Synthesis, characterization and LPG Sensing property of Polypyrrole Nanocomposites

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***Abstract*— In-situ polymerization procedures were used to create Polypyrrole /tin oxide nanocomposites. Scanning electron microscopy (SEM) and Transmission Electron Microscopy (TEM) were used to describe the nanocomposites, and thermal characteristics were investigated using a Differential Scanning Calorimeter (DSC). The samples' dc conductivity was determined as a function of temperature in the range 30–1900C, and it was discovered that increasing the concentration of Tin oxide particles enhances conductivity due to polaron hopping and composite chain length extension. Because half of the nanocomposites have the highest conductivity and sensitivity, the study suggests that these nanocomposites could be valuable in future applications.**

Keywords*— *Polypyrrole, Scanning Electron Microscopy, Conductivity, Sensitivity

I. INTRODUCTION (*HEADING 1*)

Polypyrrole (PPy) is interesting physicochemical properties such as electrical conductivity, deep black colour, ion-exchange capacity, hydrophobic nature, and strong adsorption capacity towards molecular and macromolecular species, polypyrrole (PPy) is one of the most investigated intrinsically conductive polymers (ICPs). Thin films, powders, colloidal particles, hollow particles, nanotubes, micrometer-sized composites, and nanocomposites are all possible forms of polypyrrole [1, 2]. Due to their unusual mix of electrical and optical properties and processing advantages, Alan J. Heeger and Hideki Shirakawa conducted extensive research on the new generation of "synthetic metals" in 1976. The electrical conductivity of conjugated polymers is achieved through the delocalization of the -electrons, which allows charge mobility along the polymer chain's backbone. Conducting polymers have been synthesized using an oxidising or reducing method, as well as chemical or electrochemical doping [3, 4].

The development of polymer/inorganic hybrid nanocomposites is attracting a lot of attention these days. Because of their intriguing optical and electrical properties, transparent conducting oxides (TCO) such as ITO, SnO_2, CdO, TiO_2, ZnO, $ZnSnO_4$, NiO, and others have been extensively researched. Zinc oxide (ZnO) and titanium dioxide (TiO_2) are the most appealing transparent conducting materials because of their non-toxicity, low cost, chemical stability, and ability to dope with a wide range of ions [5]. Various applications of conducting polymers have been proposed as transducers of biosensor, electrodes of rechargeable batteries, artificial nerves and muscles, gas sensors, solid electrolytic capacitor, diodes and transistors, anti-static electromagnetic shielding etc.

Tin oxide is a promising functional material because to features such great visual transparency, strong physical and chemical interactions with adsorbed species, and low operating temperature [6]. Tin oxide (SnO_2) is an n-type wide band semiconductor with an Eg of 3.6 eV at 300 K that is widely employed in optoelectronic devices, transparent conductive electrodes, solar cells, catalyst support, and gas sensing material [7]. The enormous surface to volume ratio of nanoscale materials causes them to behave differently than their bulk counterparts, as is widely known. Mechanical, thermal, chemical, electrical, and optical properties of nanoparticles are superior [8]. Tin oxide nanoparticles are commonly utilized in gas sensors as sensing elements. SnO_2 nanoparticles are synthesized by various techniques such as sol– gel, hydrothermal, co-precipitation, mechano-chemical, combustion route, spray pyrolysis, electrochemical deposition laser ablation, micro-emulsions technique etc. [9, 10]. Among all the above mention techniques the sol gel method is most suitable for the synthesis of nano-material due to its comparatively low processing cost and better control over the particle size of the crystallite.

II. EXPRIMENTAL

A. *Synthesis of Tin oxide nanoparticles*

Analytical Reagent grades of $SnCl_4.5H_2O$ and ethanol were successfully used to make tin oxide nanoparticles. To begin, a transparent sol solution was made by dissolving 3.50 gm. of tin chloride pentahydrate in 100 ml ethanol while swirling dynamically. Drop by drop, 4 mL of aqueous ammonia solution was added to the aforementioned solution under steady stirring. For purification, the gel was filtered and washed with ethanol before being dried for 4 hours at 60 ºC. The resultant powder was calcined for 2 hours at 400 ºC, yielding ash-colored tin oxide nanoparticles [11 -13].

B. *Synthesis of PPy – SnO_2 nanocomposite*

A known quantity of Aniline solution dissolved in HCl is taken in 1000 ml beaker and stirred for 5 minutes and 0.5 g (10wt %) of SnO_2 nanoparticles were added and stirred with magnetic stirrer for about 15 minutes then ammonium per sulphate was added drop by drop. Even after complete addition of ammonium per sulphate stirring was continued for another 10 minutes and allowed the precipitate for about 30-40 minutes to settle down. Now precipitate was filtered and washed with distilled water several times to remove the impurities. Finally washed with acetone and precipitate was dried on its own at room temperature and was grinded for 15 minutes with morter and pestal. Now the resultant sample is the PANI-SnO_2 nanocomposite with 10 wt% of SnO_2. In the same manner, PPy-SnO_2 nanocomposites with 20wt%, 30 wt%, 40 wt% and 50wt% of SnO_2 are synthesized.

III. Preparation of Pellet

The powders of PPY, PPY/ Tin oxide nanocomposites, so obtained from synthesis techniques discussed in early sections were crushed and finely ground in agate mortar in the presence of acetone medium. The powder is then pressed to form pellets of 10 mm diameter and thickness varying up to 2 mm by applying pressure of 90 MPa in a hydraulic press. For temperature dependent conductivity and sensor studies, the pellets of PPy and its metal oxide nanocomposites are coated with silver paste on either side of the surfaces to obtain better contacts.

IV. Characterization

The morphology of the nanocomposites in the form of powder was investigated using scanning electron microscope (SEM) Model-EVO-18 (Special Edison, Zeiss, Germany),TEM . differential scanning calorimetry (DSC) was investigated by Instrument: DSC Q20 V24.10 Build 122. DC conductivity of these nanocomposites are studied by using Keithley 6514 electrometer, sensing properties of these nanocomposites were studied using laboratory set up.

V. Results and Discussion

A. *Scanning Electron Microscopy*

Figure 1 displays the morphologies of samples, As shown by the SEM images, PPy displays a typically cauliflower-like or tumor-like structure. The addition of SnO2 with uniform spherical structure observed in the nanocomposites provides the space factors for PPy orderly growth, resulting in special morphology of PPy. PPy/SnO2 nanocomposite particle size is much less than that of PPy .Therefore, the SEM results elucidate that the reactions are remarkably effective for the PPy functionalization.

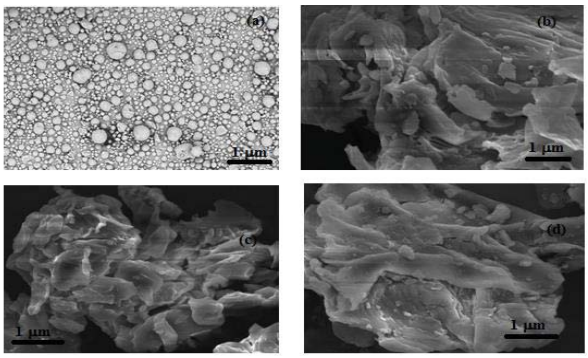

Fig.1. (a) shows that Scanning Electronic Micrograph image of pure PPy and (b) PPy/Tin oxide nanocomposites

B. *Differential Scanning Calorimetry*

The DSC curve of PPy and PPy/dopant sample have a broad characteristic endothermic dip indicates the glass transition temperature of Polypyrrole at 99 ºC as shown in figure 2. The nature of the curve indicates that the loss of water is overlapping with T_g of polymer Lack of any shoulder or melting peak beyond this region indicates amorphous nature with less sequence of the PPy molecules. The DSC curve of PPy/20 wt% composite trace has a sharp dip at higher temperature, namely at 100ºC. This sharpness indicates the better crystalline. It also contains few more inflection points from 330 ºC to 360ºC may be due to the melting of PPy chain, the other at 360ºC to 400ºC may be due to phase change of dopants.

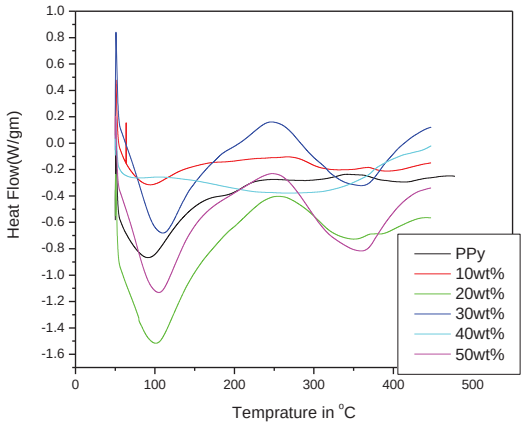

Fig.2. shows that the DSC curve of PPy and PPy/dopant samples

C. *DC Conductivity*

Figure 3 shows the temperature dependency of dc conductivity for Polypyrrole/ SnO_2 Nanocomposites over a temperature range of 30 to 190 ºC. The conductivity values of the examined composites are clearly higher than those observed for pure PPy. Up to a transition temperature, the conductivity increases continuously with temperature, demonstrating semiconductor characteristics. Conductivity rises when temperature rises due to the movement of charge carriers (polarans) from one localized state to another. The conductivity of 50wt percent of all nanocomposites is higher, indicating that conductivity is determined not only by ion mobility (SnO_2) but also by charge carrier hopping. The conductivity is proportional to the, and follows an expression of the type:

$$\sigma \quad (T) = \sigma_0 \exp [-T0/T)1/4]$$

where: σ is the conductivity, T is the temperature and σ0 is the conductivity at characteristic temperature T0.

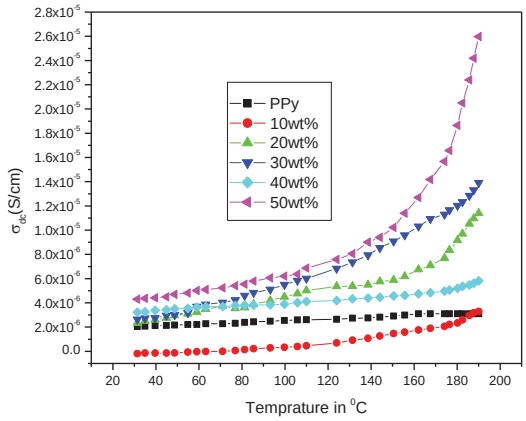

Fig.3 shows DC conductivity of PPy/SnO_2 nanocomposites.

D. Sensing Study

The sensitivity of Polypyrrole/ Tin oxide nanocomposite 50 wt% for LPG sensing is shown in figure 4. The variation in the Sensitivity of the composites could be due the following reasons. The LPG molecules induced and trapped into polymer matrix might cause it to swell leading to the disruption of conducting paths through the composites. This results in increased Sensitivity of composites. After removal of gas, the polymer returns to original size, restoring the conducting paths. The possible mechanism of detection of LPG gas by LPG is based on surface reactions. . The overall conduction in a sensor element is determined by the surface reactions, the resulting charge transfer processes with the underlying Tin oxide and the transport mechanism through the sensing material and morphology of sensing layer.

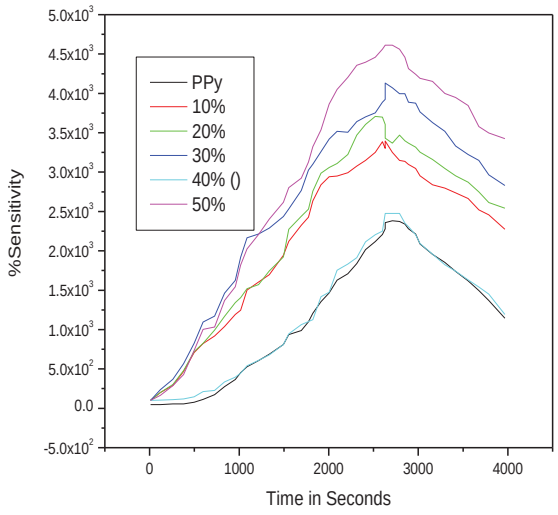

Fig.4 shows DC conductivity of PPy/SnO_2 nanocomposites.

VI. Conlusion

The fabrication of conducting PPy / SnO_2 nanocomposites utilizing an in-situ chemical polymerization technique is described in this paper. These composites' conductivity and sensing characteristics were investigated. The influence of SnO_2 concentration on conductivity qualities has been studied. At room temperature, the composites' LPG sensing characteristics were examined, and the better responsiveness of the PPy-SnO_2 nanocomposite was explained in terms of the synergetic interaction of both PPy and SnO_2 particles. The composites' rapid reaction to changes in sensitivity, as well as the linear fluctuation in the parameters, point to their potential as an effective LPG sensing material.

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