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Synthesis, Characterization and CO_2 Gas Sensing Response of 5% SnO_2 Doped Polyaniline Nano Composite

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ABSTRACT

Polyaniline and its nano-composites are synthesized using in-situ chemical oxidative polymerization technique using aniline. Nano sized SnO_2 is used as received from the manufacturer. Ammonium Per Sulphate is used as oxidizing agent for polymerization. Crystalline SnO_2 is embedded in amorphous Polyaniline. The structure of the composite was confirmed by the characterization techniques FTIR, UV Visible and XRD.

Average particle size and chain separation is determined using XRD. The little shifting of the wavelengths towards higher values in FTIR confirms the formation of Polyaniline. UV Visible studies show that the composite exhibit absorption peaks at 614 nm, 328 nm and 263 nm; which corresponds to band gap energies 2.02 eV, 3.78 eV and 4.71 eV respectively. V-I characteristic is plotted using two probe method, which indicate fairly linear or ohmic behavior of the sample with high resistivity. Gas sensing response to CO_2 is observed.

Keywords – Polyaniline, nano-composites, band gap energy, gas sensing, CO_2, SnO_2.

I. INTRODUCTION

The conducting polymers made significant impact in the field of material science due to their potential applications in many electronic devices [1]. Technological uses depend crucially on the reproducible control of the molecular and supramolecular architecture of the macromolecules via a simple methodology of organic synthesis [2]. Polyaniline is one of the important conducting polymers among all other conducting polymers because of its stability in air, easy polymerization, low cost, good conductivity, and solubility in some organic solvents. It is the type of conducting polymer whose properties can be changed by protonation state, oxidation state, and also by nature of dopant [3-6]. In general the change in properties make polyaniline a versatile material. The preparation of polyaniline composites with various materials has received great attention because of their unique properties and allocations in various electrical and electronic devices. Several reports dealing with the preparation of conducting composites such as Fe_3O_4:PANI, MnO_2:PANI, TiO_2:PANI, ZrO_2:PANI [7,8], as well as preparation and characterization of ZnO:PANIcomposites have been published [9-11] . Due to the reasons it has been studied extensively for making optical and electronic devices like LEDs, solar cells, transducers, photodetectors, etc.[12-14]. In particular SnO_2 nanostructures are of intense interest

since it can be grown by a variety of methods with different morphologies.

On the other hand, functional metal oxides have received increasing attention due to their unique physical properties. Functional oxide have two structural characteristics- cations with mixed valance states and anions with deficiencies (vacancies). By varying either or both of these characteristics, the electrical, optical, magnetic, and chemical properties can be tuned, giving the possibility of fabricating smart devices that utilizes the semiconducting , superconducting, ferroelectricity and/or magnetism offered by the oxides. Among the technologically promising functional metal oxides, tin oxide (SnO_2) is used in various opto-electronic devices like flat panel display, photoconductor and solar cells. SnO_2 is n-type semiconductor with a band gap of 3.6 eV at 300 K where as PANI is a typical conductive polymer which is usually considered as a p-type material. In view of forgoing, by synthesizing nano-composite of SnO_2/PANI, electrical and optical properties can be enhanced.

Growing industrialization and increasing pollutants from vehicular exhaust have resulted into increased air pollution. The problems related to air quality monitoring are important issues of current research activity. At present, CO_2 gas is being polluting the envirnment to a large extent. This may cause severe health problems leading to reduce the efficiency of human resources. To overcome the problem, an CO_2 gas sensor is required[ ].

In the present work the composite of polyaniline with SnO_2 was synthesized at a five (5) weight percentage by chemical oxidation polymerization method using ammonium persulphate as an oxidizing agent. With this background of multifunctionality SnO_2 is used in preparation of composite [9-11]. The formation of composite was characterized by using XRD, FTIR and UV/Visible.

II. METHODS AND MATERIAL

The chemicals aniline monomer, ammonium persulphate (APS) $(NH_4)_2S_2O_8$ and hydrochloric acid (HCl) were procured from LOBA chemicals, Mumbai. The nanosized SnO_2 powder was procured from nanolabs, Jamshedpur, Zarkhand

Synthesis of PANI-SnO2 is carried out by following process.

Polyaniline hydrochloride of weight 2.59 gm and SnO2 5% by weight is dissolved in water to make 50ml solution in a volumetric flask.

Then APS of weight of 5.71gm is dissolved in water to make 50ml solutions in another volumetric flask. Both solutions were stirred and kept for 1hour at room temperature (25c). After the time, both solutions were mixed together and stirred for 4 hours in magnetic stirrer and after that left for polymerization for 24 hours.

PANI-SnO2 precipitate was formed. It is collected on filter paper and washed several times with 100ml portion of 0.1M HCl (35% conc.) and similarly with acetone (pure 99%) till clear solution is obtained.

The material collected on filter paper is dried at 60c temperature in oven overnight till it gets moisture free powder. The yield is found to be 78.66%.

The nanocomposite was characterized using XRD, FTIR and UV/Visible.

III. RESULTS AND DISCUSSION

The PANI/ SnO_2 nanocomposite powder were used for XRD measurements using Cu(Kα) radiation of wavelength 1.5418A in a range 5˚-80˚. The nanocomposites was studied using FTIR Shimandzu, Japan in the range 500-4000 cm^{-1} and UV/Visible Shimadzu, Japan in the range 200nm to 1100nm.

a) XRD

X-Ray diffraction pattern with two prominent peaks leads the amorphous nature. The XRD spectrum of

PANI/ZnO 10wt% and PANI/SnO_2 10wt% are shown in fig. 1(a), 1(b).

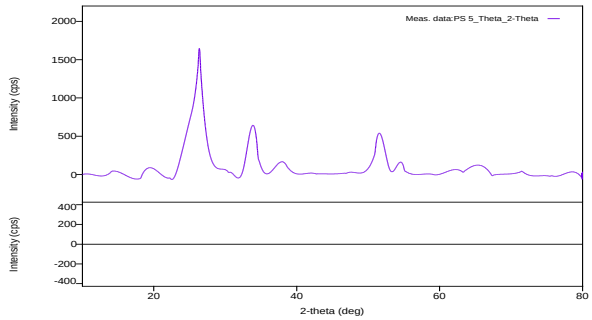

Fig. 1. XRD spectrum of PANI/SnO_2 5wt%

It is notified that sharp and well defined peaks are observed. This shows that the nanocomposite is partially crystlline in nature. The peaks are observed for the composite at 20^0, 24^0 and 25.6^0 which confirms the presence of SnO_2. Which depicts that it has crystalline nature.

The particle size could be estimated using Debye – Scherrer's formula, $D = \frac{0.9\lambda}{\beta cos\theta}$

Where, D is the particle size, β is full width at half maximum (FWHM) for stronger peaks and λ is the wave length of X-rays. From the high intensity peaks the particle size are calculated in the range 4.1 nm.

b) FTIR-

The FTIR spectra of PANI/SnO_2 5 wt% is shown in fig 2.

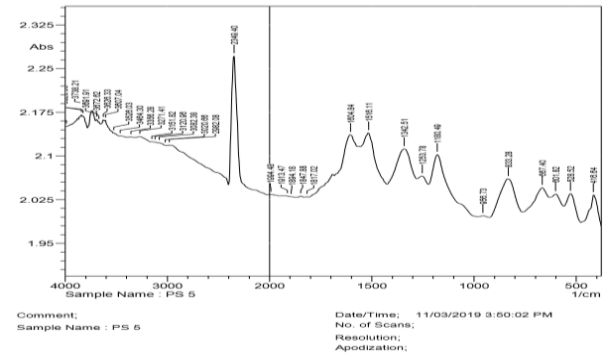

Fig 2. FTIR spectra of PANI/SnO_2 5wt%

Characterization absorptions peaks are obtained from FTIR spectrum of PANI-SnO2. The characteristic absorption bands of PANI are 667.40 cm^{-1} due to C=C strong bending mode, 1342.51 cm^{-1} represents C-N strong strechting, 2982.08 cm^{-1} represents N-H strong stretching and 3082.38 cm^{-1} indicates C-H medium streching.

c) UV-Vis

The UV-Vis spectra of nanocomposites are shown in figs. 3(a), 3(b). Using these spectra the band gap energy can be found out. The formula is, $E_g = \frac{hc}{\lambda e}$

Where h is Planck's constant (6.626 x 10^{-34} J-sec), c is the speed of light (3x 10^8 m/s), λ is the wave length and e is the charge of electron (1.602 x 10^{-19}C).

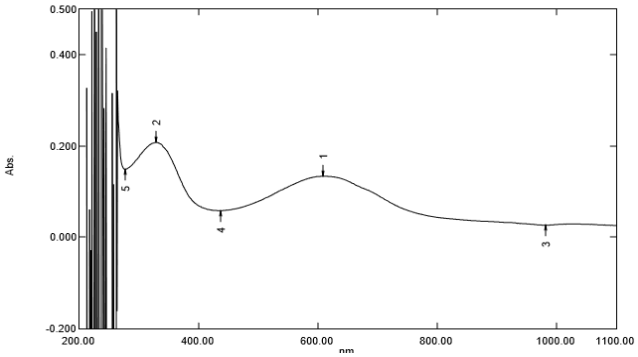

Fig. 3. UV-Vis spectra of PANI/SnO_2 5wt%

The SnO_2 doped polyaniline nanocomposite exhibits absorption peaks at 329 nm and 608 nm which corresponds to band gap energies 3.47eV and 2.02eV respectively.

d) DC Conductivity

The film of the composite is deposited using spin coating technique on pre-cleaned glass substrate using 8% polyvinyl acetate as a binder. For measurement of conductivity, juxtaposed copper electrodes, each of length 0.6 cm and 1 mm separation between them, are gently placed on the film with the help of loose spring. Mili-ammeter (Meco-301) and a dc power supply (Agronik - 30) are used for the purpose.

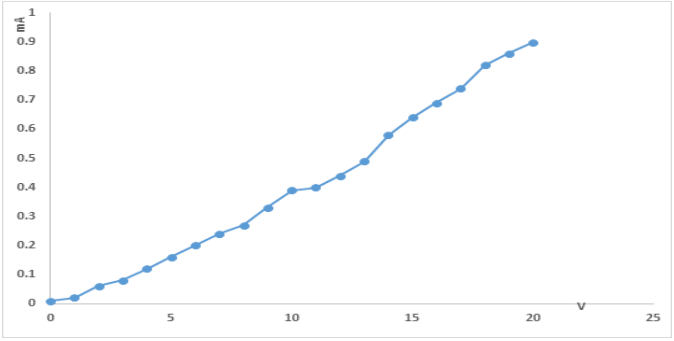

Fig. 4. V- I charectoristic of PANI/SnO_2 5wt%

The graph represents variation of current through surface of sample with voltage. The graph is fairly linear indicating ohmic nature of the sample. This may be due to transfer of electrons as charge carriers in the direction of applied field. Surface resistivity is calculated from the formula - $\rho = \frac{E}{J} = \frac{V}{I} L$

where, V is voltage applied, I is surface current and L represents length of the electrode. The surface resistivity, ρ is evaluated as 490 Ω Mtr.

e) Gas Sensing Response

Gas sensing response of the sensor is defined as change in conductance of a sample upon exposure to target gas to the original conductance in air. The figure shows gas response of PANI/SnO_2 5wt% thick film to CO_2gas at room temperature.

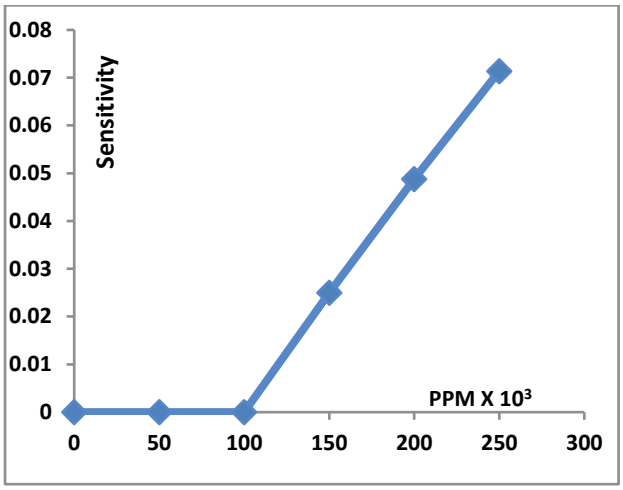

Fig. 5. Response to CO2 of PANI/SnO_2 5wt%

From fig.5, the sensitivity increases fairly linearly with concentration of the gas. The exact fundamental mechanisms to explain gas sensing response are controversial.

IV. CONCLUSION

PANI/SnO_2 5wt% nano composite has been successfully synthesized by in – situ polymerization using chemical oxidation method. The XRD- spectra of composite reveals partially crystalline nature. The peak positions differ from SnO_2 compared to its composites and this indicates that the modification has occurred in the composite structure. The size of the grains is in the nano-meter range as found out from XRD. The PANI/SnO_2 composite is promising materials which may be applicable in optoelectronic devices and organic solar cells.

V. ACKNOWLEDGEMENT

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