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Synthesis and Characterization of PANI Doped SnO2 for Humidity Sensing Application

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Abstract:

In the present work Polyaniline is prepared by polymerization of aniline under acidic conditions. Tin Oxide (SnO_2) prepaired by the Precipitation method at room temperature. Polyaniline (PANI) based Metal oxide nanocomposites were prepared by using the screen printing technique. The films were fired and optimized temperature of 60ºc for 30 minutes in an air atmosphere. The films were showing a decrease in resistance with an increase in temperature indicating semiconducting behavior. It is observed that PANI doped Metal oxide nanocomposite sensor shows a high response and sensitivity with good repeatability as compared to that of pure PANI and Metal oxide nanoparticle. The crystallinity and the crystallite size were examined by X-Ray Diffraction technique (XRD), and Scanning Electron Microscopy (SEM).Also confirms that the properties of pure polyaniline can be improved by the synthesis of Polyaniline - Metal oxide nanocomposites.

Keywords: Polyaniline , Metal Oxides Nanocomposites, Humidity sensor, XRD, SEM

Introduction:

Humidity control and monitoring are of great interest to a wide area; these include moisture sensitive products, fresh and package food, drug storage and environmental control for valuable Antiques or paintings etc. [1, 2]. The humidity chambers are designed to maintain temperature and relative humidity at set points controllable by the operator at the front panel. Air is constantly being circulated through the chamber, scheduled for comparison to set points. Heat is produced by electric resistance heaters that turn off and on for temperature control. On units with cooling there is a refrigeration unit continuously on. The Chamber humidification is reached by means of a low-pressure vapor generator injecting water vapor into the chamber through a small orifice. The water vapor is reached into the chamber at the blower discharge. Chambers were programmable and Web- enabled test chambers are also available.[4,5].The 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 [5,6].

Synthesis of Material:

A) Synthesis of Polyaniline (PANI): in genral 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 peroxysulphate in aqueous medium. both solution kept 1 hour at room tempraturethen mixed in beaker ,briefly stirred. And left at rest to polymeriz, 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 hydrochoride powder was dried in air and then in vaccum at 60°c. Polyaniline prepared under these reactions and processing condition are further referred to as standard sample.

B) Synthesis of Tin oxide (SnO_2): In preparation of SnO_2, 2 g (0.1 M) of stannous chloride dehydrate ($SnCl_2.2H_2O$) 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 obtain precipitate were mixed with 0.27 g carbon black powder (charcoal activated). The obtained mixer is kept in vacuum oven at 70 ºC for 24 hours so that the mixer gets completely in to dried powder. Then this dry product was crushed into a find powder by grinder. Now obtained product of fine nanopowder of SnO_2 was calcinated at 600^{o}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

Results And Discussion:

Figure 1, 2, shows XRD pattern of pristine material SnO_2 and Polyaniline (PANI) nanocrystalline materials respectively.

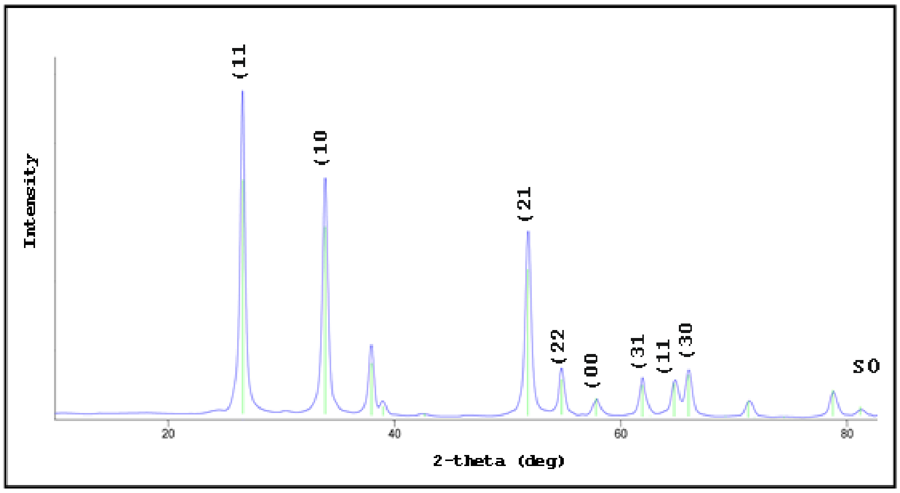


Figure 1. XRD of pristine (SnO_2) (S-0)

Figure 1. shows the XRD pattern of pristine stannic oxide (SnO_2) nanostructure synthesized by liquid phase via co-precipitation method calcinated at 600^{o}C 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 SnO_2. All the peaks match well with the standard tetragonal structure of SnO_2 with lattice constant a = b = 4.723 nm and c = 3.18 nm and its unit cell volume (V=71.48A^{o3}) . All the peaks are perfectly match with pure SnO_2 nanostructure, which indicates the high purity of obtained SnO_2 nanoparticles. The average crystalline size was found to be 28.95 nm calculated by using Debye-Scherer formula [7].

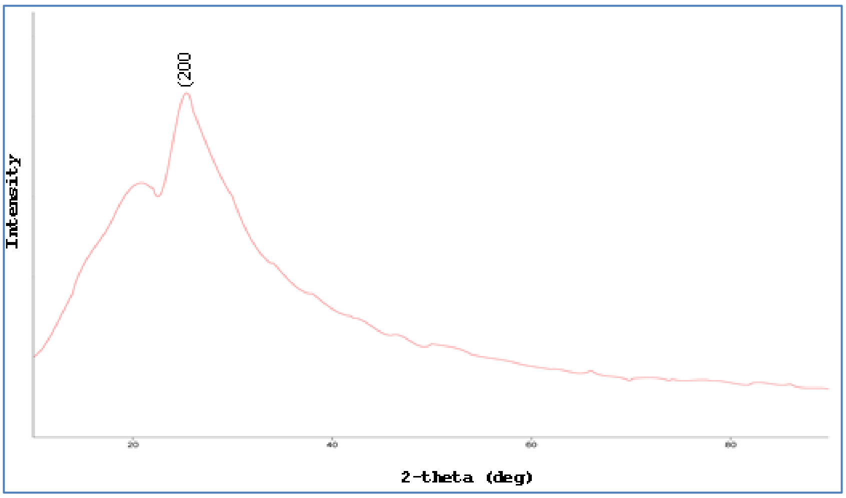


Figure 2. XRD of pristine (Polyaniline) (P-0)

Figure 2. shows the XRD pattern of pristine Polyaniline (P0) nanostructure synthesized by precipitation method and calcinated at 70°c was studied by XRD spectrometer using cuk$_\alpha$ lines the spectra indicates the almost amorphous nature of sample as shown in fig- 2. the film prepared at higher thickness show polyaniline nature having peak for polyaniline at 2θ = 25.58. Similar observations on polyaniline sample have been reported by previous worker [8]

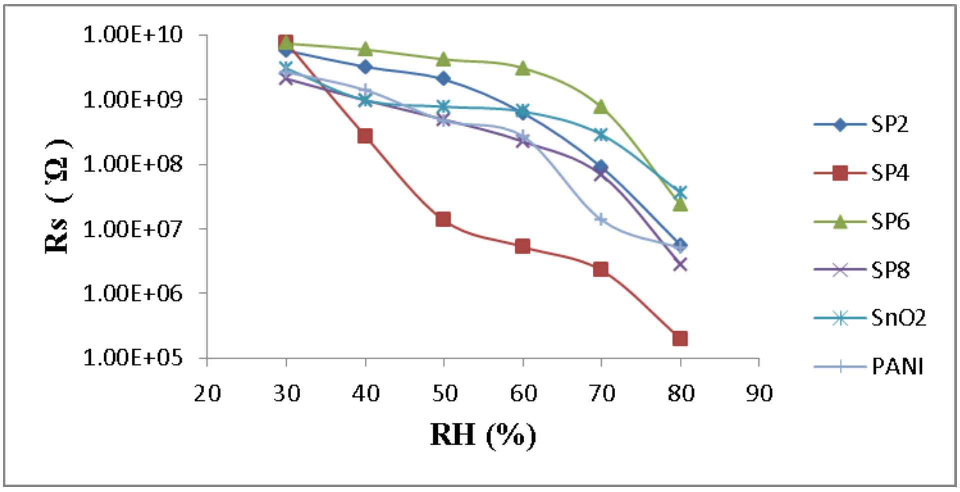


Fig. 3. Hysteresis plot

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. 3. 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-4 (40SnO_2- 60PANI) the change in value of resistance is near about from 10^{10} Ω to 10^5 Ω, these is a remarkable change in the value of resistance. it is also noted that ,by increasing the sensor temperature from 30° C to 80 ° C, its resistance decreases at a particular constant humidity due to process of adsorption and deadsorption.

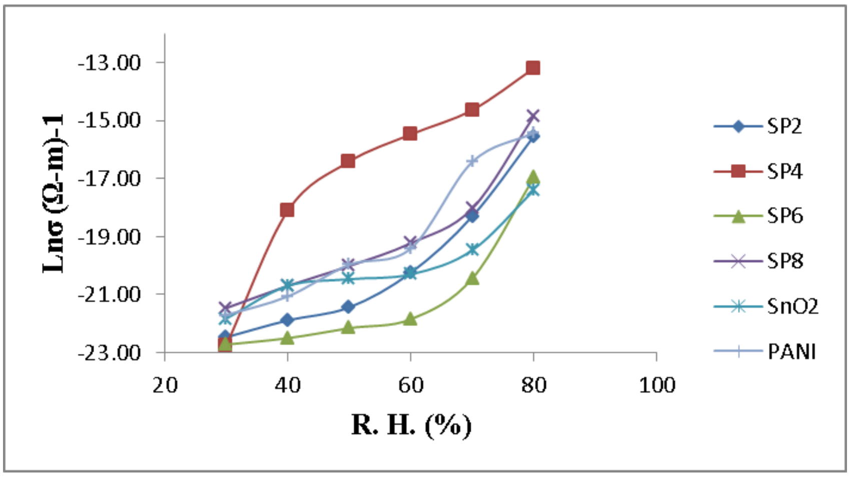
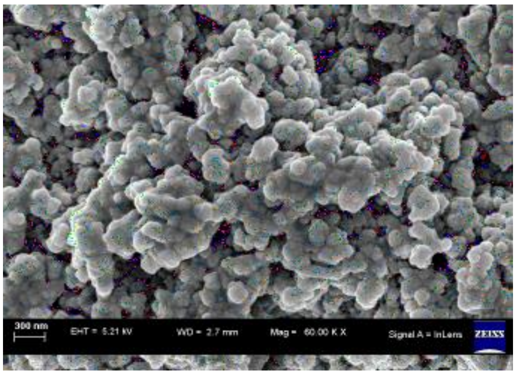
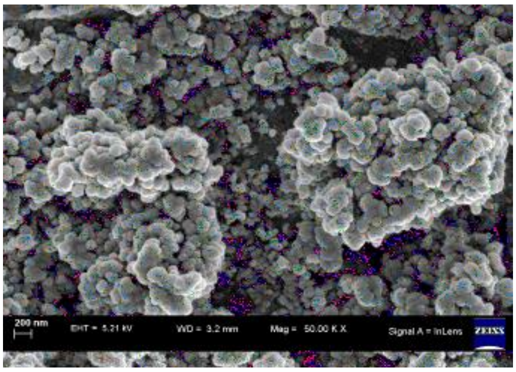


Fig. 4. Variation of Conductivity with Relative Humidity

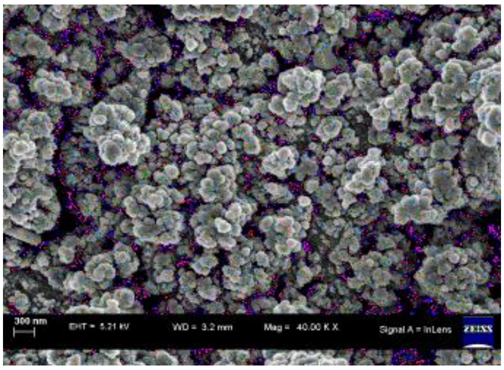
It is observed that in fig .4 The conductivity increases perfectly linearly with relative humidity from 30 to 80 % RH and decreasing relatively on same path from 80 to 30 % RH. In this case also the conductivity increases with increase in temperature and it is highest at temperature 80°C and lowest at temperature 30°C. the conductivity difference between temperature 30°C and other ones are minimizes and the conductivity curves are not seen mixed so as seen in the group of sample. This behavior is obtained due to the addition of large amount of PANI in the SnO_2 and the conductivity get stabilized through the resistance to near in the range of Megaohm (MΩ) . Hence the stability, linearity and equality are obtained in the samples.[9,10].



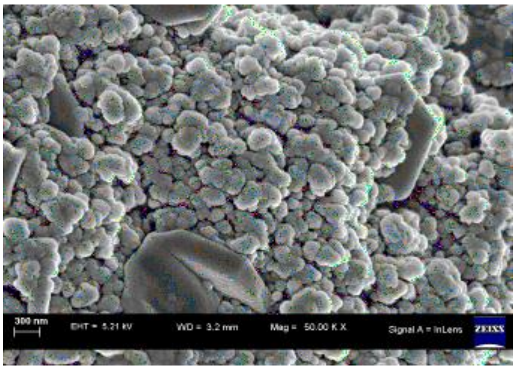
SP2 (a)



SP4 (b)



SP6 (c)



SP8 (d)

Fig. 5. FESEM Image of PANI Doped Metal Oxide Nanocomposites

The FE-SEM morphology of nanocomposites shows the particles are small sized, almost spherical flower like, rod like structure. The micrograph of SP-4 (figure.5.(b).) One can see that nanocrystalline and porous SnO_2, is formed on the surface of SP4 .By using crystalline quantization plot, these more peaks carrespond to SnO_2 , than Polyaniline .The average crystalline size is obtained by using scherrer formula and it has been found to be 40.27nm, 35.20 nm, 38.25 nm, 41.31nm , Each grain seems to be like a bead of different shape and reveals that they possess the grain size of nanometer order and shows nanoporous structure. It means that the structure is likely to facilitate the adsorption and condensation processes of water molecules because of the capillary pore and having large surface area. This porosity leads to an effective response and recovery towards humidity. [11].

Conclusions:

Nanostructured SnO_2 was successfully prepared via chemical precipitation method and PANI with IUPAC polymerization technique. Minimum crystallite size was found to be for SnO_2 (S0) is 28.95 nm , and Polyaniline 25.58 nm.The Hysteresis plot shows very significant average change in the value of the resistance from near about $10^{10}\Omega$ to 10^5 Ω during forward and reversed cycles of sample SP-4(40SnO_2 -60PANI). The conductivity and 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. The FESEM Morphology reveals that SnO_2 was uniformly mixed within the PANI matrix. The nanocomposites could be good material for detection humidity at room temperature .It means that the structure is likely to facilitate the adsorption and condensation processes of water molecules because of the capillary pore and having large surface area.

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