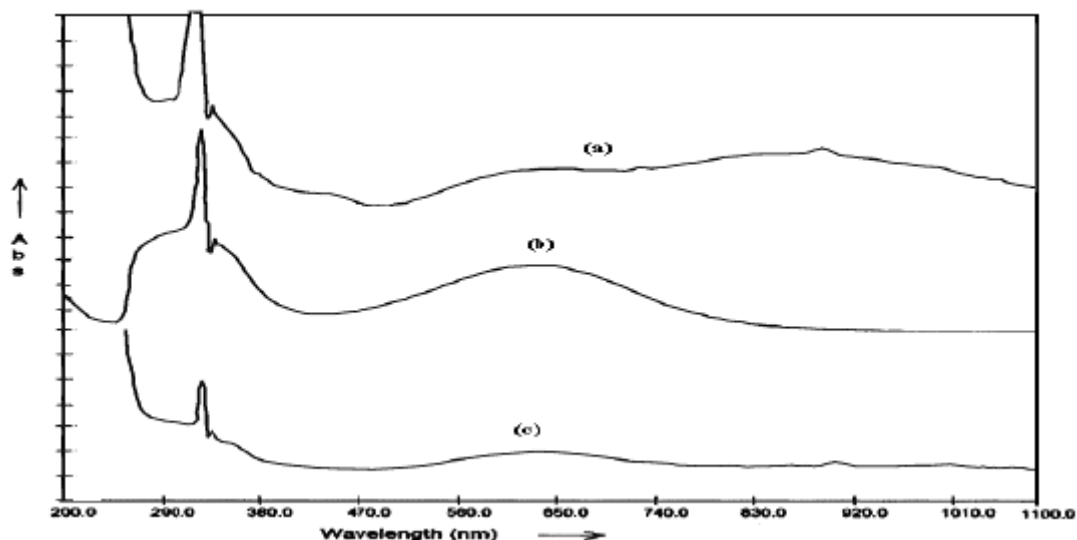


Fig. 3. UV-Visible spectrographs of: (a) PANI, (b) WO₃-PANI (50-wt%) and (c) WO₃-PANI (75-wt%).

3.5. FT-IR Study

In FT-IR spectra the band at 2924 cm⁻¹ in Fig. 4 (a) is attributed to N-H, C=N and C=C stretching modes for the quinoid (N=Q=N) and benzenoid (N-B-N) rings occurred at 1575 cm⁻¹ and 1465 cm⁻¹, the band at about 1322.5 cm⁻¹ corresponds to C-N stretching while the peak at 1162.5 cm⁻¹ is related to protonated C-N group. Peak at around 1050 cm⁻¹, the aromatic C-H in plane bending modes are in evidence. It is clear from Figs. 4 (b and c) that the incorporation of WO₃ particles in PANI leads to the shift of few bands of PANI. The bands at 1465 cm⁻¹, 1322 cm⁻¹, 1162.5 cm⁻¹ which are corresponding to the stretching modes of C=N, C=C, C-N, shifted to lower wave numbers. These obvious changes reveal that the bond strengths of C=N, C=C, C-N become weaker [14] in composite and incorporation of WO₃ particles has an effect on doping of conducting PANI. This means that there is a strong interaction between PANI macromolecule and tungsten oxide particles. Formation of heterojunctions takes place. Because tungsten is a transition metal, it has intense tendency to form coordination compound with (N⁺) atom in polyaniline macromolecule. This supports the results from UV-Visible spectra (Fig. (3 (b) and (c))).

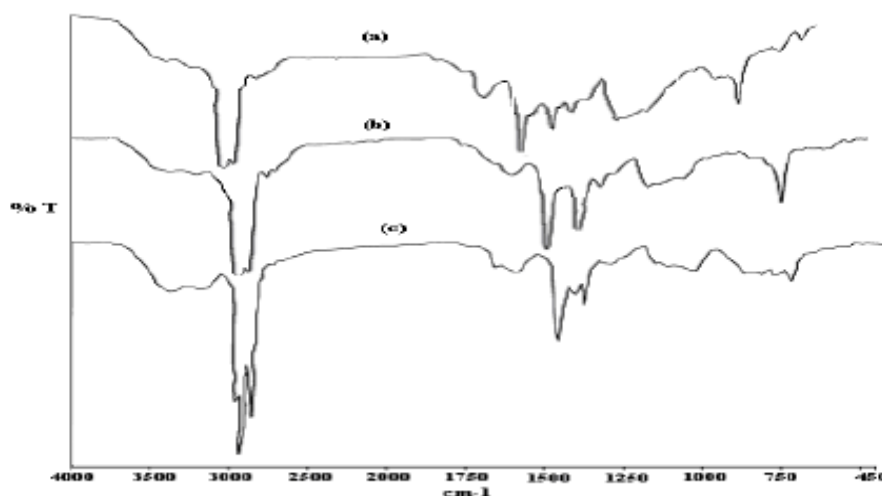


Fig. 4. FT-IR spectrographs of: (a) PANI, (b) WO₃-PANI (50-wt%) and (c) WO₃-PANI (75 wt%).

3.6. Electrical Conductivity

The semi-conducting natures of PANI and WO₃-PANI compositions were observed from the measurements of conductivity with reciprocal of temperature (Fig. 5). The semi-conducting nature in PANI must be due to large oxygen deficiency in it. The adsorption chemistry of WO₃-PANI surface would be different from the pure PANI thick film surface. The WO₃ misfits on the surface are the places where the oxygen species adsorbs. The WO₃ misfits distributed evenly on the surface would have made it possible to adsorb the oxygen ions even at low temperatures (~300 °C). From Fig. 5 it is clear that, the conductivity of thick films of PANI is somewhat more than that of the WO₃-PANI compositions. This can be attributed to the increase in potential barrier due to intergranular distance by introducing WO₃ in PANI. The conductivities of all the films were observed to increase with temperature indicating negative temperature coefficient of resistance. This behavior confirmed the semi-conducting nature of PANI and WO₃-PANI.

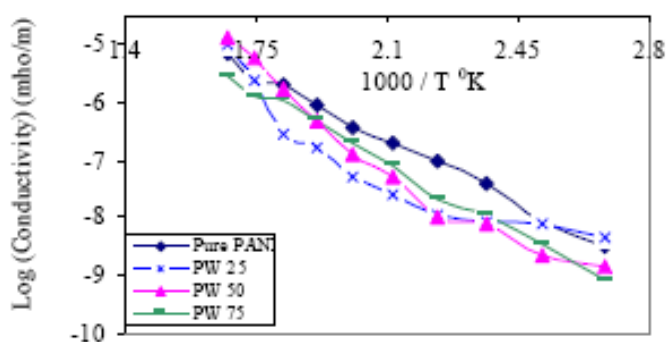


Fig. 5. Conductivity- temperature profile.

4. Sensing performance

4.1. Measurement of Gas Response, Selectivity, Response and Recovery Time

Gas response (S) is defined, as the ratio of change in conductance of the sensor on exposure of the target gas to the original conductance in air medium. The relation for S is as:

$$S = (I_g - I_a) / I_a,$$

where, I_a : is the electrical current of sensor in air medium and I_g : is the electrical current of sensor in gaseous medium. Selectivity or specificity is defined, as the ability of a sensor to respond to certain gas in the presence of more gases. Percentage selectivity factor of one gas over other is defined as, the ratio of the maximum response of other gas to the maximum response of the target gas at optimum temperature.

$$\% \text{ Selectivity factor} = (S_{\text{gas}} / S_{\text{target gas}}) * 100 \%$$

The time taken for the sensor to attain 90 % of the maximum change in conductance on exposure to the target gas is the response time. The time taken by the sensor to get back 90 % of the original conductance is the recovery time.

4.2 Sensing Performance of Pure PANI

Fig. 6 depicts the variation of gas response with operating temperature of pure PANI thick films for 1000-ppm gases. The gas responses were observed to increase with operating temperature, attain their respective maxima and fall down with further increase in operating temperature. This may be attributed to the fact that the oxygen adsorption is poor at lower temperature and maximum at a particular optimum temperature. The responses to various gases of pure PANI were observed to be poor, which may be attributed to the poor oxygen adsorption on the surface of the thick film samples. Also, thick films of pure PANI were observed to sense ammonia and chlorine, indicating less selectivity to a particular gas. So, it is the need to modify the base material (PANI) to improve its gas sensing performance and selective nature.

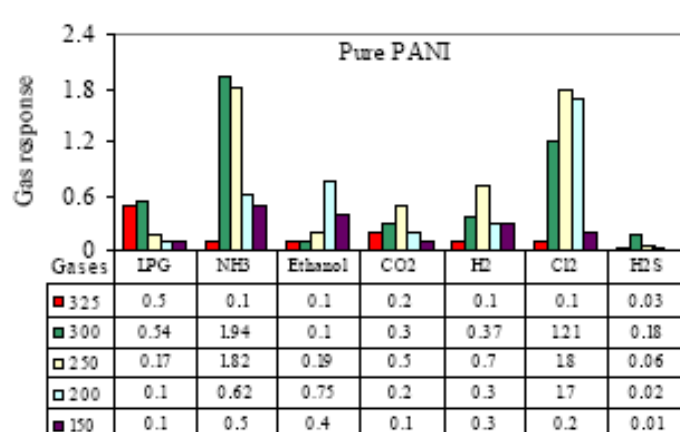


Fig. 6. Gas response of thick films of pure PANI.

4.3. Sensing Performance of WO₃-PANI Composites

4.3.1. Gas Response and Operating Temperature

It is clear from Fig. 7 that the gas response increases with operating temperature, reaches to maximum at 325 °C, and falls with further increase in operating temperature. At 325 °C, the film surface chemistry was favorable to adsorb the larger amount of oxygen, which would facilitate the sensor to oxidize the target gas (LPG) immediately giving faster and larger gas response within 2-3 sec. The LPG may burn before reaching the surface of the film at higher temperature (> 325 °C). Hence, gas response may decrease above 325 °C. In PANI/ WO₃ composite sample, the grains of WO₃ may reside in the intergranular regions of PANI. Thus the effective surface area was expected to increase crucially. N-type-WO₃/P-type polyaniline composite forms hetero-junctions. These hetero-junctions are discretely distributed as a bulk part of sample. The composite sample is in emeraldine base form of polyaniline. It was reported by Zhe Jin et al that when gas is absorbed on an emeraldine base polyaniline film, it could react with imine Nitrogen atom to generate protonated form of polyaniline resulting in significant changes in electrical conductivity [27]. In our case there is strong interaction between polyaniline macromolecule and WO₃. Presence of WO₃ helps to activate this reaction. When absorbed gas could react with imine Nitrogen atom in polyaniline, the coordination bond between tungsten oxide and Nitrogen atom in polyaniline may break down and more charges are available for conduction resulting in significant change in electrical conductivity in case of composite sample than pure polyaniline sample. Microstructure-SEM shows that the chains of compositions of WO₃ and PANI show the optimum porosity with the sample of (50 wt %) WO₃ in PANI. The optimum porosity would help to reach the gas in the interstitial sites on exposure. It also inferring that an increase of the conjugation length in PANI chains and effective charge transfer between WO₃ and PANI [19].

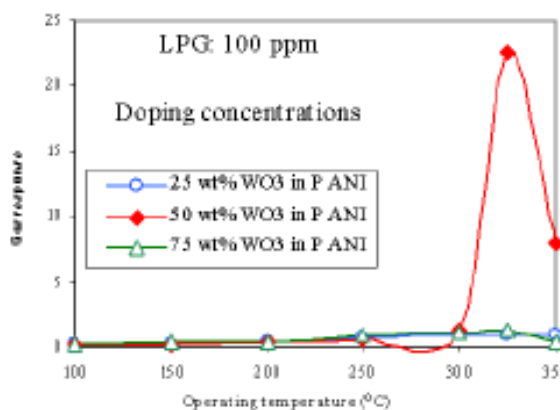


Fig. 7. Gas response and operating temperature

4.3.2. Active Region of Sensor

For thick films of WO_3 -PANI composites, the response was observed to increase continuously with increasing the gas concentration up to 100 ppm at 325 °C (Fig. 8). The active region of the sensor would be between 25 to 100 ppm; as the rate of rise of response is larger during this region. At lower gas concentrations, the unimolecular layer of gas molecules would be expected to form on the surface, which would interact with the surface more actively giving larger response. There would be multilayers of gas molecules on the sensor surface at the higher gas concentrations resulting in saturation in response.

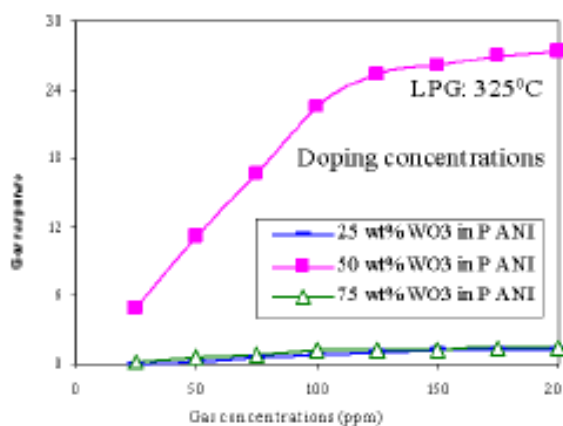


Fig. 8. Gas response and gas concentration.

4.3.3. Selectivity for LPG Against Various Gases

Fig. 9 depicts the selectivity of thick films of WO_3 -PANI composites to 100 ppm of LPG gas against various gases (1000 ppm) at 325 °C. It is clear from Fig. 8 that in contrast to pure PANI, the composites showed not only enhanced response towards LPG but also very high selectivity.

4.3.4. Response and Recovery Profile

The response and recovery profiles of 50 wt % WO_3 in PANI sensor are represented in Fig. 10. The response of the sensor was quick (14 s) and the recovery was also fast (38 s). The quick response may

be due to faster oxidation of LPG. The negligible quantity of the surface reaction product and its high volatility explains its quick response and fast recovery to its initial chemical status.

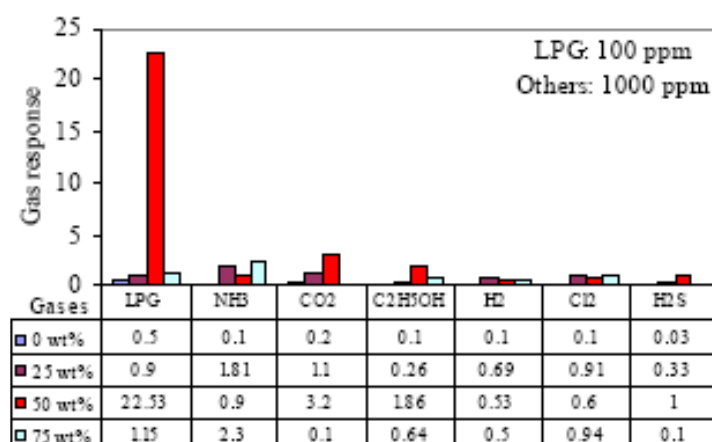


Fig. 9. Gas response to various gases.

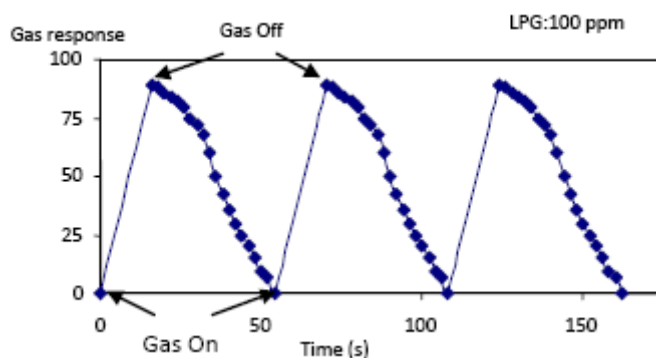


Fig. 10. Response and recovery profile.

5. Discussion

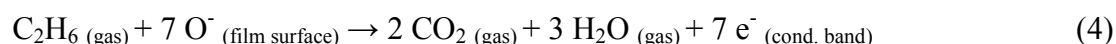
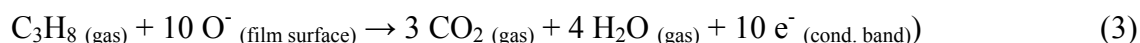
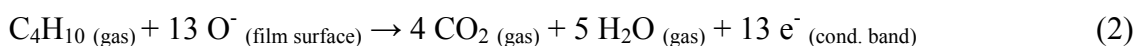
It is observed from UV-Vis absorption spectra (Figs. 3), that on addition of tungsten oxide, a broad peak at around 850 nm is removed totally (Figs. 3 b and c), and shifted at 650 nm. This blue shift is attributed to charge transfer from the benzoid of polyaniline to the quinoid units [27]. The charge compensation of the positive charge on WO_3 takes place at hetero-junctions. During polymerization of aniline in acidic medium with the help of $\text{NH}_4(\text{SO}_3)_8$ as oxidizing agent leads to the formation of $[\text{ANI}]^+$ radical cation which interact with another $[\text{ANI}]^+$ and then propagate as usual. Surface of WO_3 must be positively charged in acidic polymerization bath. Therefore adsorption of large amount of anions (O^-) seems likely to occur and compensate the positive charges on WO_3 surface. Beside this charge compensation, specific adsorption of anion (O^-) on the oxide surface may take place. These specifically adsorbed anions would work as the charge compensator for positively charged Nitrogen (N^+) atom in polyaniline macromolecule. Such charge compensation at n- WO_3 /p-polyaniline heterojunction leads to non-conducting emeraldine base form of Polyaniline. In short, on addition of optimum amount of tungsten oxide into Polyaniline, oxygen adsorption capability of the WO_3 /polyaniline composite is enhanced.

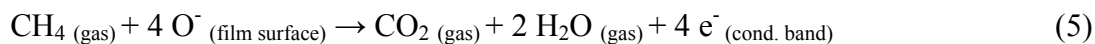
FTIR spectra of PANI/WO₃ (Fig. (4 b and c)), show few bands which are corresponding to the stretching modes of C=N, C=C, C-N, shifted to lower wave numbers. These obvious changes reveal that the bond strengths of C=N, C=C, C-N become weaker [14] in composite. This means that there is a strong interaction between PANI macromolecule and tungsten oxide particles. Formation of heterojunctions takes place. Because tungsten is a transition metal, it has intense tendency to form coordination compound with (N⁺) atom in polyaniline macromolecule. This supports the results from UV-Visible spectra. In PANI/ WO₃ composite sample, the grains of WO₃ may reside in the intergranular regions of PANI and N-type-WO₃/P-type-polyaniline composite forms heterojunctions. These heterojunctions are discretely distributed as a bulk part of sample. The composite sample is in emeraldine base form of polyaniline. It was reported by Zhe Jin et al that when gas is adsorbed on an emeraldine base polyaniline film, it could react with imine Nitrogen atom to generate protonated form of polyaniline resulting in significant changes in electrical conductivity [27]. In our case there is strong interaction between polyaniline macromolecule and WO₃. Presence of WO₃ helps to activate this reaction. When adsorbed gas could react with imine Nitrogen atom in polyaniline, the coordination bond between tungsten oxide and Nitrogen atom in polyaniline may break down and more charges are available for conduction resulting in significant change in electrical conductivity in case of composite sample than pure polyaniline sample. 50 wt% of WO₃ incorporated in pure PANI is the most sensitive element in the LPG gas sensing. Oxygen adsorption-desorption mechanism explains the sensing. Gas sensing is surface phenomena. Surface texture and its porosity are the most important phenomena in gas sensing. Addition of a typical material into the base material affects its texture and porosity. If material to be added is smaller than optimum, it would not create appropriate surface chemistry and sensor would show relatively smaller sensitivity (25 wt% of WO₃ incorporated in pure PANI). If material to be added is larger than optimum, particle size would be increased which would affect porosity. This would suffers reactivity and in turn sensitivity (75 wt% of WO₃ incorporated in pure PANI). Optimum (50 wt% of WO₃ incorporated in pure PANI) concentration of additive would enable surface to adsorb maximum number of oxygen ions. Largest are the oxygen ions adsorbed, maximum would be the number of electrons released for conduction into the material, largest would be the sensitivity, fastest would be the oxidation of the exposed target gas and faster would be transient response of the sensor.

Fig. 11 shows LPG sensing mechanism. Barrier height before exposure of is high (Fig. 11(a)) and therefore the resistance is high. On exposure of LPG, abstracted electrons (by adsorbed oxygen) are released (due to the oxidation of LPG) and resistance of the sensor abruptly reduced as indicated in Fig. 11 (b). Detail reaction chemistry of adsorption and desorption is elucidated as follows. As the butane is the major constituent (55 vol%) of LPG, it requires high temperature (~300...400 °C) to dissociate into lower alkanes. Carbon-carbon and carbon-hydrogen bonds are quite strong due to strong Vander Waals forces. They break only at high temperatures resulting in carbon and hydrogen separation. The atmospheric oxygen O₂ adsorbs on the surface of the thick film. It captures the electrons from conduction band as:



It would result in decreasing conductivity of the film. When alkanes react with oxygen, a complex series of reactions [14-16] take place, ultimately converting the alkanes to carbon dioxide and water as:





This shows n-type conduction mechanism. At higher temperature, molecular oxygen O_2 becomes O_2^- and alkanes decompose producing hydrogen ions H^+ in the reaction. The anion super-oxide O_2^- reacts with H^+ giving water molecule and molecular oxygen O_2 :



Catalase



LPG gas on exposure decomposes into carbon and hydrogen species, which react with adsorbed oxygen, liberating the captured electrons into conduction band resulting in enhancing the catalytic activity of the film surface. The high volatility of the products explain itself the fast recovery of the sensor.

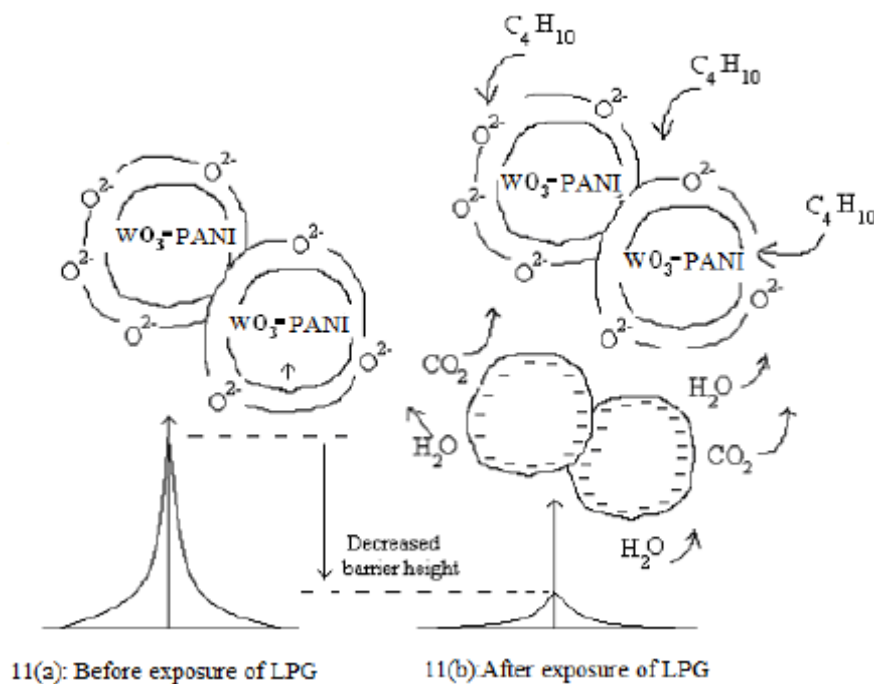


Fig. 11. LPG sensing mechanism of the sensor.

6. Summary

From the results, following statements can be made for the sensing performance of WO₃-modified PANI sensors.

- 1) Pure PANI was almost insensitive to LPG and LNG gases.
- 2) Among various additives tested WO₃ in PANI is outstanding in promoting the LPG gas sensing.
- 3) 50 wt % of WO₃ incorporated in pure PANI is the most sensitive element to LPG gas.
- 4) WO₃ modified PANI has the potential of fabricating the LPG sensor.
- 5) The sensor showed very rapid response and recovery to LPG gas.
- 6) The sensor has good selectivity to LPG against NH₃, CO₂, Cl₂, H₂, H₂S and C₂H₅OH.

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