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Preparation, Structural, Electrical and LPG Sensing Properties Study of Zinc Oxide Doped Polyaniline Composite (PnZnO)

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Polymer composites of polyaniline (Pn) and Zinc oxide doped polyaniline (PnZnO) are prepared by in-situ polymerisation method using ammonium persulphate as oxidising agent. Various composites have been prepared by varying the level of additive material Zinc oxide. The phase and morphology of all the synthesized samples were analyzed using basic characterization techniques such as X-ray diffraction (XRD) and scanning electron microscopy (SEM). The AC electrical conductivity of the samples was measured using the impedance technique within a frequency range of 10KHz to 1MHz at room temperature. Additionally, the dc electrical transport property of the composites was investigated within a temperature range of 30-2000C. The PnZnO composites showed an enhanced dc electrical conductivity with increasing temperature and ZnO concentration in polyaniline. The Activation energies were evaluated from Arrhenius plots for all compositions. The activation energy decreased with increasing ZnO concentration in polyaniline matrix. The activation energy of the prepared PnZno composites decreased in comparison with the activation energy of a Pn, while the electrical conductivity increased as the amount of ZnO was increased. The change in electrical resistance of both Pn and PnZnO composites was measured when exposed to 1000 ppm and 2000 ppm concentration of LPG gas. Both samples exhibited a rapid resistance change upon exposure to LPG gas, with the PnZnO composite demonstrating higher sensitivity and suitability for LPG sensing compared to Pn.

Keywords: Polyaniline (Pn), Zinc oxide (ZnO), LPG sensing, AC, DC, Activation energy, XRD, SEM.

1. Introduction

The organic-inorganic materials have been many advances in the production corresponding to both their chemical and physical properties [1-4]. This particular advanced development in production of such materials has provided them more appealing to researchers in a range of disciplines. One of the considerable studies is organic–inorganic composites with a controlled structure due to the hybrid materials which usually brings enhanced properties of both initially organic as well as inorganic materials [5-7]. Among all the conducting polymers (CP's), polyaniline (Pn) is considered as unique and promising candidate for potential applications in the class of CP's and most widely studied due to their unique properties such as its easy synthesis, low cost and tunable conductivity. Also, the polyaniline is well known for the environment stability. (Bae et al., 2004). It also exhibits dramatic changes in its electronic structure and physical properties on protonation (Mexiang Wan et al 1991, 1992). The insulated emeraldine base of the polyaniline can be made conductive and the conductivity can be enhanced by doping with protonic acids. By doping with protonic acids with polyaniline, which can enhance the conductivity of the composite by more than 10 orders of magnitude. The enhancement of the conductivity usually depends on the strength of the acids [8-9]. The polyaniline exhibiting variety of forms with differ in the physical and chemical properties [10].

Also, the polyaniline (Pn) has been explored as a promising material for many gas sensing applications, due to its controllable electrical conductivity and interesting redox properties associated with the chain nitrogen's. In Pn the charge delocalization can provide multiple active sites on its backbone for the adsorption and desorption of gas analyte. However, with respect to the gas species, the polyaniline is not as sensitive as metal oxides and also its poor solubility in organic solvents which limits the applications of polyaniline. The polyaniline is more suitable as a matrix for synthesis of CP's composites [11-12]. Therefore, there has been increasing interest of the researchers for the preparation of composites based on PANI for gas sensing applications.

The widely used hazardous gas is liquefied petroleum gas (LPG) in domestic and commercial region. The mixture of hydrocarbons is Liquefied Petroleum Gas (LPG) and which mainly contains propane and butane. The main inherences absence of colour and odor. Since, for the leakage detection of the LPG gas, addition of ethyl mercaptan into the LPG to show the significant odor of the LPG when leaked. The low concentration, medium and high concentration of LPG tends to form flammable mixture with air and which place very important role in detection of LPG. The many devices of gas sensors are of metal oxides semiconductors. The metal oxide semiconductors interact with the gas molecules to change its resistance and also which depends on the temperature. Moreover the metal oxide semiconductors need much high operating temperature for lower concentration detection of gas. Therefore, the combination of metal oxide and the polymer such as polyaniline place very important role in gas sensing device due to its controllable electrical conductivity, environmental stability and redox properties [13].

For the detection of H_2, LPG, NO_2, and NH_3 gases, crystalline ZnO is one of the most widely used inorganic substance. Nevertheless, its long-term stability is compromised when exposed to high temperatures, making it imperative to devise sensors that can function effectively at room temperature [14]. A chemist can develop a strong interaction between fbrous materials

and organic polymer matrix to design new man-sized organic-based polymer composite materials [15].

Here, we report the results of Pn and PnZnO composite fabrication using the in-situ method. Compared to other fabrication methods, the in-situ method is obtained using the easiest methods to fabricate composites. To the best of our literature survey in the field of LPG sensor, there is no report on PnZnO composites based LPG sensor with 1000 and 2000ppm and ZnO 50wt%. The PnZnO composites with different weight percentage were synthesized by in-situ technique and sensing characteristics of the synthesized composite to LPG were systematically investigated.

2. Experimental section:

2.1. Materials used:

The chemical reagents utilized in the synthesis process were aniline (99%), ammonium persulfate (APS) (99%), hydrochloric acid (HCl) of analytic grade. The other supplement chemicals were of analytical reagent (AR) grade. All the aqueous solutions were prepared using double-distilled water. The zinc oxide (ZnO) was used to prepare composites via chemical oxidative polymerization method. Pellets of Pn and PnZnO composites with 12.4 mm diameter and 1.18 mm thickness were prepared by applying a pressure of 5 ton.

2.1 Preparation of Polyaniline (Pn)

COP technique was adopted for the preparation of polyaniline from aromatic compound aniline, catalyst as HCL and oxidizing agent as ammonium persulfate. The 0.2M of aniline was prepared in the beaker-1 and it is mixed with the catalyst hydrochloric acid of 1N is prepared in the beaker-2 at room temperature. The mixer of aniline and hydrochloric acid was stirred by magnetic stirrer for 2 hrs at constant RPM for the completion of the reaction. Oxidation of monomer is achieved by using an oxidizing agent ammonium persulfate. Then the solution of 0.25M of ammonium persulfate was prepared in the separate beaker. The prepared ammonium persulfate solution was then added to the above prepared aniline hydrochloride solution drop wise over a period of 1h with continuous stirring and maintained temperature of about 5^0C. After adding the solution of ammonium persulfate, this reaction mixer was continuously stirred in magnetic stirrer for 8 hrs in room temperature. The dark green solution was obtained after the completion of the reaction and it is kept for overnight to sort out the particles at the base of the beaker. The precipitate formed and separated out by filtering by using vacuum pump and washed with deionised water with acetone and 1N HCL to remove other additives present in the PANI. The obtained final suspension was dried in oven at 50° C for 24 hrs. The final product was grinded into powder

2.2 Preparation of PnZnO composite:

Zinc oxide (ZnO) powder of 5%, 10%, 15%, 20% and 25% additive weight percentage is dissolved in the mass fraction after adding the oxidizing agent. After adding the ZnO, this reaction mixer was continuously stirred in magnetic stirrer for 8 hrs in room temperature. The dark green solution was obtained after the completion of the reaction and it is kept for overnight to sort out the particles at the base of the beaker. The precipitate formed and

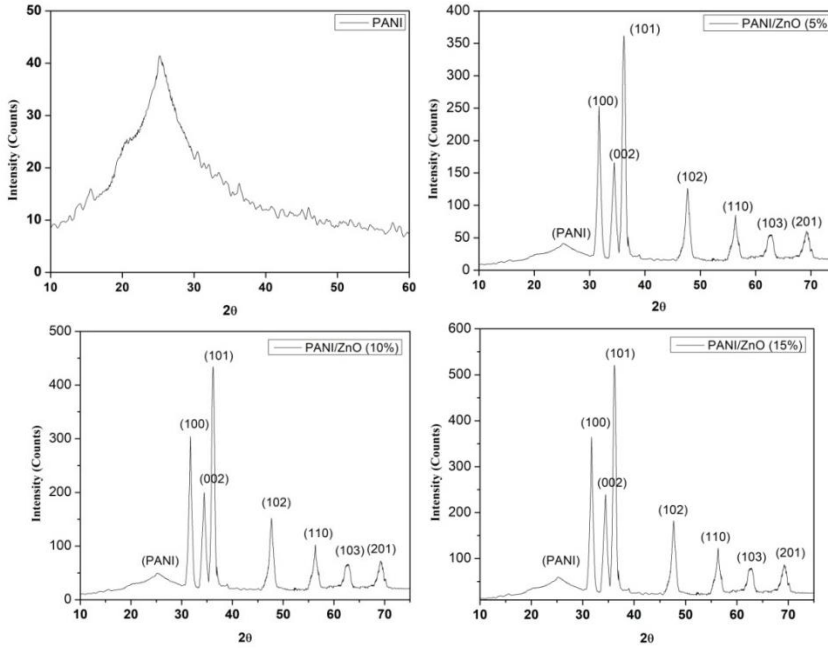
separated out by filtering and washed with deionised water with acetone. The obtained final suspension was dried in oven at 50° C for 24 hrs. The final product was grinded into powder [16].

3. Results and discussion:

3.1 XRD spectra:

The XRD spectra for the pure Pn and PnZnO composite samples were recorded in the range of 20^0 to 75^0 and have been depicted in figure-1. The XRD spectra of prepared polyaniline (Pn) exhibits a broad peak at 2θ angles around 25^0 can be assigned to the scattering from polyaniline at interplanar spacing and which is characteristics of the van der Waals distances between stacks of polyaniline ring [17]. This broad peak shows that Pn's amorphous structure contains crystalline regions.

The XRD spectra of PnZnO composite exhibits the diffraction peaks belongs to both ZnO and Pn which describes the withholding of Pn in the composite material. The XRD spectra of prepared PnZnO composite exhibits well defined diffraction peaks obtained at different 2θ angles 31.7^0, 34.42^0, 36.18^0, 47.68^0, 56.34^0, 62.54^0 and 69.18 corresponding to the hkl planes (100), (002), (101), (102), (110), (103) and (201) for the phase of the zinc oxide and which are well matched with the reported literatures and standard JCPDS data card No. 36-1451 [18-19]. The XRD spectra of composite also exhibit diffraction peak at 25^0 corresponding to PANI. No characteristic peaks other than ZnO were observed. The average crystalline size of PN was calculated by using Scherrer's formula $D = K\lambda / \beta Cos\theta$ (where $\lambda = 1.54060\text{Å}$, θ is the Bragg angle, K is the Debye Scherrer constant and β is the peak full width at half maximum of the peak. The average crystallite size of PnZnO composite was found to be 20 nm.



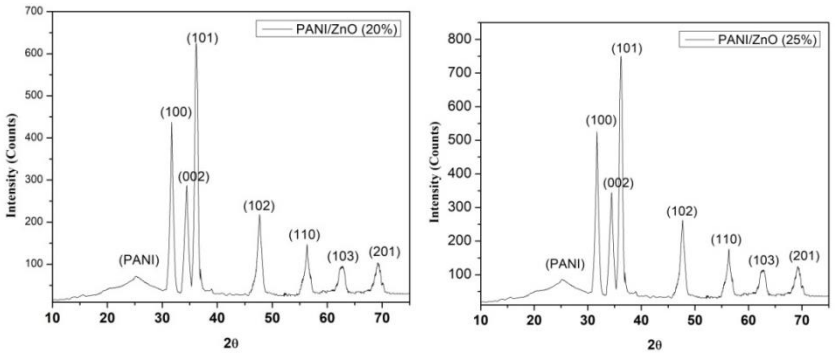


Figure-1: XRD spectra of Pn and PnZnO composites

3.2 SEM micrographs:

The SEM micrographs for the pure Pn and PnZnO composite samples being magnified at 1000 times have been depicted in figure-2. The morphology of the Pn appears to be irregular shapes, non fibrous with high densities. The SEM micrographs of the PnZnO composite suggests irregular arranged granular and flakes with sharp edge, looks non-porous and morphology with spherical, few oval-shaped particles randomly distributed, micro size round shape particles with uniformity on the surface as well as a few agglomerations. The composite micrograph describing surface morphology slightly different from that of the micrograph of Pn suggesting the possible presence of zinc oxide particles distributed in the polyaniline matrix. [20-23].

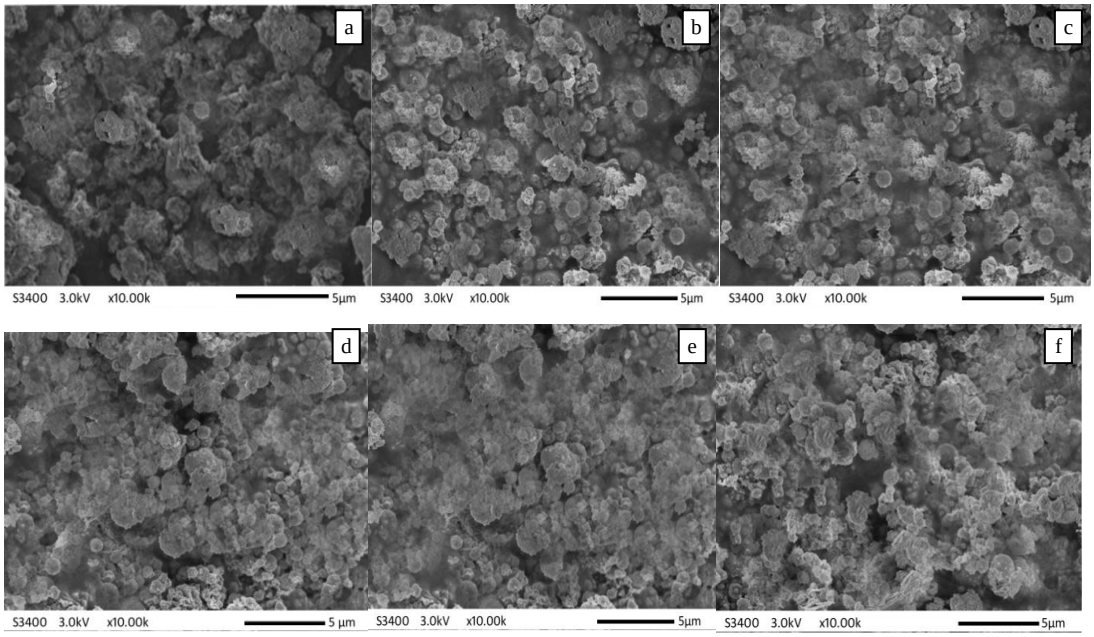


Figure-2: SEM micrographs of Pn and PnZnO composites

3.3 AC electrical conductivity:

The unusual electronic properties in the conducting polymers (CP's) is due to the π-conjugated

system. In polymers, the process of conduction is influenced by hopping mechanism where the transportation of mobile charge carriers, polarons and bipolarons along with the conjugated chains. Then the doping technique was used to introduce charge carriers in to the π-conjugated system [24-26]. The frequency dependent conductivity for disordered materials such as polymers can be due to interfacial polarization at contacts and grain boundaries of the sample [27].The ac conductivity measurements have been conducted by a typical two probe method. The frequency dependent ac electrical conductivity of Pn and PnZnO composite was depicted in fugure-3. The conductivity of both samples increases with increase in frequency. An enhancement of one order of conductivity of PnZnO composite compared to the conductivity of Pn was observed. Figure 3(b) indicates the ac conductivity as function of different wt% of ZnO. The conductivity of the composite increases with increase in the content of zinc oxide in the Pn matrix. This increase in the conductivity of PnZnO composite may due to the even distribution of zinc oxide particles and which is evident from XRD results that the increase in the crystallinity. The improved conductivity in the composite may also due to the hybridization of amorphous and crystalline structure in the composite [28-29].

Among all the prepared composite, the maximum AC conductivity value was obtained with a value of 3.19 x 10^{-2} S/m for 25 wt% ZnO at 1MHz frequency also depicted in figure 3(b). The conductivity of the composite material found increasing from 1.53 x 10^{-2} S/m for 5wt% at 1MHz with increase in the content of zinc oxide and reaching 3.19 x 10^{-2} S/m for 25 wt% ZnO at 1MHz.

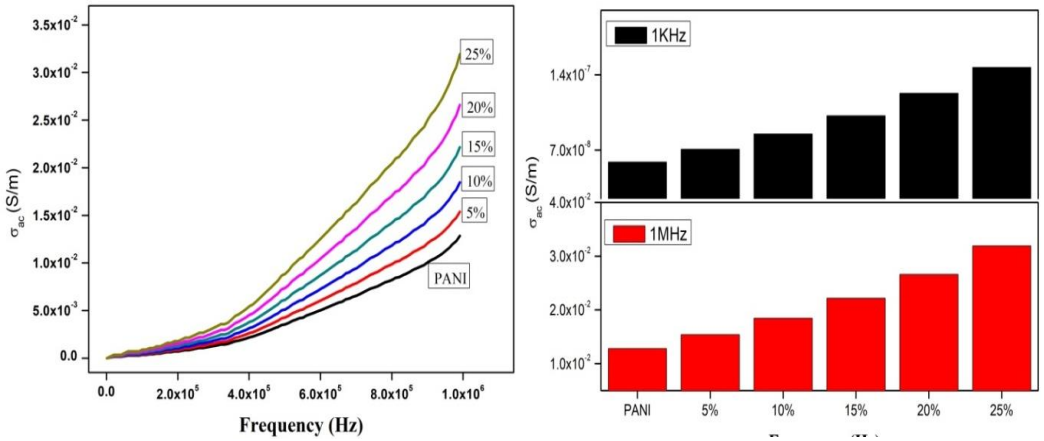


Figure-3: AC electrical conductivity of of Pn and PnZnO composite a) as function of frequency b) as function of percentage of ZnO

3.4 DC electrical conductivity

The temperature dependent dc electrical conductivity was studied to describe the charge transport mechanism in the polymer composites. The variation in the dc conductivity with change in temperature of Pn and PnZnO composites were carried out and represented in figure-4(a). The dc conductivity of all the samples increases with increase in temperature exhibits the semiconductor behavior and it rises with increase in content of ZnO in the Pn matrix. This indicates the ZnO particles gives positive influence on composite towards increase in conductivity. Figure 4(b) indicates the dc conductivity as function of different wt% of ZnO.

The result shows that the ZnO has positive influence on the temperature dependent conducting property of the Pn. The dc conductivity of both samples increases in two phases, i.e., low temperature region and high temperature region. There is higher order increase in the conductivity at higher temperature phase and lowered conductivity at low temperature phase. The increase in the conductivity at higher temperature due to excitation of electrons to the conduction band at higher temperature [30-34]. The dc conductivity of composites increase with increasing temperature and also the conductivity increases as the content of ZnO is increased in the polyaniline matrix [35].

Among the entire prepared composite, the maximum DC conductivity value was obtained with a value of 6.18 x 10^{-4} S/m for 25 wt% ZnO at 180^0 temperature also depicted in figure 4(b). The conductivity of the composite material found increasing from 4.21 x 10^{-4} S/m for 5wt% with increase in the content of zinc oxide and reaching 6.18 x 10^{-4} S/m for 25 wt% ZnO at 180^0 temperature.

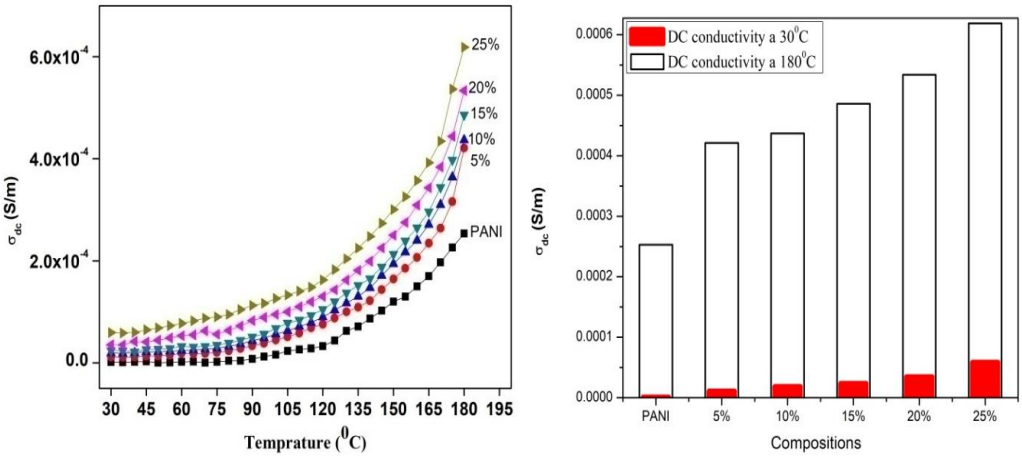


Figure-4: DC electrical conductivity of of Pn and PnZnO composite a) as function of temperature b) as function of percentage of ZnO

3.4.1 Arrhenius Plot and activation energy from dc conductivity:

It is known that the conductivity of known semiconductors, such as silicon and germanium, increases with increasing temperature because of the increased number of charge carriers in the conduction band [36-39]. The mechanism of heating the polymer film above room temperature is thermally induced. The resistance versus temperature behaviour may be understood by the activation energy values of the nanocomposite fibres according to the Arrhenius equation [40]:

$$\sigma = \sigma_0 \exp(-Ea/K_bT) \qquad (1)$$

where Ea is the activation energy (eV); T is the absolute temperature in (K); k_B is the Boltzmann constant, which is 1.38x10^{-23} (J/K) or 8.62x10^{-5} (eV); and σ_0 is the minimum electrical conductivity at 0 K.

Figure 5(a) shows a plot of σ_{dc} vs (1/T) for the different samples tested. The measured electrical conductivity and activation energy values for the different samples were calculated, and are listed in Table 1. The activation energy (eV) as function of different wt% of ZnO was depicted in figure 5(b). The activation energy found to be 0.489ev for Pn and which decreases to 0.178 ev for 25wt%. As shown in the results, the electrical conductivity (σ_{dc}) increased from 1.54x10^{-6} S/m for Pn at 303K to 6.19x10^{-4} S/m for PnZnO with 25 wt.% at 453K.

This means that the electrical conductivity (σ_{dc}) increased by two orders of magnitude PnZnO with 25 wt.% at 453K. The electrical conductivity increased due to the increased density and mobility of the charge carriers. The activation energy of the Pn decreased with increasing polyaniline content, while the electrical conductivity increased with increasing doping rate.

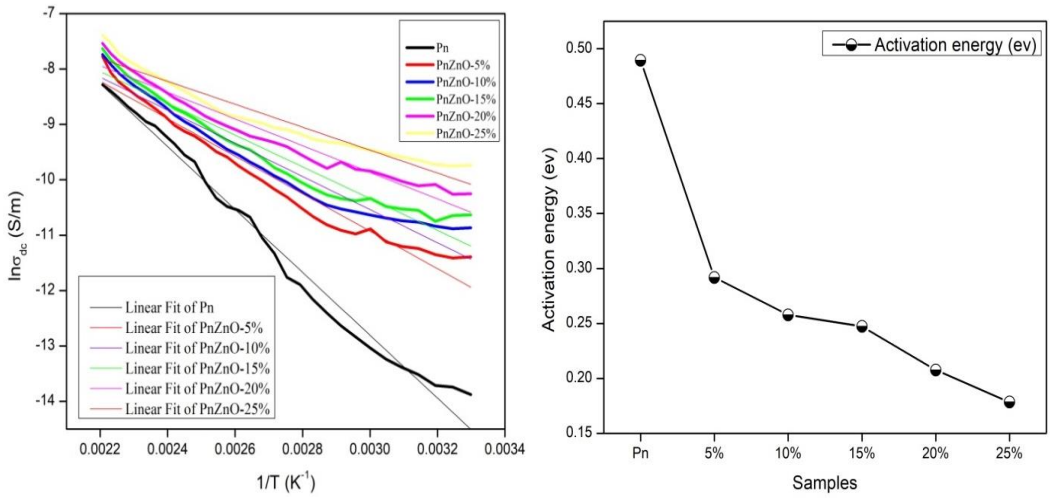


Figure-5: a) Arrhenius plot b) Activation energy

Table-1: DC conductivity and activation energy of Pn and PnZnO composites

Sample	DC conductivity (S/m) at 303.15 K	DC conductivity (S/m) at 453 K	Activation energy in ev
Pn	1.54x10^{-6}	2.53x10^{-4}	0.489
PnZnO-5%	1.13x10^{-5}	4.21x10^{-4}	0.291
PnZnO-10%	1.91x10^{-5}	4.37x10^{-4}	0.257
PnZnO-15%	2.41x10^{-5}	4.86x10^{-4}	0.247
PnZnO-20%	3.52x10^{-5}	5.34x10^{-4}	0.207
PnZnO-25%	5.91x10^{-5}	6.19x10^{-4}	0.178

3.5 LPG sensing studies:

In order to test the gas sensing ability of composite of Pn and PnZnO-25% composite at 1000ppm and 200ppm concentration at room temperature (RT) was studied by calculating change in the resistance of sensing samples with time toward LPG exposure using two probe method. The gas sensing response of the materials was investigated with the help of LPG sensing set-up which includes sensing chamber, LPG cylinder, a Keithley electrometer, a gas flow meter, a vacuum pump.

Among many parameters in LPG sensing, four major parameters which determines the ability of an LPG sensors are response time, recovery time, reproducibility and sensitivity. These four major parameters place very important role to determine the efficiency of an LPG sensor. The response and recovery time in LPG sensing describes the time required by the resistance of the sensing material to attain 10% and 90% of its maximum value respectively. [41-42]

The change in the electrical resistance of the Pn and PnZnO composite as function of time at 1000ppm and 2000ppm was depicted in figure 5(a) & (b) respectively. Initially, the resistance of the samples was found to be stabilized due to the introducing air into the gas chamber using flow meter to establish the equilibrium between oxygen adsorbed at surface of the samples and atmospheric oxygen. When the resistance was found stabilized then the LPG of 1000ppm and 2000ppm concentration was injected in the gas chamber the resultant change in the electrical resistance was noted. The resistance of the Pn and PnZnO composite increases linearly with time when exposed to 1000ppm and 2000ppm LPG concentration and reaches to steady state.

In general, when the p-type materials come in contact with the LPG which reacts with pre-adsorbed O- ions and the captured electrons are released back. The increase in the resistance of the materials when exposed to LPG may due to the decrease in the number of previously generated holes. The decrease in the number of generated holes is responsible for the releasing back of the electrons into the conduction band and thereby to the valence band. [43-44]

It is observed from plot that, the samples required around 80 seconds to attained maximum resistance with composite at higher resistance compared to Pn. This increase in the resistance of the samples may due to the transfer of electrons from Pn to gas molecules.

To regain atmospheric condition for recovery, the LPG gas was made off and resistance of Pn decreases as a part of recovery becomes stable within few seconds. When LPG no longer remains in contact with the material, the recombination process stops and resistance starts decreasing.

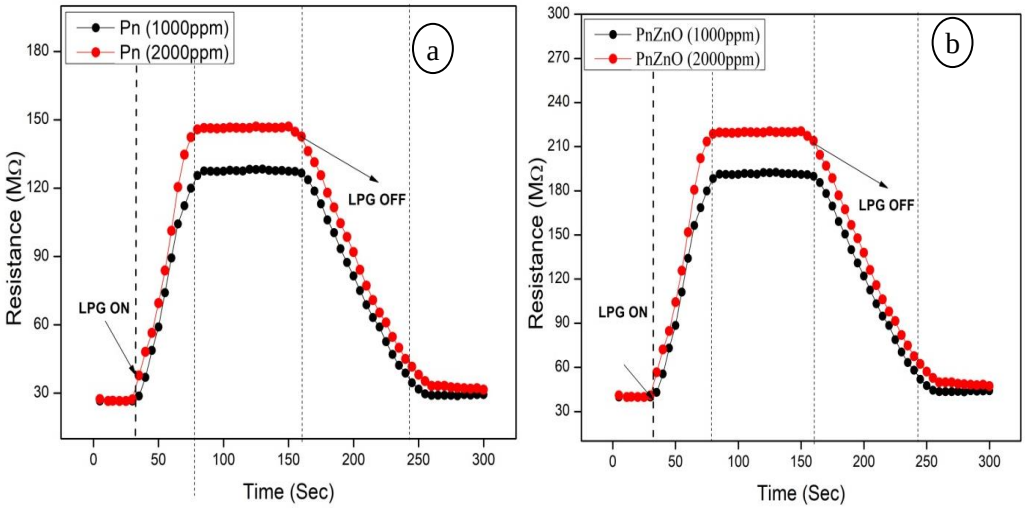


Figure-5: Variation of resistance as function of time a) for Pn, b) PnZnO composite at 1000

and 2000ppm

Sensitivity is the ratio of the resistance of the sensing material when the material is in contact with LPG (Rg) to the resistance of the sensing material when no LPG is present in the ambience of the material (Ra) [45].

The change in the electrical resistance of the Pn and PnZnO composite as function of time at 1000ppm and 2000ppm was depicted in figure 6(a) & (b) respectively. The sensitivity of the samples in terms of normalized resistance calculated by sensitivity = Ra/Rg and the Rg is the resistance of the sensor in presence of LPG gas and Ra is the initial stabilized resistance of the pallet. The composites show a higher sensitivity in comparison to Pn. Hence both these figures reveal that the response of Pn and all composite increases rapidly upon introduction of LPG gas and becomes stable within few seconds.

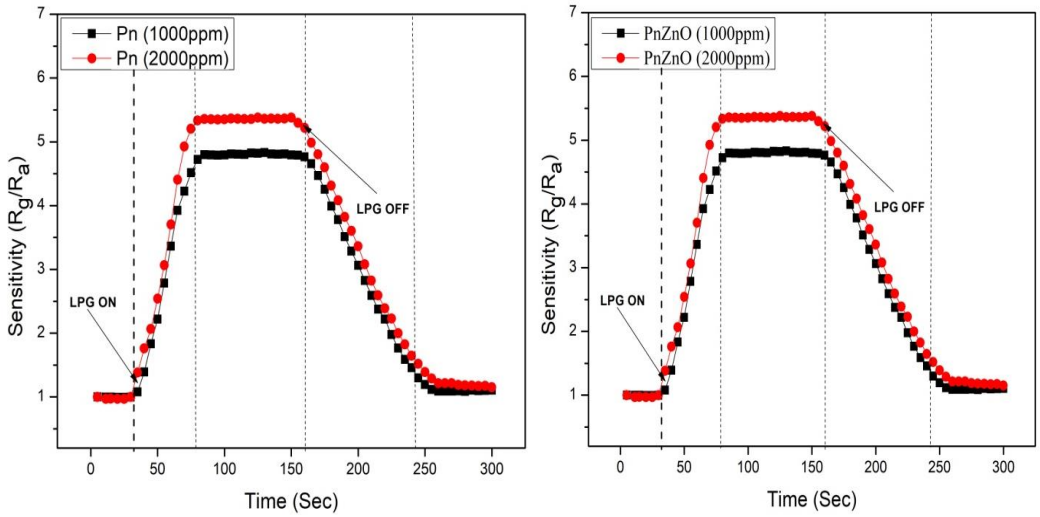


Figure-6: Sensitivity as function of time a) for Pn, b) PnZnO composite at 1000 and 2000ppm

4. Conclusion:

Pn and PnZnO composite was prepared by including ZnO particles into the PANI chain using COP method. Further the detail structural and morphological characterizations of the PANI & composite were investigated using XRD & SEM technique. XRD pattern of pure PANI describes the existence of broad peak at $2\theta \approx 25^0$ suggests the amorphous character of the prepared Pn and XRD pattern of PnZnO composite shows the presence of ZnO particles in the PANI with crystalline peaks of ZnO. The morphological investigation describes the uniform surface morphology with agglomeration of Pn semi-crystalline structure. the maximum AC conductivity value was obtained with a value of 3.19 x 10^{-2} S/m for 25 wt% ZnO at 1MHz frequency. the electrical conductivity (σ_{dc}) increased from 1.54x10^{-6} S/m for Pn at 303K to 6.19x10^{-4} S/m for PnZnO with 25 wt.% at 453K. The activation energy of the Pn found to be 0.489ev and decreasing with increase in zinc oxide content and reaches 0.178ev for 25wt%,

while the electrical conductivity increased with increasing doping rate. The LPG sensing property describes almost linear variation in the composites electrical resistance are greater stable and assist in holding the molecules of the vapor and this to be a proficient material for sensing LPG. The composite shows greater sensitivity at 1000ppm and 2000ppm compared to the Pn. Therefore, the composite can be a promising material for LPG sensing applications.

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