



Synthesis of ZnO/polyaniline Nanocomposite and its Application as Liquefied Petroleum Gas Sensor

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ABSTRACT: In the present work, we report a Liquefied Petroleum Gas (LPG) sensor based on zinc oxide/polyaniline (ZnO/PANI) nanocomposite material, with fast response and recovery at room temperature. ZnO nanoparticles of size ~25 nm were obtained by using sol gel technique. These ZnO particles were then used for synthesizing ZnO/PANI nanocomposite by in situ polymerization technique. Pure PANI was synthesized using oxidative polymerization technique. For the determination of phase and morphology of ZnO, PANI and ZnO/PANI, basic characterization techniques of X-ray diffraction (XRD) and transmission electron microscopy (TEM) were used. The characterization results showed the formation of ZnO/PANI nanocomposite material, where both ZnO and PANI retained their individual characteristic properties. To test the LPG sensing, pellets of ZnO, PANI and ZnO/PANI were subject to different concentrations of LPG. ZnO exhibited a substantial response to 400 ppm of LPG only at a temperature of 150°C and showed no response at room temperature. At the fixed concentration of 400 ppm of LPG, repetitive cycles of response and recovery were recorded for ZnO sensor at a temperature of 150°C. For pure PANI pellets, no response to LPG was observed at any temperature. ZnO/PANI based sensor was tested for LPG concentration ranging from 25-400 ppm. Interestingly a monotonic increase in sensing response to LPG was observed, which is critical for a practical sensor. ZnO/PANI sensor, was then subjected for repetitive cycles of fixed concentration of 400 ppm of LPG. Interestingly ZnO/PANI sensor response to LPG was observed to be greater as compared to ZnO sensor and that too at room temperature, which is desirable for a good sensor. Room temperature functionality ensures a lesser power consumption and safe operation. The ZnO/PANI sensor developed in this work can prove to be vital for the realization of an economical and efficient room temperature functional LPG sensor.

Keywords: Nanocomposite, Gas sensor, Liquefied Petroleum Gas (LPG), nanoparticle, nanometer,

I. INTRODUCTION

In today's world, research activity intended at improvement of sensors for hazardous gases with a quick response time has increased at a very rapid rate [1-2]. One of the hazardous gases which has been widely used in domestic and commercial areas is liquefied petroleum gas (LPG). The main attributes of LPG are absence of colour and odor. However, its characteristic smell is due the addition of ethyl mercaptan, which is done for detection of any leakage so that precautionary measure can be taken well in time. Even for low concentrations, LPG tends to form a flammable mixture with air, thus becomes a potential source of fire or explosion. High concentrations of LPG in air results in displacement of oxygen leading to aesthetic effect and also suffocation. There is wide range of application of LPG in areas ranging from household fuel source, to vehicles or large-scale industrial activities. Hence due to the harmful effects of LPG to human health, environment, and its explosive nature, a thorough monitoring of the LPG level has become crucial. In the recent years many studies have been reported on exploring different materials to be used as gas sensors, broadly falling into the category of organic and inorganic materials [3-10]. These inorganic materials are easy to synthesize, have stable nature and are economical. However, the main drawback is the high operational temperature which makes the sensor operation unsafe and there is more power consumption [11-13]. As an alternative to metal oxide sensors,

organic conducting polymers have emerged as potential candidates for gas sensors [14-18]. The organic polymers such as polyaniline (PANI), polypyrrole (PPY) etc. have been widely investigated for room temperature LPG sensing [19-23]. PANI is a favourable candidate for sensor application due to its electrical properties, stable nature and economical cost of production [24-30]. However, poor mechanical strength and low response to LPG are the serious concerns with PANI. As an alternative, there is an option to fabricate inorganic/organic composite material which can show a remarkable improvement in the sensing properties [31-34]. Patil *et al.*, fabricated polyaniline/ZnO composite for LPG sensing and reported sensing behaviour for 1000 ppm of LPG [35]. Lokhande *et al.*, reported CdSe/PANI based LPG sensor with room temperature functional operation [36]. However, it is desirable to develop a sensor which is operational at room temperature and can respond to a wide range of concentrations of LPG. The goal of the present research work is to develop an efficient, economical and room temperature functional LPG sensor. ZnO is a stable material however its response to LPG occur at high temperature, which make the cost of operation of sensor high and also high temperature is unsafe due to explosive nature of LPG. Hence in the present work we have fabricated ZnO/PANI nanocomposite material-based sensor, having useful LPG sensing properties of both ZnO and PANI and tested the sensor at room temperature.

II. EXPERIMENTAL DETAILS

A. Synthesis of ZnO nanoparticles

ZnO nanoparticles were synthesized by a simple and economical sol gel process. Initially the precursor salt, zinc acetate ($Zn(Ac)_2 \cdot 2H_2O$) was dissolved in 250 mL of the methanol as solvent. The solution was then subjected to continuous stirring and sonication for 30 minutes. The solution transformed from turbid to transparent after 20 minutes. This clear solution was then transferred to a bigger flask, maintained at a constant temperature of 65°C under reflux condition. After 5 hours, white precipitate was observed, which was then collected by centrifugation. The precipitate was then dried at 80°C for 2 hours to remove the solvent. The dried precursor was then annealed at 300°C for 3 hours, which resulted in formation of pure ZnO devoid of any traces of salt used in the process. This pure ZnO was used for further characterization and formation of ZnO/PANI nanocomposite material and sensor.

B. Synthesis of ZnO/PANI nanocomposite

To synthesize ZnO/PANI nanocomposite material, ZnO nanoparticles were dispersed in a solution containing aniline monomer and HCl. The solution was maintained at a temperature of 5°C, to avoid any secondary reaction. After 30 min. ammonium persulfate was added drop wise which acted as oxidant. The ZnO/PANI nanocomposite formed in the process was then collected by simple filtration and dried at a temperature 50°C for 2 hours. The dried material appeared as lumps, so it was grounded into fine powder using mortar pestle. This fine powder of ZnO/PANI was used for further characterization. Pure PANI was also synthesized using above technique of oxidative polymerization and was then made into fine powder. The fine powder of ZnO, PANI and ZnO/PANI were separately compressed to obtain the pellets of 1.5 cm diameter and 1 mm thickness by applying a pressure of 100 MP using a hydraulic press. On each pellet corresponding to ZnO, PANI and ZnO/PANI, two top contacts of circular geometry were made by thermal evaporation of Au. These pellets were tested for LPG sensing using two probe method. The electrical currents of ZnO, PANI and ZnO/PANI nanocomposite material in air (Ia) and in the presence of LPG (I_g) were measured and using the following relation the gas response was calculated.

$$S(\%) = \frac{I_a - I_g}{I_a} \times 100$$

$$\text{Or } S\% = \frac{\Delta I}{I_a} \times 100$$

For different concentrations of LPG, the response and recovery time of the sensor was recorded for a fixed potential of +2 V

III. RESULTS AND DISCUSSION

A. Structural and Morphological Studies

The combined XRD pattern of ZnO, PANI and ZnO/PANI are shown in Fig. 1. The typical XRD pattern for the ZnO nanoparticles corresponds to hexagonal wurtzite structure (JCPDS No. 36-1451). The average crystallite size of ZnO was calculated to be ~ 25 nm using FWHM value of the most intense peak, from the line broadening of the XRD peaks using Scherrer's formula. The X-Ray diffraction pattern of PANI clearly exhibited peaks at $2\theta = 150, 200$ and 250 . The characteristic peaks at $2\theta = 200$ and 250 corresponds to parallel and perpendicular periodicity to the polymer chain [37-39].

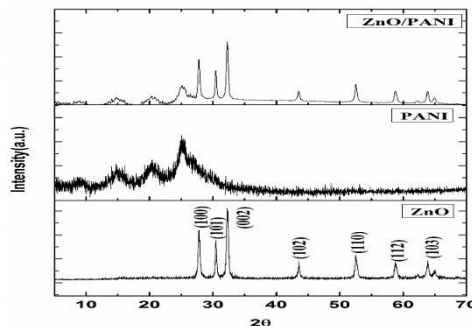


Fig. 1. X-Rays diffraction pattern of ZnO, PANI and ZnO/PANI.

The XRD pattern of ZnO/PANI nanocomposite exhibited XRD peaks corresponding to ZnO and PANI respectively, indicative of retention of the parent materials. This is especially useful to harness the useful properties of both the materials for gas sensing application. Fig. 2(a) explicitly shows the TEM image of ZnO nanoparticles annealed at 300°C. The particle size was observed to be distributed over a range and used to determine the mean crystallite distribution. Fig. 2 (a) shows TEM image of ZnO nanoparticles which are annealed at 300°C (b) HRTEM image of ZnO showing wurtzite morphology (c) size distribution of ZnO showing average crystallite size of 25 nm (d) TEM image of PANI showing fibrous morphology (e) TEM image of ZnO/PANI nanocomposite.

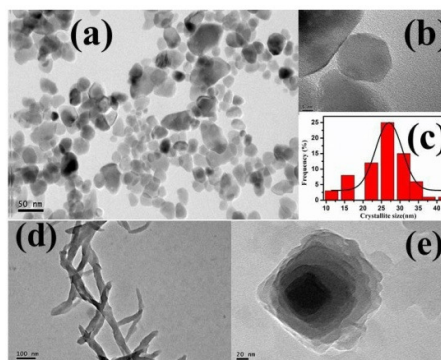


Fig. 2

HRTEM revealed a distorted hexagonal (wurtzite) morphology, as shown in Fig. 2(b). The size distribution of 300°C annealed sample, with a mean crystallite size of 25 nm is shown in Fig. 2(c). To investigate the morphology of pure PANI a separate reaction was set up to carry out oxidative polymerization of polyaniline. In the reaction vessel TEM grids were appropriately placed which were subsequently drawn out of the reaction vessel after completion of the reaction and dried at room temperature. Fig. 2(d) shows a well-connected fibrous morphology of PANI with diameter in nanometer scale. Fig. 2(e) shows the TEM image of ZnO/PANI nanocomposite with ZnO nanoparticle in the core and PANI deposition around it.

B. System for testing sensor

The sensing system consisted of a metallic test cell to place the sensor, mass flow controller to control LPG concentration, labview program and Keithley 2450 source meter as shown in Fig. 3. Pellets of ZnO, PANI and ZnO/PANI nanocomposite were each separately placed in the test cell fitted with spring contacts. The stage of test cell also had a heater, to provide the

requisite temperature to the sensor, if needed. For ZnO/PANI sensor all measurements were done at room temperature.

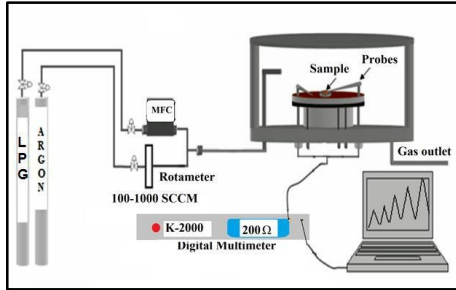


Fig. 3. Gas sensing arrangement with mass flow controller, source meter and sample holder.

C. LPG sensing studies

LPG sensing was carried out on pellets of ZnO, PANI and ZnO/PANI nanocomposite employing two-probe method. After recording the I-V characteristic in air at room temperature, controlled quantity of LPG \sim 400 ppm was introduced into the test chamber and I-V characteristics were again recorded for 10, 15, 20 and 25 seconds as shown in Fig. 4(a)

It is observed from Fig. 4(a) that pure ZnO didn't showed any response to LPG at room temperature. On the other hand for pure PANI a slight variation in the current was observed on exposure to LPG as shown in Fig. 4(b) and (c) shows the marked variation in the resistance of ZnO/PANI heterojunction for 400 ppm of LPG at room temperature. Thereafter LPG sensing measurements were extensively performed on pellets of pure ZnO and ZnO/PANI nanocomposite.

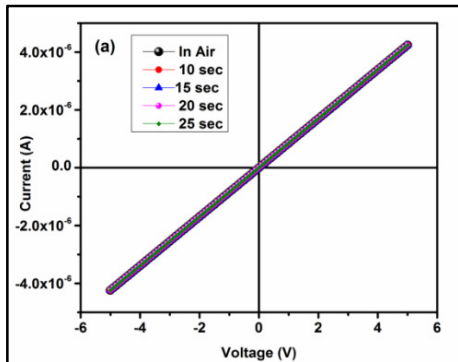


Fig. 4. (a) I-V characteristics of ZnO under exposure of 400 ppm of LPG.

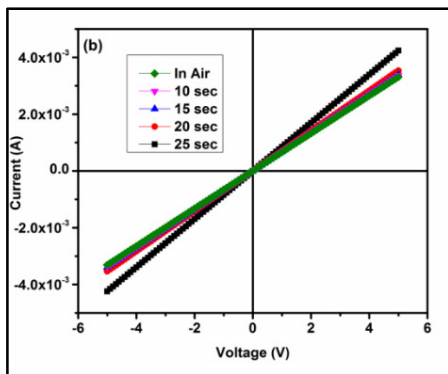


Fig. 4. (b) I-V characteristics of PANI under exposure of 400 ppm of LPG.

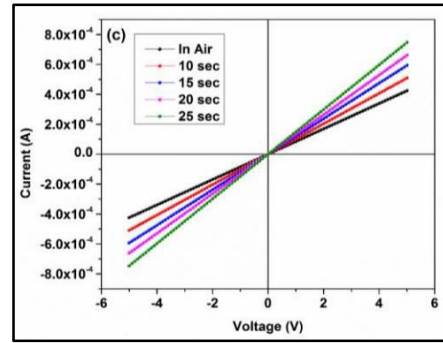


Fig. 4 (c) I-V characteristics of ZnO/PANI nanocomposite under exposure of 400 ppm of LPG.

The sensor based on ZnO/PANI nanocomposite was tested to LPG in the range 25-400 ppm at room temperature, as shown in Fig. 5(a). The gas response exhibited a prominent increase from 5% to 55% corresponding to 25 ppm and 400 ppm of LPG without any saturation. This remarkable ability of ZnO/PANI sensor developed in this work to sense LPG at room temperature is of great practical relevance. For further sensing measurements, 400 ppm of LPG was selected. Practical sensors should be able to sense the target gas multiple times, which ensures the practicability of the sensor. ZnO/PANI sensor was subjected to multiple repetitive cycles of 400 ppm of LPG as shown in Fig. 5(b). The response and recovery cycles were observed to be consistent for ZNO/PANI sensor, showing a critical characteristic of a good sensor. For a comparative study sensor based on pure ZnO was subjected to 400 ppm of LPG. Fig. 5 (c) shows the gas response of ZnO sensor, recorded at different temperatures. There was no response of ZnO sensor towards LPG at room temperature however when the ZnO sensor was heated to an elevated temperature of 150°C, the response was observed. The gas response further increased when operational temperature was increased to 250°C. However, for ZnO sensor, we selected 150°C as operational temperature for further LPG sensing so to work with minimum temperature required for its functionality. Fig. 5(d) shows the repetitive cycles of response and recovery of ZnO sensor towards 400 ppm of LPG at a temperature of 150°C. However, the observed response and recovery time was greater than the ZnO/PANI sensor. Also, high operational temperature of 150°C in case of ZnO sensor was a disadvantage in view of extra power consumption, finally adding to the cost of sensor operation. Pure PANI pellets were not able to exhibit any prominent response to LPG at any temperature ranging from 25°C to 80°C. Further heating PANI pellets resulted in the degradation of the polymer. In view of all the sensing investigations, it was found that ZnO/PANI nanocomposite-based sensor developed in this work is more economical and practical as compared to the sensors based on pure ZnO and pure PANI. To further ascertain the practical working conditions of the sensor, the performance of the sensor in dry air and in moist air has been carried out for evaluation at 400 ppm of LPG, as shown in Fig. 6(a). The sensor response was higher in dry air and it decreased when relative humidity was increased. This can be attributed to the decrease in the resistance of the ZnO/PANI on increasing humidity. The response time for 400 ppm of LPG at room temperature is 35 s and recovery time is 55 s, which further shows the relevance of ZnO/PANI nanocomposite sensors.

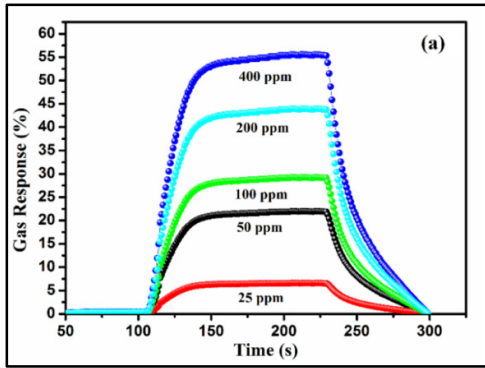


Fig. 5. (a) Shows response of ZnO/PANI to LPG in the range 25-400 ppm at room temperature.

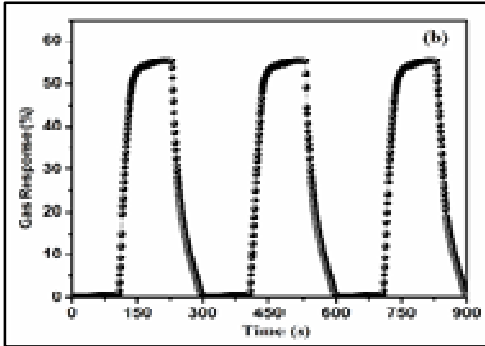


Fig. 5 (b) Shows the repetitive response of ZnO/PANI sensor to 400 ppm of LPG at room temperature.

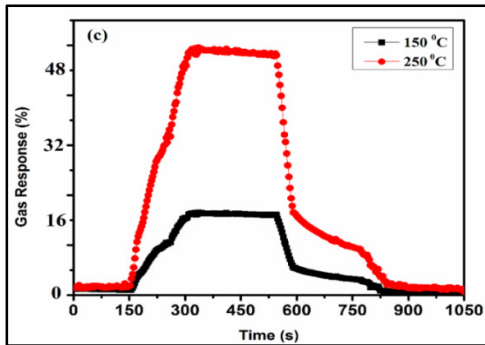


Fig. 5. (c) shows the response to 400 ppm of LPG at 150°C and 250°C.

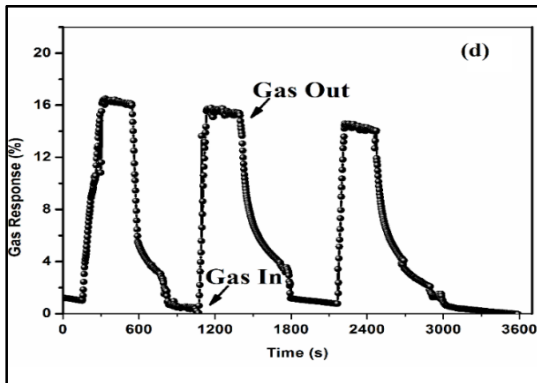


Fig. 5 (d) Shows the repetitive response of ZnO sensor to 400 ppm of LPG at 150°C.

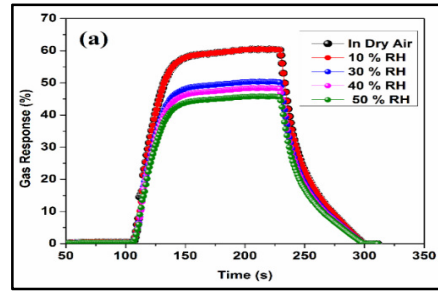


Fig. 6. (a) Performance of the ZnO/PANI sensor in dried air and in moist air.

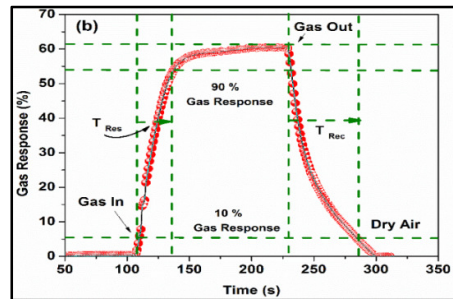


Fig. 6 (b) Steady state response has been demonstrated, for a fixed concentration 400 ppm of LPG.

Practical sensors should be able to sense the target gas for a large range which ensures the practicability of the sensor. ZnO/PANI sensor was subjected to LPG in the range 25-400 ppm, the corresponding calibration curve is shown in Fig. 7(a), indicative of non-saturation of sensor response.

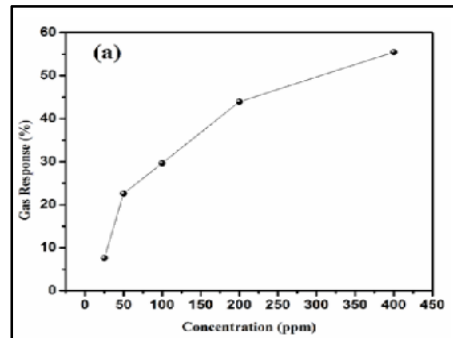


Fig. 7. (a) Shows calibration plot of gas responses of ZnO/PANI sensors versus concentration of LPG in the range 25-400 ppm.

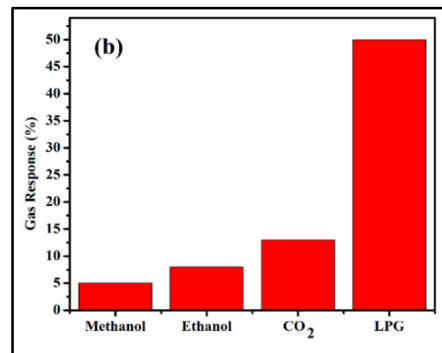


Fig. 7(b) Response of ZnO/PANI sensor to LPG and different organic vapours.

Another important characteristic of a practical sensor is its selectivity to the target gas. Here we have investigated the response of ZnO/LPG sensor for potentially interfering gases like methanol, ethanol and CO₂ as shown in Fig. 7 (b). Out of all the gases, the best response of ZnO/PANI sensor was observed for LPG gas which was approximately 10, 8 and 4 times as compared to methanol, ethanol and CO₂. This selective response of ZnO/PANI sensor towards LPG further proved its usefulness as a practical sensor.

D. LPG sensing mechanism

The sensing mechanism of ZnO sensor towards LPG at elevated temperature is due to electrons generation mediated by thermal excitation, which is absent at room temperature [40]. The schematic of LPG sensing by ZnO is shown in Fig. 8. Exposure of ZnO surface to the ambient leads to the adsorption of gas molecules. These gas molecules further capture the electrons, from the conduction band and forms oxide ions. Exposure of ZnO surface to the ambient leads to the adsorption of gas molecules. These gas molecules

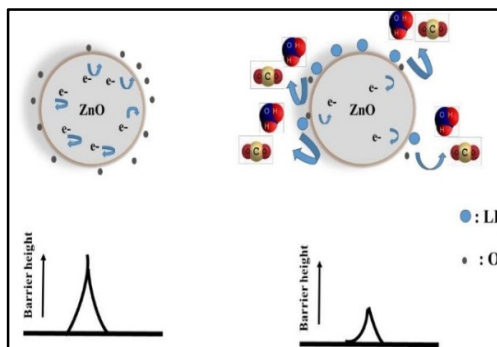


Fig. 8. Schematic of LPG sensing by ZnO sensor.

further captures the electrons from the conduction band and forms oxide ions. The interaction of LPG with these oxide ions results in the release of trapped electrons in the conduction band and decreasing the barrier height. LPG on reaction with surface oxygen gets converted into CO₂ and H₂O. On the other hand ZnO/PANI forms numerous p-n heterojunctions throughout the surface and the charge separation occurs at the polymer-nanoparticles interface.

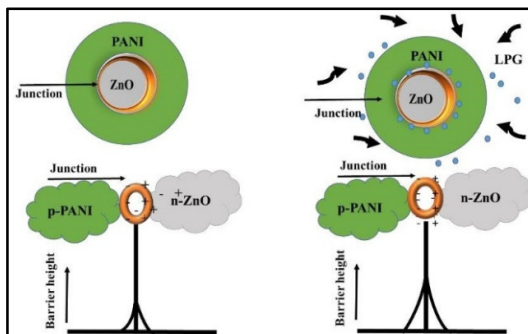


Fig. 9. Shows variation of barrier height of ZnO/PANI sensor on interaction of LPG at junction.

The LPG sensing mechanism of ZnO/PANI heterojunction sensor can be well explained by potential barrier dependent model as shown in Fig. 9. When LPG molecules reaches the interface of PANI and ZnO, the potential barrier height alters and hence there is a change in the current across the junction, at room

temperature [41]. From attributes like room temperature LPG sensing, minimal effect of humidity on sensor, it is concluded that p-polyaniline/nZnO heterojunction can be a potential candidate for room temperature LPG sensor application.

IV. CONCLUSIONS

The fabrication of highly sensitive, selective and room temperature operational ZnO/PANI nanocomposite-based LPG sensor is reported. A remarkable response and recovery is exhibited by ZnO/PANI to LPG in a wide range varying from 25 ppm to 400 ppm. On subjecting the sensor to repetitive cycles of 400 ppm of LPG consistent gas response was observed, a critical attribute of an efficient sensor. These properties and low cost of fabrication makes ZnO/PANI nanocomposite-based sensor a very effective for room temperature detection of LPG.

Conflict of interest. No.

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