Nanotechnology Perceptions
ISSN 1660-6795
www.nano-ntp.com

The Study Investigates the Preparation, Structural, Electrical and LPG Sensing Properties of Composite Material Comprising Nickel Oxide -Doped Polyaniline

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Objectives: To investigate the electrical and LPG sensing characteristics nickel oxide doped polyaniline (PnNiO) composite. Methods: The composite of polyaniline (Pn) doped with nickel oxide [PnNiO] was synthesized using a chemical oxidative polymerization technique. The phase and morphology of all the prepared samples were examined using basic characterization techniques such as X-ray diffraction (XRD) and scanning electron microscopy (SEM).The electrical properties are examined through conductivity measurements, while the LPG sensing properties are evaluated using various concentrations of LPG gas and monitoring the material's response. Findings: The study reveals the successful synthesis of the composite material comprising nickel oxide-doped polyaniline. Structural analysis confirms the incorporation of nickel oxide into the polyaniline matrix, leading to alterations in its crystalline structure and surface morphology. Electrical conductivity measurements demonstrate enhanced conductivity compared to pure polyaniline. Furthermore, the composite material exhibits promising LPG sensing properties, showing a significant response to varying concentrations of LPG gas. Novelty: the comprehensive investigation of a composite material composed of nickel oxide-doped polyaniline for its structural, electrical, and LPG sensing properties. The incorporation of nickel oxide into the polyaniline matrix and its subsequent effects on the material's properties contribute to its novelty. Additionally, the study provides insights into the potential application of the composite material as a sensitive and selective LPG sensor, which could have implications in various industries requiring gas sensing technology.

Keywords: Polyaniline (Pn), Nickel oxide (NiO), LPG sensing, AC, DC, XRD, SEM.

1. Introduction

Among the various conducting polymers, polyaniline [Pn] is focused as one of the potential and interesting material due to its ease in preparation, exciting electrochemical, optical property. Also the polyaniline is very potential material towards gas sensing uses due to its well-regulated electrically, environmentally and redox properties. Interestingly, the Pn offers various active sites on its backbone for the adsorptions and desorption of gas analyte. The many application of the Pn is lowered due to its poor solubility in organic solvents and also the Pn is not much sensitive like metal oxides. Since, conducting polymers shows new novel properties when the conducting polymers combined with the metal oxides and which enables the further account of polymer structure. Therefore, the synthesis of Pn composites with metal oxide increased the interest of researcher towards many applications [1-2]. The demand of high-quality resources for electromagnetic compatibility is alarmingly rising [3]. 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 [4-6]. The investigation of various materials has been greatly influenced by nanostructured metal oxides like TiO2, ZnO, NiO, CuO, graphene, and functionalized graphene. Because of its exceptional electrical, catalytic, and magnetic properties, nickel oxide (NiO) has drawn the most attention in gas sensing applications [7]. Among the various conducting polymers, polyaniline [Pn] is considered as one of the potential and interesting material due to its ease in preparation, exciting electrochemical, optical property. Also the polyaniline is very potential material towards gas sensing uses because of its stable environment and regulated electrical conductivity and redox properties [8]. It is also regarded as a promising material for gas sensor design due to its affordability, high sensitivity, quick response, and recovery speed [9]. In the current effort, we report on PnNiO was synthesized using a chemical oxidative polymerization technique. The PANI delivers more dynamic surface area for the gas sensing reaction, and on the other hand, NiO nanoparticles nucleated over polymer chains contribute to enhanced conductivity and stability of the nanocomposite material by interlinking the Pn polymer chains. The phase and morphology of all the samples were analysed using basic characterization techniques such as X-ray diffraction (XRD) and scanning electron microscopy (SEM). The AC 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 30-200^{0}C. It was observed that the incorporation of nickel oxide into the polyaniline matrix had an impact on the ac electrical properties of the samples. The variation of the electrically resistivity of both PANI and PnNiO composites was measured when exposed to 1000 ppm concentration of LPG gas. Both samples exhibited a

rapid resistance change upon exposure to LPG gas, with the PnNiO composite demonstrating higher sensitivity and suitability for LPG sensing compared to Pn. Here, we report the results of Pn and PnNiO 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 PnNiO composites based LPG sensor with 100ppm and NiO 50wt%. The PnNiO composites with different wt percentage were synthesized by in-situ technique and sensing characteristics of the synthesized composite to LPG were systematically investigated.

2. Methodology

The chemical substances used in the production 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 results were preparing by double-distilled water. The nickel oxide (NiO) was used to prepare composites via chemical oxidative polymerization method. Pellets of Pn and PnNiO 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 synthesize of polyaniline from 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 normal temp. The mixer of aniline and hydrochloric acid was stirring by magnetic stirrer for 2 hrs at constant RPM for reaction process. 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.

2.2 Preparation of PnNiO composite:

Following the same procedure of pure Pani Synthesis here in additional different weight percentage of metal oxides of (Nickel oxide (NiO) powder) 10%, 20%, 30%, 40% and 50% is dissolved in the mass fraction after adding the oxidizing agent. This solution was stirred for 6 hour at normal finally the dark green solution was obtained after the end of the reaction and it is kept for overnight to sort out the particles at the base of the beaker. The precipitate obtained and separate out by filtering and cleaned with deionised water with acetone. The obtained final suspension was dried in oven at 50° C for 24 hrs. The finally crushed into powder form [10].

3. Results and Discussion

3.1 XRD spectra:

The crystallinity and chain packing of the synthesized polymer nanocomposites were examined by X-ray difraction analysis. It is reported in the literature that plain PANI shows partially crystalline nature and also its characteristic peak at $2\theta = 25^0$[11]. The XRD spectra for the pure Pn and PnNiO composite samples were recorded in the range of 10^0 to 85^0 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 [12]. This broad peak shows that Pn's amorphous structure contains crystalline regions.

The XRD spectra of PnNiO composite exhibits the diffraction peaks belongs to both NiO and Pn which describes the withholding of Pn in the composite material. The XRD spectra of prepared PnNiO composite exhibits well defined diffraction peaks obtained at different 2θ angles 37.42^0, 43.3^0, 62.34^0, 75.4^0 and 79.38^0 corresponding to the hkl planes (111), (200), (220), (311) and (222) for the phase of the nickel oxide and which are well matched with the reported literatures and standard JCPDS data card No. 73-1523 [13-14]. Previously it is reported a (Arunkumar Lagashetty @el) defining the peaks of the NiO and Pn. The continuation to the previous work reported it is illustrated the determination of the average crystallite size of composite especially for all the composites of weight percentage 5, 10, 15, 20 and 25. The average crystallite size of Pn was determined 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 PnNiO composite was found to be 20 nm.

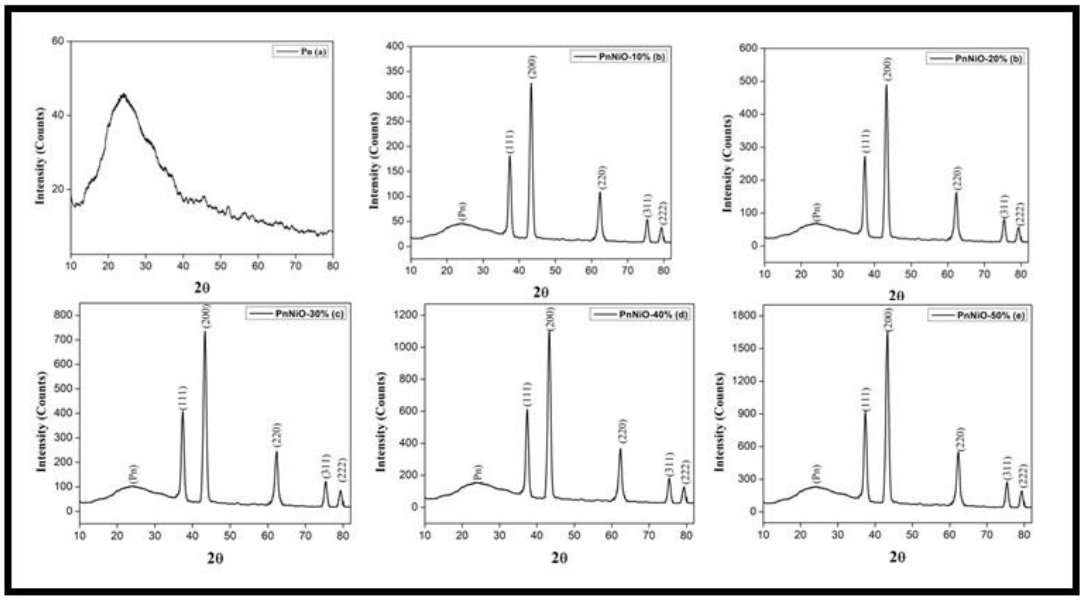

Figure-1: XRD spectra of Pn and PnNiO composites

3.2 SEM micrographs:

The SEM micrographs for the pure Pn and PnNiO 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 PnNiO 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 nickel oxide particles distributed in the polyaniline matrix [15-16]. It can be concluded that the gradual increase in granular size and change in morphology, which enables the transportation of charged particles through the carbon back bone of the polymer chains [17]. Previously reported (Omolola E. Fayemi at.al.) with 1µm pixel with 10K magnification. The average diameter of the particles were measured using imageJ software with considering the image known size of 5µm.

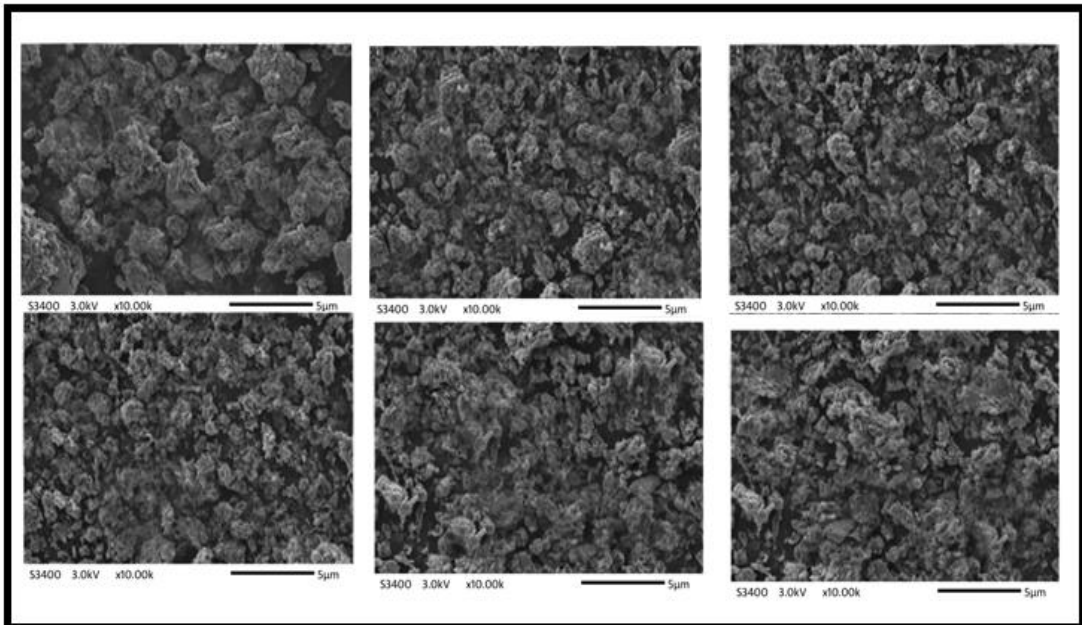

Figure-2: SEM micrographs of Pn and PnNiO composites

3.3 AC electrical conductivity:

In case of the ungroomed substances like polymers, the frequency dependent electrical conductivity of such materials is mainly arises due to the interfacial polarization at contacts, grain boundaries and existence of heterogeneous in the sample [18-19]. The unusual conductivity of the conductive polymers contain a π-conjugated system accountable for their rare electrical properties. The hopping mechanism is usually dominated in the polymers conductivity in which the mobile charge carriers, polarons and bipolarons, are transported along the conjugated chain. Further when the polymer is doped then the charge carriers are introduced into π-conjugated system [20]. With the help of two probe method we measure the AC

conductivity the frequency dependent ac electrical conductivity of Pn and PnNiO composite was depicted in fugure-3. The conductivity increases with increase in frequency. An decrement of one order of conductivity of PnNiO composite compared to the conductivity of Pn was observed. Figure 3(b) indicates the ac conductivity as function of different wt% of NiO. The conductivity of the composite decreases with increase in the content of nickel oxide in the Pn matrix [21-22]. The contrasting in the image is due to the difference in spreading from variety surfaces area as an effect of geometrically difference b/w polyaniline and dispersed oxide sample[23]. The AC conductivity values obtained here for the NiO-PANI composites is the highest when compared to similar (B manjunatha et..al. 2022, DOI: 10.53555/ecb/2022.11.10.49) previously reported works.

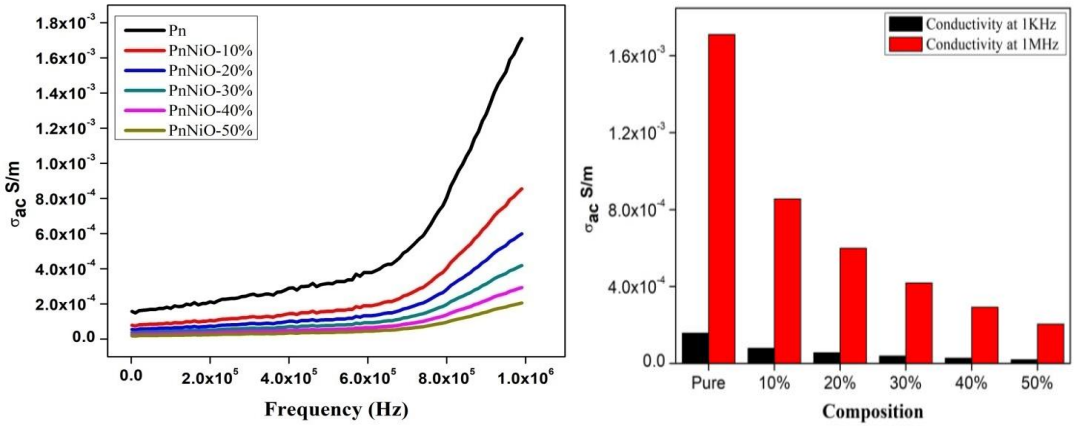

Figure-3: AC electrical conductivity of Pn and PnNiO composite a) as function of frequency b) as function of percentage of NiO

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 PnNiO 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 decreases with increase in content of NiO in the Pn matrix. This indicates the NiO particles gives negative influence on composite towards decrease in conductivity. Figure 4(b) indicates the dc conductivity as function of different wt% of NiO. The result shows that the NiO has negative 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 [24-28]. B manjunatha et..al. 2022, DOI: 10.53555/ecb/2022.11.10.49) previously reported works.

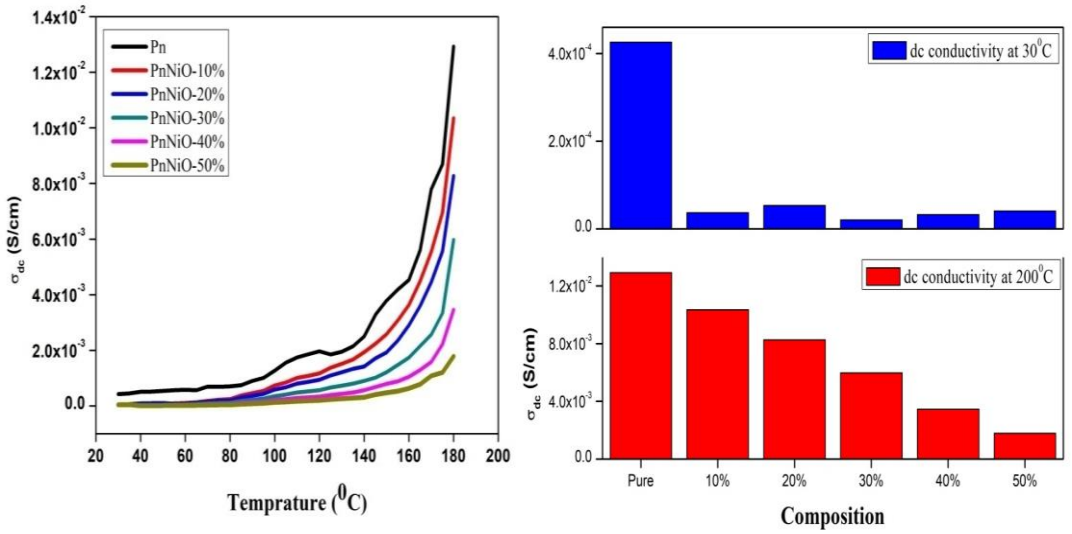

Figure-4: DC electrical conductivity of Pn and PnNiO composite a) as function of frequency b) as function of percentage of NiO

3.5 LPG sensing studies:

The gas sensing behavior of Pn and PnNiO-50% composite upon interaction with LPG at 1000ppm 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 change in the electrical resistance and sensitivity of the samples as function of time at 1000ppm was depicted in figure 5(a) & 5(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. The stabilized resistance at this state is known as resistance in the presence of air (Ra). When the resistance was found stabilized then the LPG of 1000ppm concentration was injected in the gas chamber the resultant change in the electrical resistance was noted. The resistance of the Pn and PnNiO composite increases linearly with time when exposed to 1000ppm LPG concentration and reaches to steady state. It is observed from plot that, the samples required 230 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. A weak charge transfer between the polymer matrix and the analyte gas is the reason why electrons are removed by the gas in Pn [29]. This change in resistance is attributed to the fact that when LPG interacts with polyaniline, the mobility decreases and resistance across the sample increases [30]. The change in the resistance is known as resistance in the presence of LPG (Rg). The higher change in the resistance of the composite was observed compared to Pn. The change in sensitivity of the Pn and PnNiO composite as function of time is shown in figure-5(b). 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 Pani and those figures reveals that the response of Pn and all composites increase quickly upon overview of LPG gas and develop steady within few

seconds [31]. The response time values found less than 150 seconds here for the NiO-PANI composites is the less when compared to similar previously reported works (Pallavi T. Patil et.al 2015). Here the sensitivity of the sensor found to be higher value with 1000ppm concentration of LPG. Also few literature (S. L. Patil et .al., 2011)found with 100ppm for NH_3 sensing

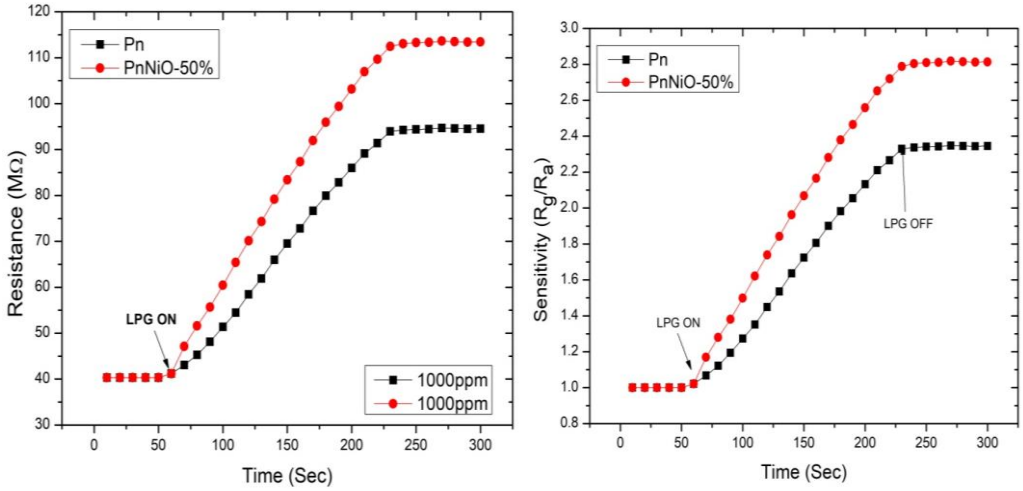

Figure-5: a) Variation of resistance as function of time b) sensitivity as function of time

4. Conclusion:

Pn and PnNiO composite was prepared by including NiO 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. The XRD spectra of PnNiO composite exhibits the diffraction peaks belongs to both NiO and Pn which describes the withholding of Pn in the composite material. The homogenous mixer of the NiO and Pn was confirmed by the SEM, in which the NiO particles were incorporated in PANI matrix. The multiple phases of the nanocomposite material enhance the overall performance of the sensing behavior of the material. The morphological investigation describes the uniform surface morphology with agglomeration of Pn semi-crystalline structure. The electrical properties such as ac conductivity enhances with increase in frequency and increases as NiO doped in the matrix of the Pn. The maximum AC conductivity value was obtained with a value of 8.55 x 10^{-4} S/m for 10 wt% NiO at 1MHz frequency. Among all the prepared samples, the maximum DC conductivity value was obtained with a value of 1.292 x 10^{-2} S/cm for Pn at 180^0C. 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 compared to the Pn. Therefore, the composite can be a promising material for LPG sensing applications. It is observed from plot that, the samples required 170 seconds to attained maximum resistance with composite at higher resistance compared to Pn. While comparing the sensing behaviour for LPG gas, PnNiO composite composite shows fast response and recovery as compared to

that of pure Pn.

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