



Humidity Sensing Properties of SnO₂ Doped with Polyaniline

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Abstract

The use of sensors by human being has been day by day increasing at an outstanding rate in the last few decades and modern society depends heavily on the use of the sensors for variety of purposes. Over the last eras a variety of chemical sensors have been developed based upon semiconductors, which monitor different characteristic sensor properties like conductivities for electronic conductivity sensors, impedance for capacitance sensors, potentials for field effect sensors or temperatures for calorimetric sensors. In this work we have synthesized nanocomposites of conducting polymer and metal oxides by chemical route method. In this work the material SnO₂ and polyaniline (PANI) were used with different weight percent and prepared pristine and mixed nanocomposites by suitable techniques and calculate change in the relative humidity. It is observed that PANI doped SnO₂ nanocomposites sensor shows a high response and sensitivity with good repeatability as compared to that of pure PANI and SnO₂ nanoparticle. The effect of hysteresis plot of sensors, sensitivity of the sensors were studied. The crystallinity and the crystallite size were examined by X-Ray Diffraction technique and surface morphology of synthesis is studied by Scanning electron microscopy (SEM),

Keywords: Polyaniline, SnO₂ nano composites, Humidity sensor, Screen printing technique

1. Introduction :

There is a growing demand for a sensing system that has high sensitivity, wide dynamic range, good stability, quick response, good reproducibility, simple structure and minimal cost. Metal oxide films sensitive to humidity have been reported earlier where sensing has been done using optical means. However, metal oxide humidity sensors depending upon measurements of electrical parameters require high temperature operation and consume significant amount of power. Humidity control and monitoring are of great interest to a wide area; these include moisture sensitive products, fresh and pack-age food, drug storage and environmental control for valuable Antiques or paintings etc. [1, 2]. Humidity sensors that are available in the market include dew point, infrared, catalytic and tin oxide-based sensors, which may be expensive, or require high temperature operation and consume significant amount of power and high cost of maintenance [3]. Much research has been focused on the development of humidity sensitive material [4–6]. Among these are the investigation of using conducting polymers such as polyaniline, polypyrrole, and polythiophene for humidity and gas sensing [7–9]. Generally, conducting polymers behave quite similarly on exposure to humidity. A resistance decrease (or conductivity increase) is observed due to the formation of H-bonding between water molecules and the nitrogen centre of the polymer backbone. This facilitates the proton exchanges to increase the doping level of the conducting polymer and so the number of charge carriers increases, resulting in an increase in conductivity [10].

2. Synthesis of Material:
A) Synthesis of Polyaniline (PANI): In general is synthesized using two major polymerization approaches: electronic and chemical polymerization. In the present work polyaniline is synthesized by chemical polymerization method in which 0.2 M aniline hydrochloride is used as monomer unit. The synthesis is done by oxidative polymerization with 0.25 M ammonia peroxydisulfate in aqueous medium. Both solutions kept 1 hour at room temperature then mixed in beaker, briefly stirred. And left at rest to polymerized, next day, the PANI precipitate was collected on a filter, washed with three 100 ml portion of 0.2 M HCL and similarly with acetone. Polyaniline hydrochloride powder was dried in air and then in vacuum at 60°C. Polyaniline prepared under these reactions and processing condition are further referred to as standard sample.

B) Synthesis of Tin oxide (SnO₂): In preparation of SnO₂, 2 g (0.1 M) of stannous chloride dehydrate (SnCl₂·2H₂O) is dissolved in 100 ml water. After complete dissolution, about 4 ml ammonia solution is added to above aqueous solution with magnetic stirring. Stirring is continued for 20 minutes. White gel precipitate is immediately formed. It is allowed to settle for 12 hrs. Then it is filtered and washed with water 2-3 times by using deionized water. The obtained precipitate were mixed with 0.27 g carbon black powder (charcoal activated). The obtained mixture is kept in vacuum oven at 70 °C for 24 hours so that the mixture gets completely in to dried powder. Then this dry product was crushed into a fine powder by grinder. Now obtained product of fine

nanopowder of SnO₂ was calcinated at 600°C up to 6 hours in the auto controlled muffle furnace (*Gayatri Scientific, Mumbai, India.*) so that the impurities from product will be completely removed.

3. Characterization :The above synthesized PANI- SnO₂ composites are structurally and surface morphologically characterized by using different technique like X- ray diffraction (XRD),the x-ray diffraction patterns of the prepared samples are obtained by Siemens D 5000 X-ray diffractometer using CuK α radiation ($\lambda = 1.717 \text{ \AA}$). The diffractograms are recorded in terms of 2θ in the range 40°-50° at ambient temperature with scanning rate of 2° per minute .The surface morphology of polyaniline and its composites are studied by using Leica's SEM (modal S 440) at 10kv.

3.1. XRD Pattern of SnO₂

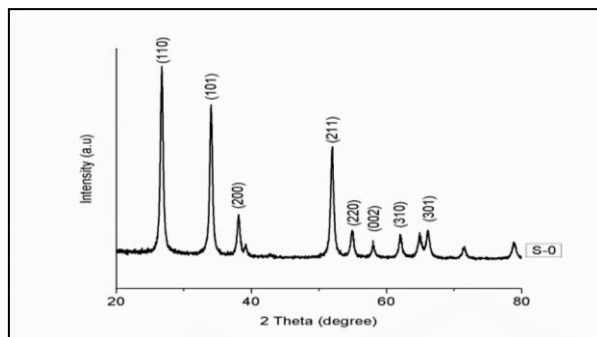


Fig. 3.1 XRD of Pure SnO₂

XRD pattern of pristine Tin oxide (SnO₂) nanostructure synthesized by liquid phase method via chemical wet reaction method were calcinated at 800°C as shown in figure 3.1. it is clearly observed that the highest intensity peak is obtained at (110) crystal planes and other peaks lying at (101), (200), (211), (220) and (002) of SnO₂. All the peaks match well with the standard tetragonal structure of SnO₂ with lattice constant $a = 4.723 \text{ nm}$ and $c = 3.238 \text{ nm}$ and its unit cell volume ($V=72.24\text{A}^3$). All the peaks are perfectly match with pure SnO₂ nanostructure, which indicates the high purity of obtained SnO₂ nanoparticles. The average crystalline size was found to be 24.21 nm calculated by using Debye-Scherer formula.

3.2. Scanning Electron Microscopy:

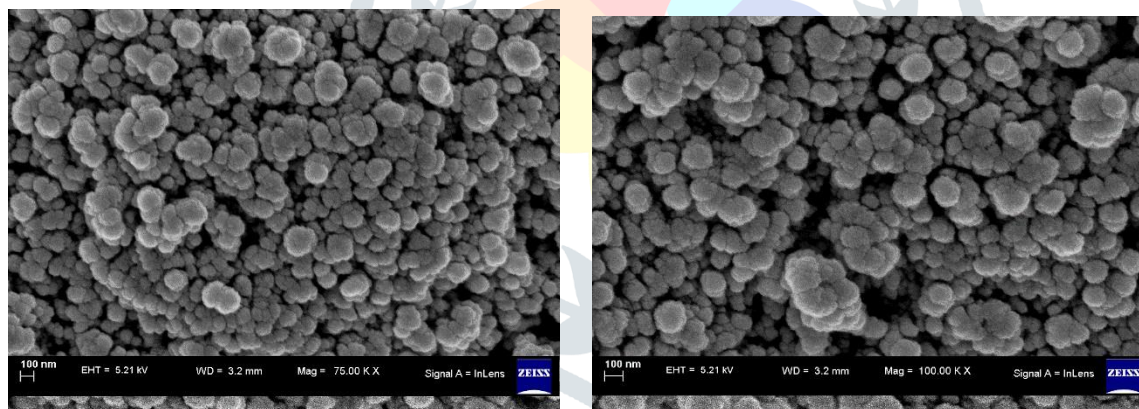


Figure 3.2. Scanning Electron Microscopy (SEM)

Figure 3.2. shows the Scanning Electron micrograph of sample pristine SnO₂ thick films in this the particles are found to in the tetragonal shape within the particle size in the range of about 20.3 nm to 31.2 nm.

4 . Result and Discussions:

4.1. Hysteresis plot

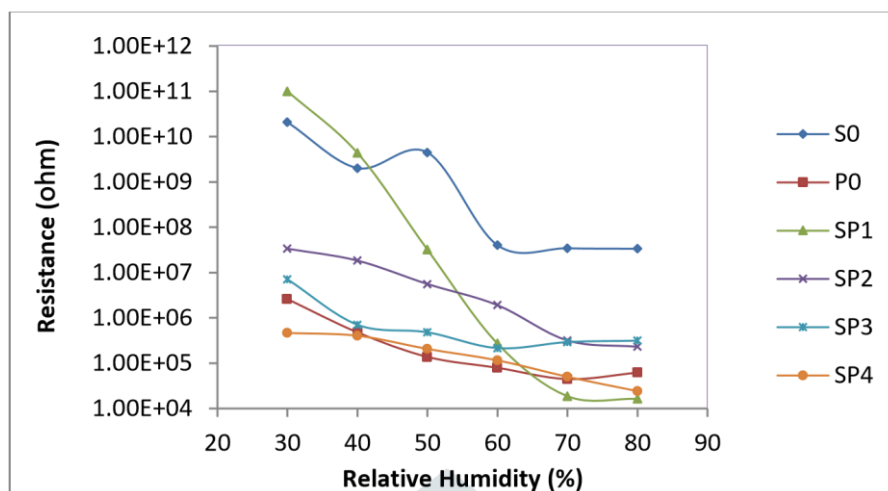


Figure: 4.1. Hysteresis plot

Hysteresis plot shows the variation between resistances of sample with respect to the relative humidity in increasing and decreasing order from 30 to 80 % RH as shown in the fig. 4.1. A very small hysteresis present during forward and reverse cycle of relative humidity, where as a very significant average change observed in the value of resistance of sample, in the sample SP-1 (10 SnO₂ – 90 PANI) the change in value of resistance is from 10¹¹Ω to 10⁴Ω, this is a remarkable change in the value of resistance was observed respectively.

4. 2.Sensitivity:

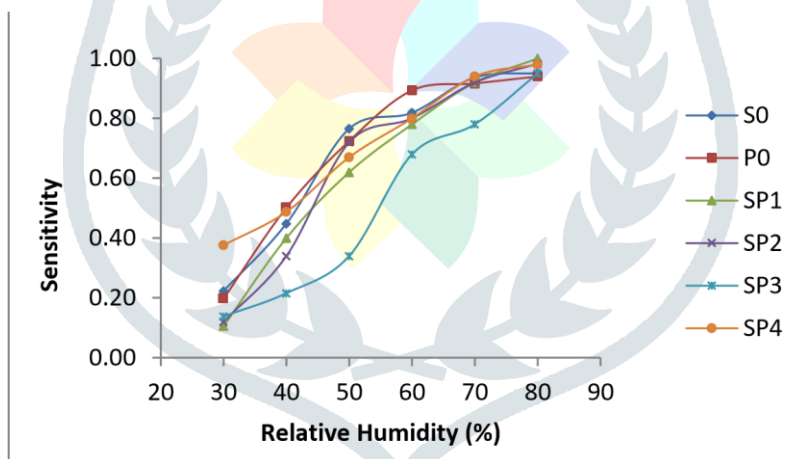


Figure: 4.2. Variation of sensitivity with Relative Humidity

In the above samples the sensitivity is found to be increasing with the RH for all the samples of thick films sensors and it is increasing up to some particular RH and then afterward it remains continuous as shown in fig. 4.2.

For higher RH the sensitivity is found to be higher in case of all samples of thick films. The sensitivity of SP-1 (10 SnO₂-90PANI) is more than sample SP-2, SP-3, and SP-4 and also from the pristine samples. The (SnO₂-PANI) composite sensors exhibits significantly higher sensitivity than sensor constructed specially from SnO₂ nanoparticles and PANI itself due to the formation of heterogeneous interface between them and more adsorption site was created to absorbed more water vapours.

5. CONCLUSIONS:

Nanostructured SnO₂ was successfully prepared via chemical precipitation method and PANI with IUPAC polymerization technique. Minimum crystallite size was found to be for SnO₂ is found to be 24.21 nm. The crystalline size i.e. grain size are in the range of nano meter surface morphology was confirmed by using Scanning electron microscopy (SEM). The Hysteresis plot shows very significant average change in the value of the resistance from 10¹¹Ω to 10⁴Ω during forward and reversed cycles of sample SP-1(10 SnO₂-90PANI). The sensitivity is found to be increasing with the RH for all the samples of thick films and it is

increasing up to some particular RH and then afterward it remains constant. Amongst all the prepared samples SP-1 is more sensitivity than other prepared composite samples.

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