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## Development of electrospun polyaniline/ZnO composite nanofibers for LPG sensing

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### Abstract

In this paper we report the highly sensitive at low temperature and low concentration of liquefied petroleum gas (LPG) sensor based on D-Camphor-10-Sulphonic acid (CSA) doped Polyaniline/Polyethylene Oxide (PANI/PEO) and PANI/ZnO/PEO nanofibers fabricated by electrospinning technique. Nano-sized particles of zinc oxide (ZnO) were synthesized using sol-gel method. The emeraldine base form of polyaniline (PANI) and PANI/ZnO synthesized by chemical oxidative polymerization were treated with CSA for better solubility and conductivity and then blended with PEO solution to get nanofibers using electrospinning. The morphology and structure of electrospun nanofibers of CSA doped PANI/PEO and PANI/ZnO/PEO were investigated by SEM, UV-VIS, FTIR and XRD. The change in electrical resistance of CSA doped PANI/PEO and PANI/ZnO/PEO for 1000 ppm of LPG was measured. Both the samples showed a rapid and reversible resistance change upon exposure to LPG gas at 1000 ppm concentration. Maximum sensitivity was achieved at 36 °C for PANI/ZnO/PEO indicating suitability of nanofibrous PANI/ZnO/PEO towards LPG sensing as compared to pure ZnO and PANI.

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*Keywords:* Zinc oxide; Polyaniline; Nanofibers; Nanocomposites; Electrospinning; LPG Sensing.

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### 1. Introduction

Liquefied Petroleum Gas (LPG) is the mixture of hydrocarbons mainly propane and butane. Accidental leakage of LPG even at low concentrations creates a serious threat to human lives and personal safety as it is a flammable gas. The Lower Explosive Limit (LEL) as specified by National Institute for Occupational Safety and Health (NIOSH) and Occupational Safety and Health Administration (OSHA) standards for chemical hazards is 21,000 ppm (2.1% by volume in air) for propane and 19,000 ppm (1.9% by volume in air) for butane. The

Permissible Exposure Limit (PEL) for LPG as specified by NIOSH and OSHA standards is 1000 ppm. LPG is primarily used as fuel for vehicles and as cooking gas for household applications. Precise monitoring of leakages of LPG even at low concentrations can be beneficial to prevent accidental explosions, (Dutta et al., 2011). Sensors have become an integral part of the modern civilization owing to its significance, where metal oxides have played a major role as reliable sensor materials. Recent advances in the development of nanostructured catalysts such as metal oxide nanoparticles, nanowires, nanorods and nanobelts provide the opportunity to greatly increase the response of these materials, as sensor performance is directly related to granularity, porosity, and ratio of surface area to volume in the sensing element, (Ghosh et al., 2010). Zinc oxide (ZnO) a wurtzite n-type semiconductor which is in its different forms with unique properties, such as, direct band gap (3.37 eV), high exciton binding energy (60 meV), and good resistivity ( $10^{-3}$  to  $10^5$   $\Omega\text{cm}$ ) is one amongst the widely explored functional metal oxide semiconductor, (Kondawar et al. 2014, Chougule et al., 2012). Gas sensor devices commonly employ metal oxide semiconductors, the principle of which is based on the change in conductivity by interaction with gas molecules and this property is mostly dependent on the operating temperature. Thus, metal oxide gas sensors require high operating temperature. Moreover such gas sensors have generally disadvantages of poor selectivity and low sensitivity to the very low concentration of gases. Therefore several different approaches have been explored in order to overcome these issues. Recently conducting polymers found place as a gas sensor because of chief merits such as easy synthesis and room temperature operation (Dhawale et al., 2008). The conducting polymers have improved many aspects of the gas sensors especially in lowering the operating temperature to around room temperature. In addition to this, the ability to incorporate specific binding sites into conducting polymers promises the improvement of selectivity and sensitivity of material.

Among the various conducting polymers, Polyaniline (PANI) has been investigated as a potential material for gas sensing applications, due to its controllable electrical conductivity, environmental stability and interesting redox properties associated with the chain nitrogen's. It is the unique type of conducting polymer in which the charge delocalization can, in principle, offer multiple active sites on its backbone for the adsorption and desorption of gas analyte (Kargirwar et al., 2011). However, PANI is not as sensitive as metal oxides towards gas species, and its poor solubility in organic solvents limits its applications, but it is suitable as a matrix for preparation of conducting polymer nanocomposites (Kondawar et al., 2009) and (Deshpande et al., 2009). Therefore, there has been increasing interest of the researchers for the preparation of nanocomposites based on PANI for gas sensing applications. Hybridization of metal oxide and conducting polymer could improve the properties of pure metal oxides or conducting polymers based gas sensor and the new synthesized material shows the synergistic effect of these two materials. The Nanofibers of polymeric materials have large aspect ratio, large surface area to volume ratio and large porosity, such materials are best for sensing of different gases due to larger absorptive capacity for gas analyte. Among various methods to build nanoscale structure, electrospinning (ES) is prominent as a facile and versatile approach to produce long uniform nanofibers of organic, inorganic and organic/inorganic hybrid materials (Lin et al., (2012). There have been a large number of reports on high performance one-dimensional nanosized sensitive materials fabricated via ES (Lee et al. 2011, Pinto et al. 2011 and Barnes et al. 2006). However, it was found that great challenges exist while trying to integrate the electrospun nanofibers into electrical/electronic devices (Liu et al. (2004). One of the obstacles is the realization of good adhesion of electrospun layer to substrate and good electrical contact. Joshi et al. (2008) and Dhawale et al. (2008) developed various thin films based heterojunction as room temperature LPG sensors.

In present investigation, experiments have been carried out for the fabrication of sensitive and selective LPG sensor based on electrospun nanofibers of PANI/ ZnO nanocomposites. To the best of our literature survey in the field of LPG sensor, there is no report on electrospun nanofibers of PANI/ ZnO nanocomposites based LPG sensor. The nanofibers of PANI/ZnO nanocomposites were synthesized by electrospinning technique and sensing characteristics of the synthesized nanofibers to LPG were systematically investigated.

## 2. Experimental

### 2.1. Materials

Aniline (99.5%) was procured from E. Merck and distilled under reduced pressure prior to use. Zinc Acetate dehydrate  $[(\text{CH}_3\text{COO})_2 \text{Zn} \cdot 2\text{H}_2\text{O}]$  (99%), N-Methyl 2-pyrrolidone (98.5%) Polyethylene oxide (PEO) [Mol. Wt 1,00,000], Ammonium persulphate (98%), and D-Camphor-10-Sulphonic acid  $[(\text{C}_{10}\text{H}_{16}\text{O}_4\text{S})]$  Mol. Wt 232.30] were purchased from Hi-media and used as received. All chemicals were of analytical grade and solutions were prepared with double distilled water.

### 2.2. Synthesis of ZnO nanoparticles

ZnO nanoparticles were synthesized by a sol-gel process using zinc acetate dihydrate as a source for Zn (Chougule et al. 2012). The 5.393g of  $\text{Zn}(\text{Ac})_2 \cdot 2\text{H}_2\text{O}$  was added to 40ml of methanol and mixture was stirred vigorously at 60°C for 1 h to form gel. After gel formation, heating was stopped and the solution was stirred continuously until the formation of a white powder. The powder was separated by filtration, washed several times with methanol and dried by dryer for 20 min. The powder was annealed in zone furnace at 700°C for 1 h to get the ZnO nanoparticles.

### 2.3. Synthesis of PANI/ZnO nanocomposite

In a typical polymerization reaction, PANI was synthesized by chemical oxidative polymerization. To oxidize 0.2M aniline with 0.25M Ammonium Persulphate in acidic aqueous medium, aniline was dissolved in 50ml of 1.0M HCl in a beaker with constant stirring for 15 min. In another beaker, ammonium persulphate (oxidant) was dissolved in 50ml of 1.0M HCl. Both the solutions mixed together with constant stirring for polymerization. During the polymerization process, the colour changed from colourless to light blue and then to dark green. The solution was left at rest and stirring was stopped. Next day, the PANI precipitate was collected on a filter paper, washed with distilled water, then by 20ml of 0.1M HCl and similarly with Methanol. PANI (emeraldine) powder was dried in vacuum at 80°C. This method gives the high yield of the material (Stejskal and Gilbert, 2002). A similar procedure followed for the synthesis of PANI/ZnO nanocomposites, but this time the addition of as-synthesized ZnO nanoparticles with aniline solution followed by sonication was done.

### 2.4. Preparation of solution of PANI/ZnO nanocomposite for Electrospinning

Normally PANI and its composite in emeraldine salt (ES) form are not soluble directly in any organic solvent and it is difficult to process it in conducting form. Therefore, PANI (ES) and its composites were converted into emeraldine base (EB) form using ammonium hydroxide and then protonated with D-Camphor-10-Sulphonic acid (CSA) to make it conducting and soluble for drawing nanofibers (Ghosh et al., 2010). In the typical process, as-synthesized PANI and PANI/ZnO nanocomposites were separately used to prepare PANI (EB) and PANI/ZnO (EB) form. For that, doped PANI and PANI/ZnO i.e. emeraldine salt (ES) precipitate was dissolved separately in 35ml of liquor ammonia solution, with stirring for overnight at room temperature, and filtered next day. The wet powder was washed thoroughly with double distilled water and dried in vacuum at 60°C to obtain emeraldine base, denoted as dedoped PANI and dedoped PANI/ZnO hereafter (Shahi et al., 2011). 0.05g of PANI (EB) and PANI/ZnO (EB) composite powders were separately mixed with D-Camphor-10-Sulphonic acid (CSA) by grinding in a smooth agate mortar for 1h. Then PANI/CSA and PANI/ZnO/CSA mixtures were separately added to 5ml N-Methyl 2-pyrrolidone (NMP) and stirred for overnight to get homogeneous solutions (Shahi et al., 2011 and Ghosh et al., 2010). Next day, 2 ml of 10% Polyethylene oxide (PEO) solution (dissolved in hot distilled water) in PANI/CSA/NMP and PANI/ZnO/CSA/NMP solution separately at room temperature to make it viscous for the fabrication of nanofibers by electrospinning process.

### 2.5. Preparation of electrospun nanofibers of PANI / ZnO nanocomposite

Nanofibers of PANI/ZnO nanocomposite were prepared by electrospinning technique. The process of spinning fibers with the help of electrostatic forces is known as electrospinning, (Subbiah et al., 2005). Electrospinning is a process of creating solid continuous fibers of material with diameter in the micro- to nanometer range by using electric fields. Compared to mechanical drawing, electrospinning produces fibers of thinner diameters via a contactless procedure, (Garg and Bowlin, 2011). PANI/CSA/PEO and PANI/ZnO/CSA/PEO solutions were used to fabricate nanofibers via electrospinning. Nanofibers were drawn by ESPIN-NANO machine purchased from PECO Chennai-India. The basic elements of the electrospinning apparatus consists of a hypodermic syringe (2 ml disposable syringe), a high voltage power supply (0-50kV), a grounded collector (Aluminum foil) and a syringe pump to control the flow rate of solutions. About 2 ml of the solution was placed in the hypodermic syringe and the collector was grounded and situated at 18 cm from the tip of the needle of syringe. The flow rate was auto-controlled to 0.4 ml/hr. As the voltage applied to the needle increased to about 18 kV (Shahi et al., 2011), the electric force on the polymer droplet at the end of the needle overcomes the surface tension and a polymer jet exits toward the cathode in short bursts, (Pinto et al., 2011). As the solvent evaporates in air, thin fibers of PANI/CSA/PEO and PANI/ZnO/CSA/PEO of varying diameters and lengths covered the substrate.

## 3. Results and discussion

Structural information of the doped PANI and PANI/ZnO nanocomposites obtained using FT-IR spectra which were obtained in the range from  $400\text{ cm}^{-1}$  to  $4000\text{ cm}^{-1}$  with a Bruker Alpha infrared spectrometer whereas the optical characteristics were studied using UV-Vis absorption spectroscopy (UV-1800 Shimadzu Spectrophotometer) in the wavelength range of 200–800 nm. The X-ray diffraction (XRD) measurements of PANI and PANI/ZnO nanocomposites were performed with Rigaku Miniflex-II instrument having Cu-K $\alpha$  radiation source of wavelength  $1.540\text{ \AA}$  and the scattered radiation diffraction patterns were collected within the range of  $2\theta = 10 - 70^\circ$ . The surface morphology of the prepared nanofibers was investigated by Scanning Electron Microscopy (SEM) with Carl Zeiss EVO18. The liquefied petroleum gas (LPG) sensing behavior of the CSA doped PANI/PEO and PANI/ZnO/PEO composite nanofibers was analyzed by measuring the change of an electrical resistance of the nanofibers in air and LPG exposure in temperature range of 30 - 90°C. The electrical resistance was measured using programmable Keithley Instruments Inc. Model 2000.

### 3.1. UV-Vis Spectroscopy

To investigate the interaction of ZnO nanoparticles with PANI, the UV-visible absorption spectra of synthesized nanofibers were recorded and analyzed. The UV-visible spectrum corresponding to pure PANI, PANI/ZnO and pure ZnO are shown in Fig. 1. The absorption peak of ZnO was recorded at 380 nm. It had shown two absorption bands characteristic of peak PANI at wavelengths 288 nm and 388 nm. The absorption band in the UV region is attributed to the chain of the aromatic nuclei and corresponds to the  $\pi-\pi^*$  transitions and the absorption band appearing in the visible region, attributed to an interaction between the benzenic nuclei and the quinone di-imine structure. The UV-visible spectrum of the PANI/ZnO has an absorption peak at 383nm, which is blue shifted compared with the absorption peaks of PANI (388nm) and of ZnO nanoparticles (380 nm) (Chougule et al., 2008). This may be because of interactions between PANI chains and ZnO nanoparticles which cause easy charge transfer from PANI to ZnO via hydrogen bonding (Talwar et al., 2014 and Olad et al., 2012).

### 3.2. Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectra of PANI and PANI/ZnO nanofibers are shown in Fig. 2. The characteristic peaks of emeraldine salt form of PANI at  $1592\text{ cm}^{-1}$  (C=C stretching mode of the quinoid rings),  $1481\text{ cm}^{-1}$  -  $1440.56\text{ cm}^{-1}$  (C-C stretch in benzenoid ring and C-H mixed vibrations),  $1411.74\text{ cm}^{-1}$  (C-C stretch in quinoide, C-H bending in benzenoid ring),  $1297.38\text{ cm}^{-1}$  (C- N stretching, C-H bending) and  $1141.53\text{ cm}^{-1}$  (S=O bonding for camphor sulfonic acid),  $808.78\text{ cm}^{-1}$  (C-H out of plane bonding in benzenoid ring) and  $661.10\text{ cm}^{-1}$  -  $720.92\text{ cm}^{-1}$  (C-C, C-H bonding mode

of aromatic ring) appear in the FTIR spectrum of PANI-ZnO composite, indicating the formation of PANI in the composites. The peaks in the range  $3377.92\text{ cm}^{-1}$  corresponds to N-H band stretching vibration. Comparing to the corresponding peaks of pure PANI, the peaks of PANI/ZnO shifted towards lower wave number (Chougule et al., 2012). This shifting of peaks may be due to the action of hydrogen bonding between the hydroxyl groups on the surface of ZnO nanoparticles and the amine groups in the PANI molecular chains. Such interaction between PANI and ZnO nanoparticles is also observed by Lee et al. (2011), Zolriasatein et al. (2012) and Patil et al. (2012).

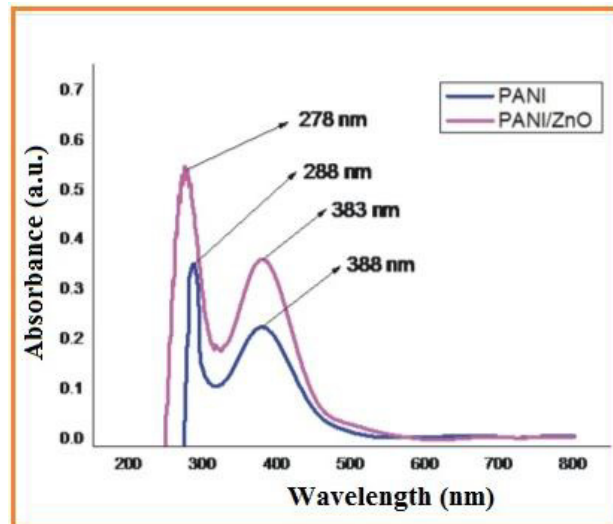


Fig. 1: UV-Vis spectra of pure PANI and PANI/ZnO nanocomposite

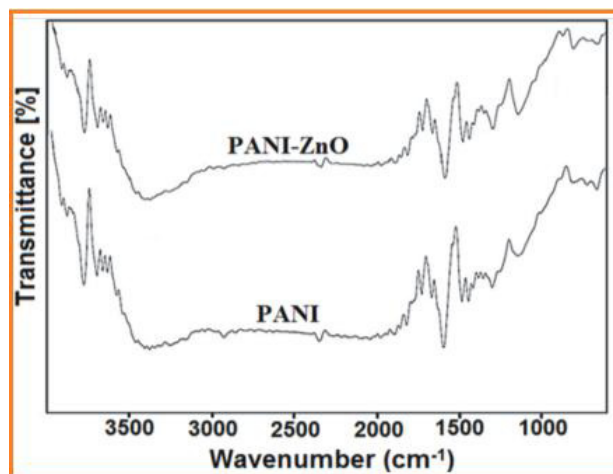


Fig. 2: FTIR spectra of PANI and PANI-ZnO nanofibers.

### 3.3. X-ray Diffraction (XRD):

Analysis of X-ray diffraction (XRD) patterns was used to investigate the structure of PANI/ZnO nanocomposite. Fig. 3 shows the XRD patterns of pure PANI and PANI/ZnO nanocomposite. The characteristic peaks of PANI were found at  $2\theta=15.3$ ,  $20.4$  and  $26.28$  corresponding to  $(0\ 1\ 1)$ ,  $(0\ 2\ 0)$  and  $(2\ 0\ 0)$  crystal planes of PANI. For PANI/ZnO, intense peaks at  $32^\circ$   $(1\ 0\ 0)$ ,  $34.5^\circ$   $(0\ 0\ 2)$ ,  $36.5^\circ$   $(1\ 0\ 1)$  and small peaks were found at  $48^\circ$   $(1\ 0\ 2)$ ,  $50.36^\circ$   $(1\ 1\ 0)$  and  $64.08^\circ$   $(1\ 0\ 3)$  which become more intense as we increase wt% of ZnO powder. It was found from

the XRD plot that with the addition of ZnO nanoparticles during polymerization, the crystalline nature of PANI powder enhanced significantly (Talwar et al., 2014, Olad et al., 2012 and Ding et al., 2012). The diffraction peaks of PANI/ZnO composites have been indexed to the hexagonally wurtzite structured ZnO which were well matched with that in JCPDS file 36-1451. Similar observations of increase in lattice parameters were also reported by Ahmed et al, (2011).

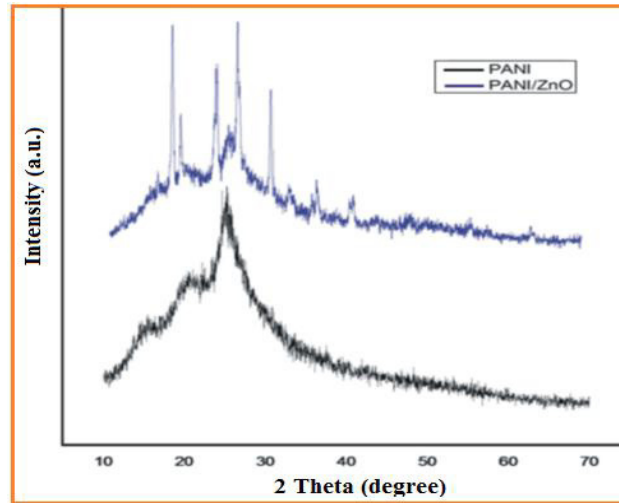


Fig. 3: XRD pattern of PANI and PANI/ZnO nanocomposite

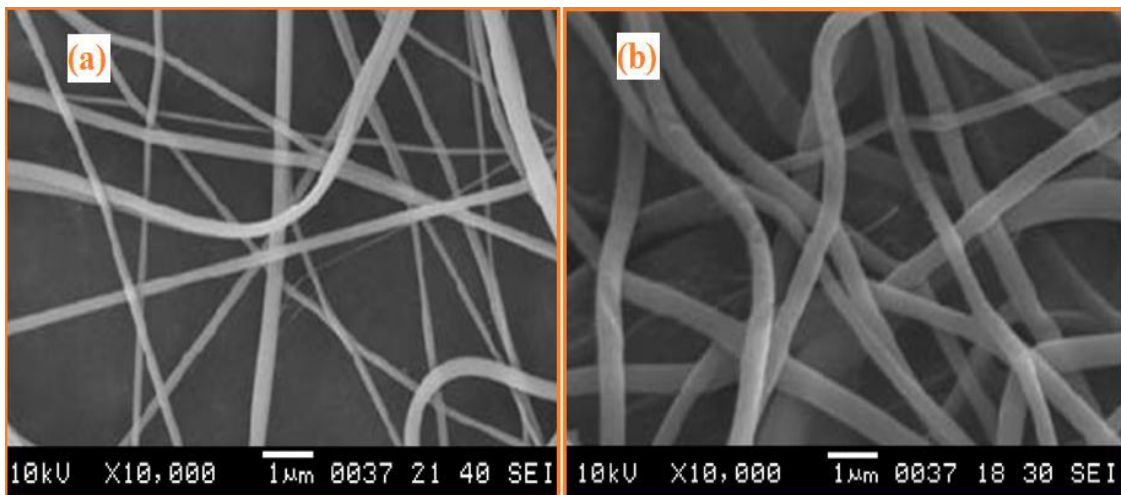


Fig. 4: SEM images of (a) PANI nanofibers and (b) PANI/ZnO nanofibers

#### 3.4. Scanning Electron Microscopy (SEM)

SEM images of PANI and PANI/ZnO nanofibers are shown in Fig. 4(a) and 4(b) respectively. SEM images show smooth fibers with several millimeters in length and diameter in the range of about 200 nm to 300 nm. While comparing images of PANI and PANI/ZnO, fibers of composite are well aligned with more porosity and encapsulation of PANI over ZnO due to which the diameter of composite increased indicating the adsorption of LPG gas molecules by composite nanofibers as compared to that of pure PANI



### 3.5. LPG sensing

In order to test the gas sensing ability of composite nanofibers, the gas sensor unit was specially designed as shown in Fig. 5. Through the external connections, temperature controlled current-voltage (I-V) characteristic of the conducting nanofibers was recorded using Keithley 2000 Multimeter. I-V characteristics of the composite nanofibers before and after exposure of gases were recorded for 1000ppm concentration. From I-V characteristics, the change in resistance was noted. The electrical resistance of composite nanofibers in air ( $R_a$ ) and in the presence of gas ( $R_g$ ) was measured.

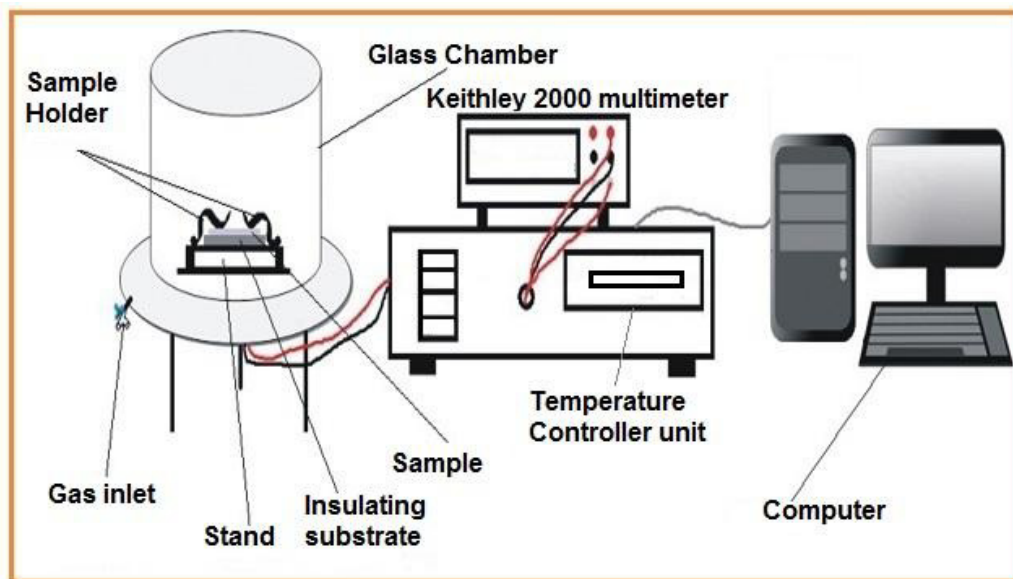


Fig. 5: Schematic Diagram of laboratory Gas Sensor set up

### 3.6. Dynamic Response and Sensitivity

The change in electrical resistance of the composite nanofibers was measured at varying temperatures from room temperature to higher temperature (from 30°C to 90°C) in the absence and presence of LPG of 1000ppm. Fig. 6(a) shows the sensitivity of PANI and PANI/ZnO nanofibers with the temperature. The sensitivity was found to be increased with temperature and reached to the maximum sensitivity at 36 °C and then decreased for higher temperature. LPG is reducing gas (Tudorache et al., 2009). In case of PANI nanofibers, sensitivity increased from 1.11% to 7.33% at 36°C but as the temperature increased from 36°C to 90° it gets started to decrease the sensitivity from 7.33% to 1.25%. In case of PANI/ZnO nanofibers same nature was followed but the sensitivity factor increased from 4.55% to 8.73% at 36°C but as the temperature increased from 36°C to 90°C, the sensitivity gets started to decrease from 8.73% to 0.7%. Addition of ZnO in polymer matrix increase in the band gap by which electrical conductivity decreases but the sensing properties enhance. From graph it is revealed that the 36°- 37°C is an optimum operating temperature for LPG, because LPG is highly inflammable gas and at higher temperatures explosion can occur. As described above, the physical properties of conducting polymers strongly depend on their doping levels. Fortunately, the doping levels of conducting polymers can be easily changed by chemical reactions with different metal oxides and carbon based materials at room temperature, and this provides a simple technique to detect various toxic gases and vapours. Most of the conducting polymers are doped / undoped by redox reactions; therefore, their doping level can be altered by transferring electrons. Electron transferring can cause the changes in resistance and work function of the sensing material. The work function of a conducting polymer is defined as the minimal energy needed to remove an electron from bulk to vacuum energy level. This process occurred when PANI and PANI/ZnO composite nanofibers were exposed to LPG and other redox-active gases. Electron acceptors can

remove electrons from the aromatic rings of PANI. When this occurs at p-type conducting polymer, the doping level as well as the electric conductance of the conducting polymer is enhanced (More et al., 2010). An opposite process will occur when detecting an electro-donating gas. However, this mechanism has not been understood clearly. Further studies are still needed to make the mechanism clear. There are some attempts proposed to explain the change of electrical properties of PANI and PANI/ZnO in presence of gases. The first one is based by modification of electrical conductivity of conjugated polymers in presence of oxidizing or reducing agents that acts as dopants for the polymer chain. Another explanation is based on modification of physical properties of the polymers, i.e., morphology, crystallinity, etc in presence of gases that could also explain the change of electrical conductivity through an increase in the interchain electron transfer. In the present case, the action of LPG on sensor active elements based on conducting polymer depend on their adsorption on polymer surface followed by diffusion through the inner- domain spaces (Tudorache et al., 2009).

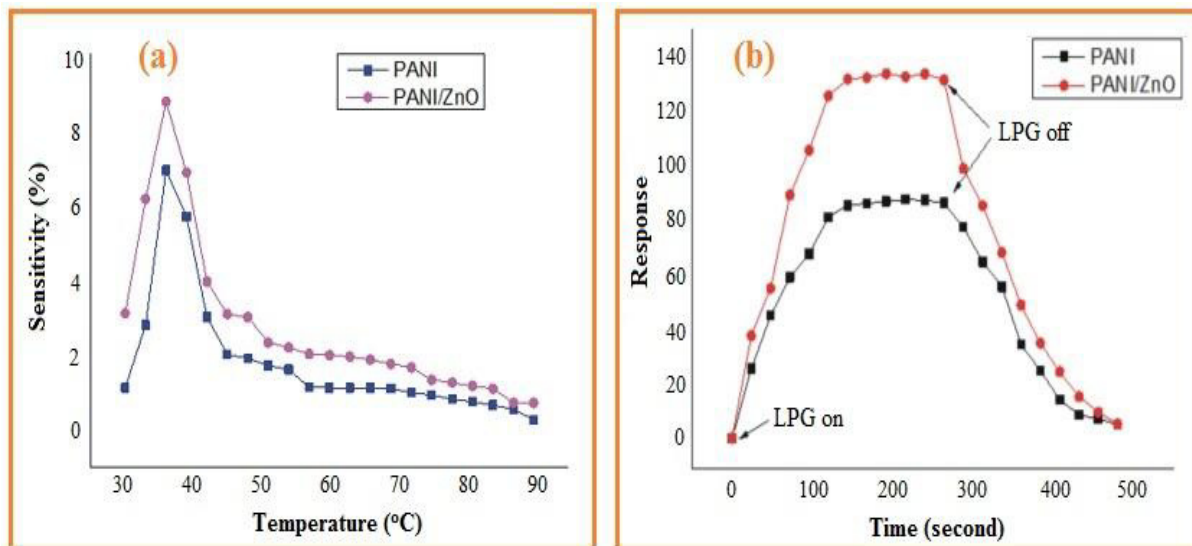


Fig. 6: (a) Sensitivity and (b) Gas response of PANI and PANI/ZnO nanofibers

The response/recovery time is an important parameter used for characterizing a sensor. It is defined as the time required to reach 90% of the final change in resistance, when the gas is turned on and off, respectively. A graphical representation of the response vs. time of PANI and PANI/ZnO nanofibers at 1000ppm concentration for LPG is shown in Fig. 6(b). As the time of exposing the gas to the sensor increases, response of sensor gradually increases and the maximum response was obtained around at 150 seconds for both the samples. The response time was found to be 100 seconds for PANI/ZnO and 110 seconds for pure PANI. As the response approaches to 133.6 for PANI/ZnO and 87.9 for PANI it becomes saturated (Srivastava, 2013). The recovery time was long i.e. 185 seconds for PANI/ZnO and 195 seconds for pure PANI. This may probably be due to the heavier nature of LPG and the reaction products are not leaving from the interface immediately after the reaction (Dhawale et al., 2008). The nanofibers attained the maximum response in short time upon exposure of 1000ppm LPG and dropped rapidly when the gas was removed from testing atmosphere indicating that the sensor has fast response and recovery. While comparing response and recovery of two samples, PANI/ZnO nanofibers are good sensors than pure PANI for LPG at room temperature.

#### 4. Conclusion

PANI and PANI/ZnO composite nanofibers were successfully fabricated by using electrospinning technique. The interaction between ZnO and PANI was confirmed from UV-VIS, FTIR and XRD characterizations. SEM shows the clear nanofibers of as-synthesized PANI and PANI-ZnO composite with diameter in the range 200 – 300 nm.



The sensing properties of PANI and PANI/ZnO composite nanofibers for LPG gas show both the sensors have highest sensitivity nearer to room temperature. While comparing the sensing behaviour for LPG gas, PANI/ZnO composite nanofibers shows fast response and recovery as compared to that of pure PANI nanofibers indicating that the PANI/ZnO is the best candidate (optimized concentration) for LPG detection at room temperature.

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