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LPG Sensing Properties Study of Cobalt Oxide and Nickel Oxide Doped Polyaniline Composite

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Abstract

Background/Objectives: To synthesise and evaluate NiO- and Co_3O_4-doped polyaniline composites for enhanced LPG gas sensing performance using chemical oxidative polymerization techniques.

Methods/Statistical analysis: By means of chemical oxidative polymerization, NiO and CoO_4 nanoparticles were integrated into the polyaniline matrix. Using XRD and SEM, structural and morphological studies have been carried out. To evaluate the gas detection capabilities, electrical resistance changes were measured upon exposure to 1000 ppm LPG. Pure Pn was used as the control for comparative analysis of response time and sensitivity.

Findings: The successful creation of NiO- and CoO_4-doped polyaniline nanocomposites with uniform distribution was validated by XRD and SEM data. Both composites showed significantly improved LPG sensing performance over pure Pn. PnNiO exhibited the best response, reaching maximum resistance in ~230 seconds, indicating faster response and higher sensitivity. These improvements are attributed to enhanced charge transport and the synergistic effect of metal oxide doping. Compared to existing reports, these results provide novel insights into the role of multi-phase polymer–metal oxide interactions in gas sensing. This study contributes to the development of highly responsive and selective LPG sensors based on conductive polymer composites.

Novelty/Applications: Enhanced LPG sensing using polyaniline doped with metal oxides offers improved sensitivity and response time for industrial gas leak detection systems.

Key words: Polyaniline (Pn), Nickel oxide (NiO), Cobalt oxide (Co_3O_4), LPG sensing, XRD, SEM

1. Introduction

Due to their outstanding potential for solid-state devices, polymeric materials have attracted more and more attention from researchers [1]. The identification and monitoring of harmful gases in the environment is extremely important for both environmental and human safety [2]. The increasing use of LPG has made it necessary to take precautions against potentially fatal accidental explosions by implementing quick and accurate leak detection. Previously documented semiconductor-based sensors, including the SnO2-ZnO-based NO2 gas sensor[3]. The last few years have seen the use of chemically deposited TiO2 thin films exposed to electron beam radiation [4], Fe2O3 thin films as NO2 sensors [5], nanocatalyst (Pt, Ag, and CuO) doped SnO2 thin film based sensors [6], CdO [7], and Ce doped NiO nanoparticles as selective NO2 gas sensors [8]. Recent research on LPG gas sensors based on NiO/PANI composites has documented gas sensing response at room temperature [9].

Additionally, use of a polythiophene/zinc oxide nanocomposite for liquefied petroleum gas sensing was documented. PTh/ZnO3 was discovered to have lower detection limit of 600 ppm. When compared to pure PTh-based sensor at 2400 ppm, the PTh/ZnO3-based sensor demonstrated sensing responsiveness and reversibility that were approximately 1.58 and 4.9 times higher, respectively. These days, the most researched materials are inorganic and conductive polymer nanocomposites. Composites made of inorganic materials and conductive polymers are currently one of the most researched material types. The most common technique for creating these composites is the summarisation of inorganic particles into essentially conducting polymers. By adding nanoparticles to conducting polymers, excellent selectivity and sensitivity can be produced. This also enhances the performance of the material even in the field of sensing materials and applications [10]. Nowadays many number of conducting polymers are present for various industrial applications, which include polythiophene, polyaniline, polyacetylene, polypyrrole and polyindole [11-12]. These conducting polymers have potential in various industrial applications which including medical and health monitoring devices, LEDs, various sensor materials and storage devices [13].

Polyaniline [Pn], one of the many conducting polymers, is regarded as a promising and intriguing material because of its optical, electrochemical, and ease of synthesis. Also, the polyaniline is very potential material towards gas sensing uses because of its stable environment and regulated electrical conductivity and redox properties [14]. Nanostructured metal oxides involving NiO, TiO2, ZnO, graphene, CuO, as well as functionalised graphene, have had a significant impact on the study of different materials. In gas sensing applications, nickel oxide (NiO) has garnered the greatest attention due to its remarkable electrical, catalytic, and magnetic capabilities. Because of its high sensitivity, low cost, fast reaction time, recovery speed, it is also seen to be a promising material for gas sensor design[15].

Here, we provide the outcomes of the in-situ approach of fabricating Pn, PnNiO, and Co3O4 composites. The in-situ method yields composites employing the simplest techniques as compared to other production techniques. As far as our review of the literature on LPG sensors is concerned, no reports of 100 ppm and 50 wt% PnNiO and Co3O4 composite-based LPG sensors have been found. The Co3O4 and PnNiO The in-situ method was used to create the composites with varying weight percentages, and the synthetic composite's sensing properties to LPG were thoroughly examined[16].

2. Materials and methods

Analytical-grade hydrochloric acid (HCl), ammonium persulfate (99%), and aniline (99%) were the chemical reagents utilised in the preparation process. Analytical reagent (AR) grade chemicals made up the remaining supplement compounds. Double-distilled water was used to prepare each solution.

- Composites were made utilising the chemical oxidative polymerisation process with nickel oxide (NiO). Pn and PnNiO composite pellets measuring 12.4 millimetres in diameter and 1.18 millimetres in thickness have been created using 5-tonne pressure.
- Composites were made utilising the chemical oxidative polymerisation process with cobalt oxide (Co3O4). Pn and Pn Co3O4 composite pellets measuring 12.4 millimetres in diameter and 1.18 millimetres in thickness have been made using 5-tonne pressure..

2.1 Preparation of Polyaniline (Pn)

The COP process was used to create polyaniline from aniline, using ammonium persulfate as an oxidising agent and HCL as a catalyst. Catalyst hydrochloric acid of one N was created in beaker-2 at room temperature and combined with 0.2M aniline that has been prepared in beaker-1. To complete the reaction, the aniline and hydrochloric acid mixture was agitated for two hours at a steady RPM using a magnetic stirrer. Ammonium persulfate, an oxidising agent, is used to oxidise monomers. Next, a 0.25M ammonium persulfate solution was made in a different beaker. Then, over an hour, while stirring constantly and keeping the temperature at roughly 50°C, prepared ammonium persulfate solution has been added drop by drop to previously made aniline hydrochloride solution. This prepared mixer was constantly swirled with a magnetic stirrer for eight hours at room temperature after ammonium persulfate solution has been added. After mixer was finished, a dark green

solution was produced, which was left overnight to separate the particles at the beaker's base. After the precipitate was created and filtered out using a vacuum pump, it was washed with acetone, deionised water, as well as 1N HCL to get rid of any remaining additives in the Pn. After that, the finished sample has been dried in an oven set to 50 degrees Celsius for 24 hours. The resulting final suspension was crushed into a powder.

2.2 Preparation of PnNiO composite:

NiO powder of 10wt%, 20wt%, 30wt%, 40wt% and 50wt% is suspended in the mass fraction after substitution of oxidising agent. This solution mixer was stirred using magnetic stirrer continuously for eight hours at room temperature. After the reaction was finished, a dark green solution was discovered, and it was left overnight to separate the particles at the beaker's base. Deionised water and acetone were used to filter and clean the last suspension. The final suspension that was produced has been further dried for 24 hours at 50 degrees Celsius in an oven. Finished item has been ground into a powder. Flow chart illustrates the sequential synthesis process.

2.3 Preparation of Pn Co_3O_4 composite:`

Cobalt oxide (Co_3O_4) powder of 10wt%, 20wt%, 30wt%, 40wt% and 50wt% is suspended in the mass fraction after substitution of oxidising agent. This solution mixer was stirred using magnetic stirrer continuously for eight hours at room temperature. After the reaction was finished, a dark green solution was discovered, and it was left overnight to separate the particles at the beaker's base. Deionised water and acetone were used to filter and clean the last suspension. The resulting final suspension has been further dried in an oven set to 50 degrees Celsius for 24 hours. The final product was reduced to a powder[17].

3. Results and discussion

3.1 XRD spectra:

Figure 1(a) shows XRD spectra of pure PnNiO and Pn composite materials, which have been recorded between 10^0 and 85^0. Scattering from polyaniline at interplanar spacing, that is, a feature of van der Waals distances among stacks of polyaniline rings, is responsible for the large peak in the prepared polyaniline (Pn) XRD spectra at 2θ angles around 25^0 [14–15]. This wide peak shows that there are crystalline areas in Pn's amorphous structure. Pn withholding in the composite material is described by the diffraction peaks of the PnNiO composite's XRD spectra, which are displayed in Figure 1(b) and pertain to both NiO and Pn. XRD spectra of the prepared PnNiO composite show distinct diffraction peaks at various 2θ angles (37.42^0, 43.3^0, 62.34^0, 75.4^0, and 79.38^0). These peaks correspond to the hkl planes (111), (200), (220), (311), (222) for the nickel oxide phase and are in good agreement with JCPDS data card No. 73-1523, 71-1179, and the published literature[18-19]. Additionally, the composite's XRD spectra show a diffraction peak at 25^0 that corresponds to PANI. It was discovered that the PnNiO composite's average crystallite size was 20 nm.

The Pn withholding in the composite material is described by the diffraction peaks of the Pn Co3O4 composite XRD spectra, which are displayed in Figure 1(c) and pertain to both Co3O4 and Pn. The prepared Pn Co3O4 composite's XRD spectra show distinct diffraction peaks at various 2θ angles (18.93^0, 31.04^0, 36.36^0, 45.02^0, 59.14^0, and 65.12^0), which correspond to the hkl planes (111), (220), (311), (400), (511), and (440) for the cobalt oxide phase. Both the published literature and the standard JCPDS data card No. 42-1467 [20–22] agree well with these peaks. Additionally, the composite's XRD spectra show a diffraction peak at 25^0 that corresponds to PANI. It was discovered that the Pn Co3O4 composite's average crystallite size was 23 nm.

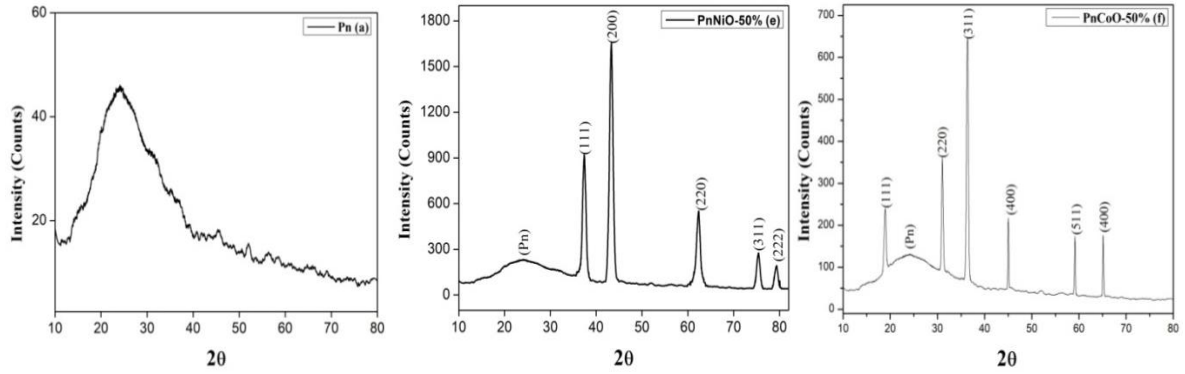

Figure 1: XRD spectra of Pn, PnNiO and Pn Co_3O_4composites

3.2 **SEM micrographs**:

Figure 2(a-c) shows SEM micrographs of pure Pn, PnNiO, Pn Co3O4 composite samples enlarged thousand times. The Pn's morphology seems to be non-fibrous, irregularly shaped, and highly dense. A range of agglomerations, a few randomly distributed oval-shaped particles, micro-sized spherical particles with surface homogeneity, and irregularly spread granules and flakes with sharp edges are all seen in the SEM micrographs of the PnNiO composite. It also seems non-porous. Morphology of Pn is irregular and non-fibrous with high density. SEM micrographs of Pn Co_3O_4 show irregularly arranged granular flakes with sharp edges. Pn Co_3O_4 appears non-porous. Morphology shows spherical with a few oval-like particles randomly distributed.

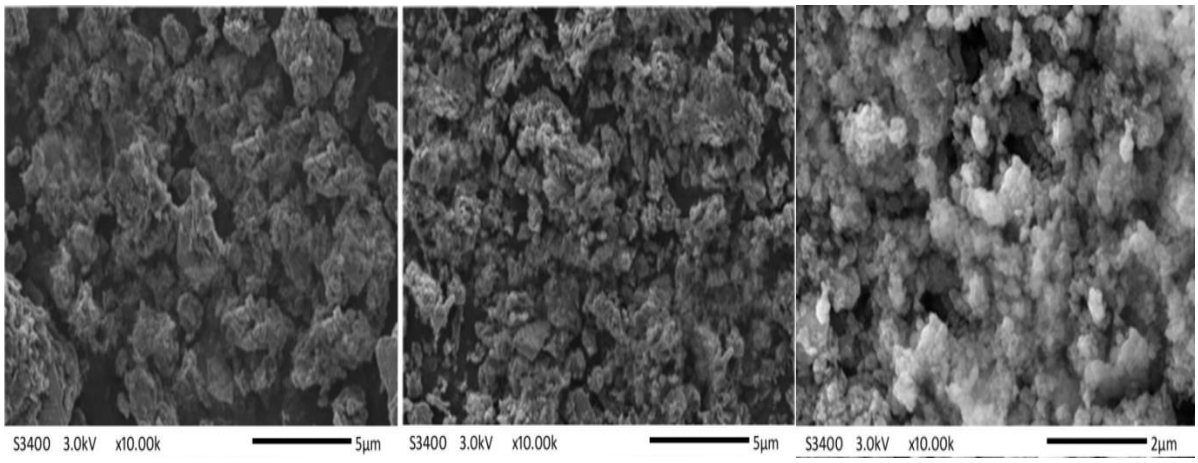

Figure 2: SEM micrographs of Pn, PnNiO and Pn Co_3O_4 composites

3.3 **LPG sensing studies:**

Provided LPG sensing response graphs illustrate the time-dependent sensitivity behavior of pristine polyaniline (Pn) and its composites doped with 50% nickel oxide (PnNiO-50%) and cobalt oxide (Pn Co_3O_4-50%). The ratio of resistance in presence of gas (Rg) to resistance in air (Ra), or Rg/Ra, is plotted against time in seconds to determine sensitivity. The graphs show that all materials initially maintain a baseline sensitivity value of ~1.0 until the point of LPG exposure, which is marked at approximately 50 seconds in both graphs. Upon introduction of LPG, all samples exhibit a gradual increase in sensitivity, indicating interaction with the target gas. Among the three samples, the PnNiO-50% composite demonstrates the highest sensitivity and fastest response, reaching a peak Rg/Ra ratio of approximately 2.85 around 200 seconds. In comparison, the Pn Co_3O_4-50% composite reaches a slightly lower maximum sensitivity of ~2.75 at about 180 seconds. Meanwhile, the pristine Pn lags in both magnitude and rate, attaining a maximum Rg/Ra of only ~2.3 around 240–250 seconds. These variations demonstrate how metal oxide doping improves polyaniline's gas sensing capabilities. Nickel oxide, a p-type semiconducting metal oxide, introduces active catalytic sites and facilitates oxygen adsorption

and charge transfer, leading to enhanced sensitivity and faster response dynamics [23-25]. Similarly, cobalt oxide also improves gas response through increased surface activity and formation of heterojunctions with the Pn matrix, although slightly less effectively than NiO [26-27]. When comparing performance quantitatively, PnNiO-50% shows a ~24% improvement in maximum sensitivity over pristine Pn, while Pn Co_3O_4-50% improves it by ~20%. This suggests that NiO-doped Pn is more effective in enhancing LPG sensing capabilities than its Co_3O_4-doped counterpart. Overall, these results affirm the synergistic interaction between polyaniline and transition metal oxides, which improves gas molecule adsorption, enhances electron mobility, and thus significantly boosts sensing performance. High-performance gas sensors for safety and environmental applications could be developed using such composites. This indicates that metal oxide doping greatly enhances gas-sensing properties of polyaniline composites, with NiO in this investigation being marginally more efficient than Co3O4.

The ratio of the sensing material's resistance when it comes into contact with LPG (Rg) to its resistance when there is no LPG present in the surrounding environment is known as sensitivity [28].

Significance

Nickel oxide acts as a p-type semiconducting metal oxide, which forms a heterojunction with polyaniline, enhancing charge transfer and increasing the material's interaction with LPG molecules. This leads to a marked increase in sensitivity and response speed, demonstrating the potential of NiO-doped Pn in advanced gas sensing systems [24]. Co_3O_4 contributes to higher oxygen vacancy density and promotes catalytic oxidation of LPG molecules. The interaction between Co_3O_4 and Pn generates synergistic effects, forming a p–p heterojunction that improves the charge carrier mobility and gas-sensing performance [26-27]. Sensitivities and selectivity in nanostructure PANI research with the addition of different metal oxides are not well explained by prior data [29].

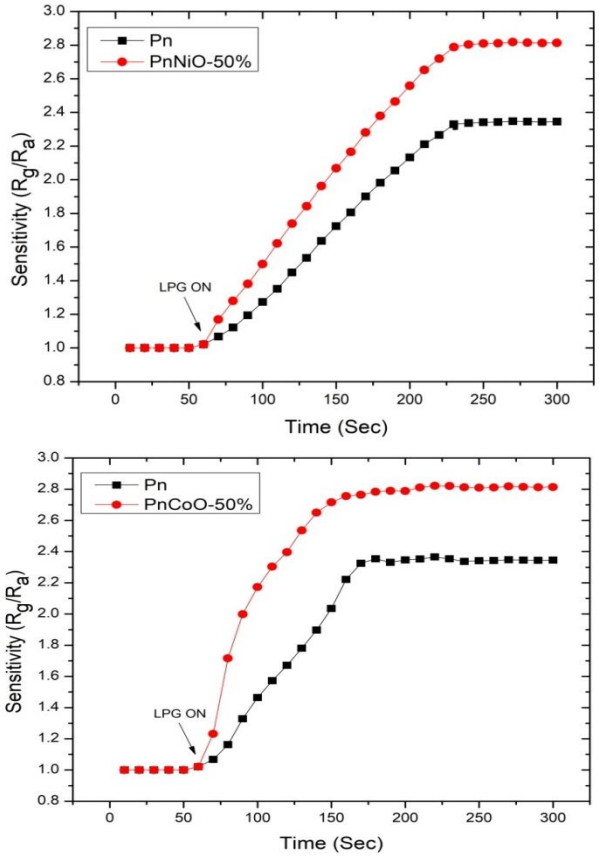

Figure 5: sensitivity as function of time of Pn, PnNiO and PnCoO composite

Material	Max Sensitivity (Rg/Ra)	Response Time (to Max)	Sensitivity Increase over Pn
Pn	~2.3	~240–250 sec	-
PnNiO-50%	~2.85	~200 sec	~24%
Pn Co_3O_4**-50%**	~2.75	~180 sec	~20%

Table shows sensitivity and response time.

4. Conclusion

COP (Chemical Oxidative Polymerisation) method has been successfully utilised to create Pn (polyaniline) or its composites with NiO and Co3O4 by adding NiO and Co3O4 particles to the PANI chain. XRD (X-ray diffraction) and SEM (scanning electron microscopy) have been used to extensively examine morphological as well as structural properties of synthesised materials. The PnNiO composite's XRD spectra showed diffraction peaks for both NiO and Pn, suggesting that Pn was retained in the composite. The presence of Co3O4 particles within the PANI matrix was also confirmed by the XRD pattern of the PnCoO composite, which showed crystalline peaks of CoO_4. For pure PANI, a broad peak at $2\theta \approx 25°$ indicated its amorphous nature. SEM analysis confirmed a homogeneous distribution of metal oxide particles within PANI matrix in both composites. NiO and Co_3O_4 particles were well embedded in the semi-crystalline structure of Pn, resulting in a uniform surface morphology with noticeable agglomeration.Both composites demonstrated enhanced LPG sensing behavior compared to pure Pn. The electrical resistance of the composites exhibited a nearly linear variation upon exposure to LPG, indicating stable sensing behavior. Effective adsorption and interaction with LPG molecules are made possible by the synergistic effect of several phases inside the nanocomposites, which is responsible for the improved performance. In contrast to pure Pn, the PnNiO composite demonstrated a faster reaction and recovery time, as well as increased sensitivity at 1000 ppm LPG concentration. It also required approximately 170 seconds to reach maximum resistance, reflecting its efficient sensing capability. These results imply that, because of their improved sensitivity, stability, and response behaviour, PnNiO and Pn Co3O4 composites are potentially viable options for LPG sensing applications.

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