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

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Abstract:
The in-situ polymerization process is used to prepare the composites of polyaniline (Pn) and magnesium oxide doped polyaniline (PnMgO), with ammonium persulphate acting as an oxidizing agent. Magnesium oxide has been added in different amounts to prepare a variety of composites. All of the synthesized samples' phases and morphologies were examined utilizing fundamental characterization methods like scanning electron microscopy (SEM) and X-ray diffraction (XRD). Using the impedance approach, the samples' AC electrical conductivity was determined at room temperature throughout a frequency range of 10 KHz to 1MHz. Furthermore, the composites dc electrical transport property was examined throughout a 30-200^{0}C temperature range. The dc electrical conductivity of the PnMgO composites increased as the temperature and MgO concentration in polyaniline increased. For every composition, the activation energies were calculated using Arrhenius plots. As the concentration of MgO in the polyaniline matrix increased, the activation energy decreased. When the amount of MgO was increased, the electrical conductivity improved, but the activation energy of the obtained PnMgO composites dropped when compared to the activation energy of a Pn. When Pn and PnMgO composites were subjected to 1000 ppm and 2000 ppm concentrations of LPG gas, the electrical resistance of each material changed. When exposed to LPG gas, both samples showed a quick change in resistance, although the PnMgO composite was more sensitive and appropriate for LPG sensing than the Pn sample.

Keywords: Polyaniline (Pn), Magnesium oxide (MgO), LPG sensing, AC, DC, Activation energy, XRD, SEM

1. Introduction:

Due to their unusual properties of conducting polymers like poly-pyrrole (PPy), polyaniline (PANI), and polythiopene (PTh) due to their high electrical conductivity, intriguing electrochemical properties, and ease of processing, polymer nanocomposites have emerged as one of the materials that have been studied the most in recent years [1-2].

The organic-inorganic materials have been many advances in the production corresponding to both their chemical and physical properties [3-6]. 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 study 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 [7-9]. 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 [10-11]

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 [12-13]. 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 [14]. Compared to sensors made of organic compounds, metal oxide is a more stable active material for chemical sensors. Metal oxide materials such as ZnO and MgO have the ability to detect a variety of gases, such as CO, NO2, NH3, H2S, H2, and LPG [15]. Due to its numerous uses as an active catalyst [16-17], sensor [18-19], anti-reflective coatings [20], superconductors [21], adsorbents [22], batteries [23], and other applications, magnesium oxide (MgO) stands out among other metal oxides.

Here, we report the results of Pn and PnMgO 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 PnMgO composites based LPG sensor with 1000 and 2000ppm and MgO. The PnMgO composites with different wt 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 magnesium oxide (MgO) was used to prepare composites via chemical oxidative polymerization method. Pellets of Pn and PnMgO 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 vaccum 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 PnMgO composite:

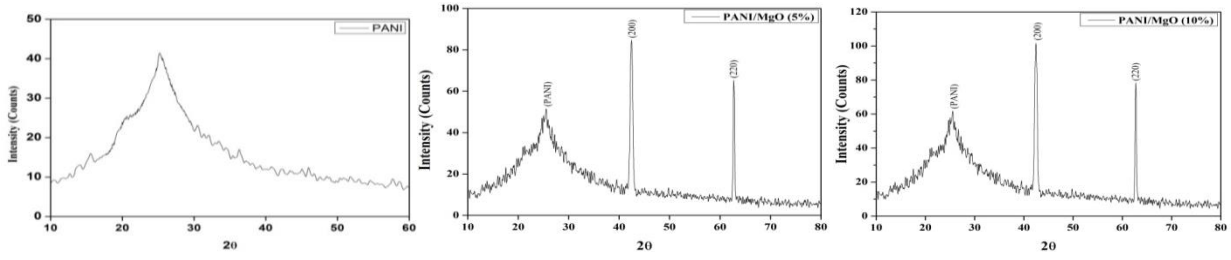
Magnesium oxide (MgO) powder of 5%, 10%, 15%, 20% and 25% additive weight percentage is dissolved in the mass fraction after adding the oxidizing agent. After adding the MgO, this reaction mixer was continuously stirred in magnetic stirrer for 8 hrs in room temperature. The dark green solution was obtained 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 [24]. The flow chart of step by step synthesis procedure is shown in flow chart.

3. Results and discussion:

3.1 ***XRD spectra:***

The XRD spectra for the pure Pn and PnMgO 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 [25]. This broad peak shows that Pn's amorphous structure contains crystalline regions.

The XRD spectra of PnMgO composite exhibits the diffraction peaks belongs to both MgO and Pn which describes the withholding of Pn in the composite material. The XRD spectra of prepared PnMgO composite exhibits well defined diffraction peaks obtained at different 2θ angles 42.44^0 and 62.72^0 corresponding to the hkl planes (200) and (220) for the phase of the magnesium oxide and which are well matched with the reported literatures and standard JCPDS data card No. 87-0653 [26]. The XRD spectra of composite also exhibit diffraction peak at 25^0 corresponding to PANI. No characteristic peaks other than MgO were observed. The average crystalline size of PN was calculated by using Scherrer's formula $D = K\lambda / \beta Cos\theta$ (where $\lambda = 1.54060$ 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 PnMgO composite was found to be 92 nm. Here the sharp diffractions peak of all composites indicates strong intensity of crystalline structure of the composites[27].



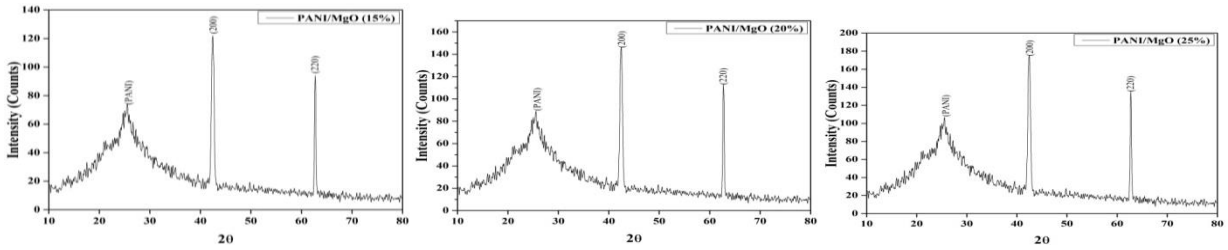


Figure-1: XRD spectra of Pn and PnMgO composites

3.2 SEM micrographs:

The SEM micrographs for the pure Pn and PnMgO 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 PnMgO 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 magnesium oxide particles distributed in the polyaniline matrix. [28-29].

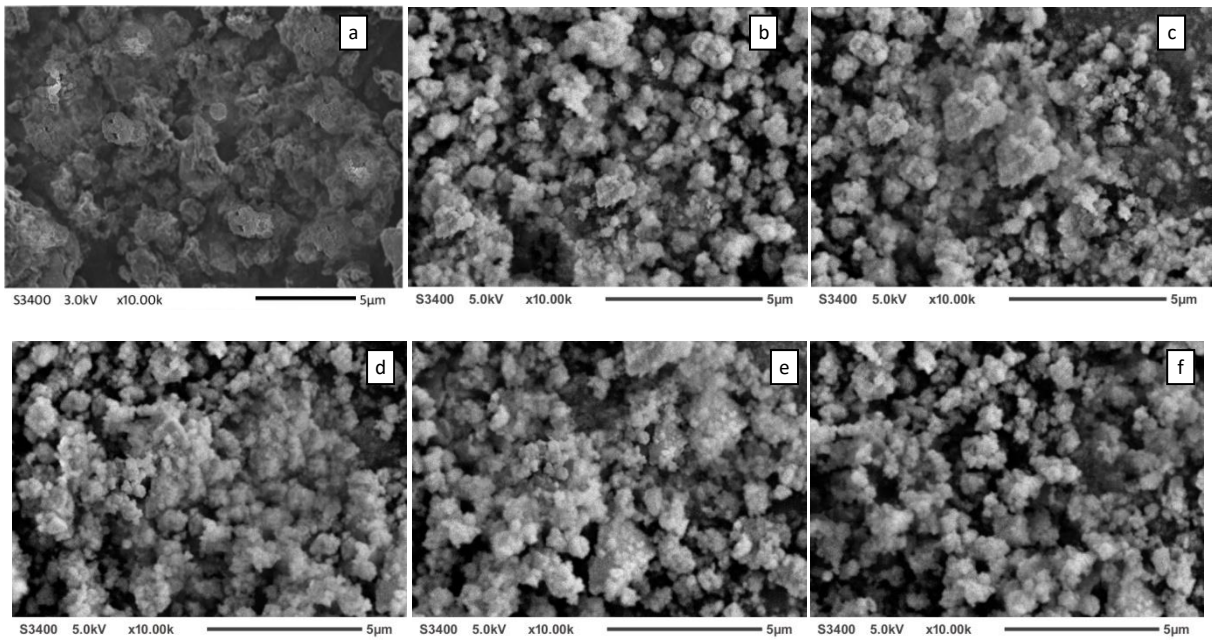


Figure-2: SEM micrographs of Pn and PnMgO composites

3.3 AC electrical conductivity:

The ac conductivity measurements have been conducted by a typical two probe method. The frequency dependent ac electrical conductivity of Pn and PnMgO composite was depicted in fugure-3. The conductivity of both samples increases with increase in frequency. An enhancement of one order of conductivity of PnMgO composite compared to the conductivity of Pn was observed. Figure 3(b) indicates the ac conductivity as function of different wt% of MgO. The conductivity of the composite increases with increase in the content of magnesium oxide in the Pn matrix. This increase in the conductivity of PnMgO composite may due to the even distribution of magnesium 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 [30-31].

Among all the prepared composite, the maximum AC conductivity value was obtained with a value of 3.01 x 10^{-2} S/m for 25 wt% MgO at 1MHz frequency also depicted in figure 3(b). The conductivity of the composite material found increasing from 1.2 x 10^{-2} S/m for 5wt% at 1MHz with increase in the content of magnesium oxide and reaching 3.01 x 10^{-2} S/m for 25 wt% MgO at 1MHz.

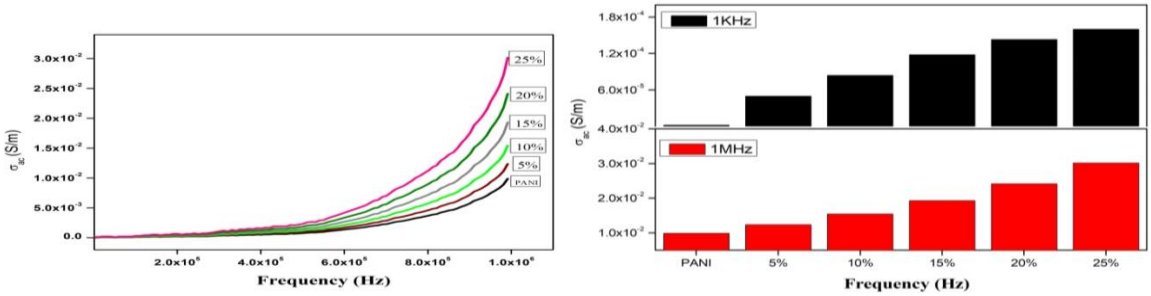


Figure-3: AC electrical conductivity of of Pn and PnMgO composite a) as function of frequency b) as function of percentage of MgO

3.4 DC electrical conductivity

The variation in the dc conductivity with change in temperature of Pn and PnMgO 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 MgO in the Pn matrix. This indicates the MgO particles gives positive influence on composite towards increase in conductivity. Figure 4(b) indicates the dc conductivity as function of different wt% of MgO. The result shows that the MgO 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 excitation of electrons to the conduction band at higher temperature [32-34].

Among all the prepared composite, the maximum DC conductivity value was obtained with a value of 1.8 x 10^{-3} S/m for 25 wt% MgO at 180^0 temperature also depicted in figure 4(b). The conductivity of the composite material found increasing from 3.5 x 10^{-4} S/m for 5wt% with increase in the content of magnesium oxide and reaching 1.8 x 10^{-3} S/m for 25 wt% MgO at 180^0 temperature. Compared to PANI and PANI/ MgO the conductivity of PANI/WO3 composites increased. This increase in the dc conductivity may due to the presence of MgO in Polyaniline[35].

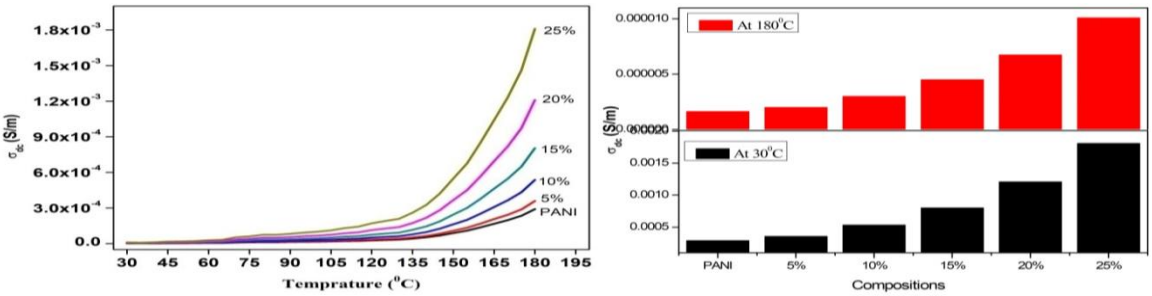


Figure-4: DC electrical conductivity of of Pn and PnMgO composite a) as function of temperature b) as function of percentage of MgO

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]:

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

where Ea is the activation energy (eV); T is the absolute temperature in (K); k_B is the Boltzmann constant, which is $1.38\text{x}10^{-23}$ (J/K) or $8.62\text{x}10^{-5}$ (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 MgO was depicted in figure 5(b). The activation energy found to be 0.427ev for Pn and which decreases to 0.379 ev for 25wt%. As shown in the results, the electrical conductivity (σ_{dc}) increased from $1.62\text{x}10^{-6}$ S/m for Pn at 303K to $1.8 \text{ x } 10^{-3}$ S/m for PnMgO with 25 wt.% at 453K.

This means that the electrical conductivity (σ_{dc}) increased by two orders of magnitude PnMgO 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.

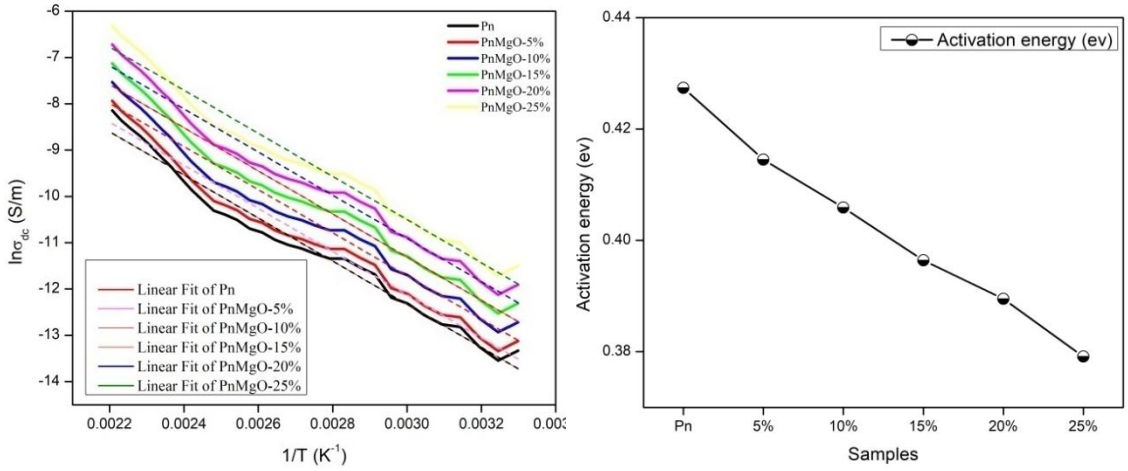


Figure-5: a) Arrhenius plot b) Activation energy

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

Sample	DC conductivity (S/m) at 303.15 K	DC conductivity (S/m) at 453 K	Activation energy in ev
Pn	$1.63\text{x}10^{-6}$	$2.91\text{x}10^{-4}$	0.427
PnMgO-5%	$2.00\text{x}10^{-6}$	$3.58\text{x}10^{-4}$	0.414
PnMgO-10%	$3.00\text{x}10^{-6}$	$5.36\text{x}10^{-4}$	0.405
PnMgO-15%	$4.50\text{x}10^{-6}$	$8.04\text{x}10^{-4}$	0.396
PnMgO-20%	$6.75\text{x}10^{-6}$	$1.21\text{x}10^{-3}$	0.389
PnMgO-25%	$1.01\text{x}10^{-5}$	$1.81\text{x}10^{-3}$	0.379

3.5 LPG sensing studies:

In order to test the gas sensing ability of composite of Pn and PnMgO-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 as function of time at 1000ppm and 2000ppm was depicted in figure 5(a-b) & 6 (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 PnMgO 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 Pn and PnMgO required around 65 seconds at 1000ppm and 75 seconds at 2000ppm to attained maximum resistance with composite at higher resistance. 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.

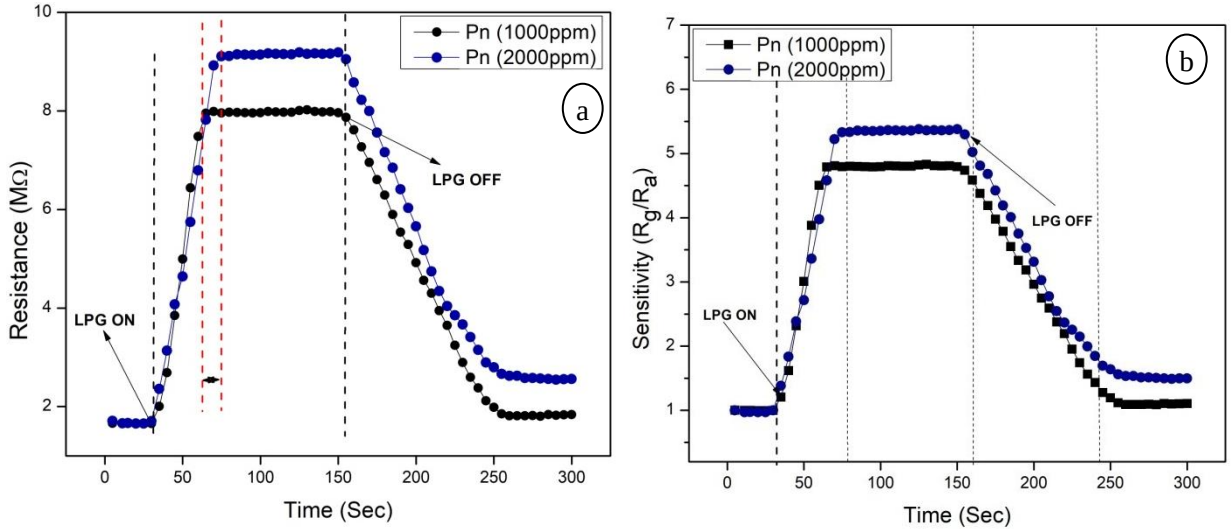


Figure-5: Variation of resistance as function of time a) for Pn, b) PnMgO composite at 1000 and 2000ppm

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 composites show a higher and faster response in comparison to pure PANI sensor [46]. 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.

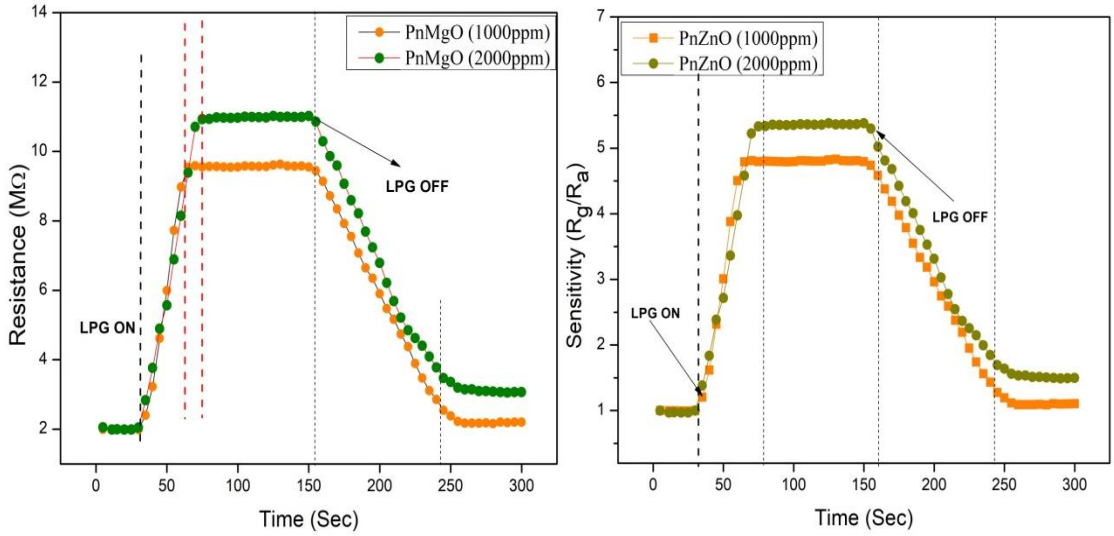


Figure-6: Sensitivity as function of time a) for Pn, b) PnMgO composite at 1000 and 2000ppm

4. Conclusion:

Pn and PnMgO composite was prepared by incorporating MgO particles into the Pn chain using the COP technique. Moreover, the Pn and composite were examined in detail for their morphological and structural characteristics using the XRD and SEM techniques. The large peak at 2θ≈250 described by the pure Pn XRD pattern reveals the amorphous nature of the produced Pn, while the PnMgO composite's XRD pattern demonstrates the presence of MgO particles in the Pn with crystalline MgO peaks. The morphological analysis explains the aggregation of Pn semi-crystalline structure and homogenous surface morphology. The maximum ac conductivity value 3.01 x 10^{-2} S/m measured at 1MHz frequency for 25 weight percent of MgO. The dc conductivity of the composite material was seen to improve with increasing magnesium oxide content, starting at 3.5 x 10^{-4} S/m for 5wt% and reaching 1.8 x 10^{-3} S/m for 25 wt% MgO at 1800C temperature. The activation energy of Pn was determined to be 0.427 eV, and it decreased as the magnesium oxide content increased, reaching 0.379 eV at 25 weight percent. In contrast, the electrical conductivity increased as the doping rate increased. The composite's electrical resistance exhibits an essentially linear variation, which helps to stabilize and retain the vapor molecules, making it an effective material for LPG sensing. In comparison to the Pn, the composite exhibits higher sensitivity at 1000 ppm and 2000 ppm. As a result, the composite material exhibits potential for use in LPG sensing applications.

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