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Electrical and Gas Sensing Properties of Conducting Polyaniline/SnO_2 Nanocomposites

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Abstract: ***PANI/SnO_2 has been synthesized and studied in terms of XRD, SEM, DC conductivity variation of temperature, AC conductivity, dielectric behavior can be studied variation of frequency at room temperature and Gas sensing properties with help of H_2S gas injected at room temperature. The conducting polymer nanocomposite of PANI - SnO_2 has been prepared by in situ polymerization method. The morphology of this nanocomposite has been studied by employing Scanning Electron Microscope (SEM). The Transport properties of these samples have been characterized by studying DC conductivity and AC conductivity. The AC conductivity and dielectric behavior of the nanocomposite have been investigated in the frequency range of 5Hz to 5 MHz. These nanocomposites have been found to have high dielectric constant which may be correlated with polarization. It has been also observed that both dielectric loss and dielectric constant decreases with an increase in frequency.***

Keywords: PANI, Tin Oxide, XRD, SEM, Dielectric, Nanocomposites, Conducting Polymer

1. Introduction

The conducting polymer/inorganic nanocomposites having unique physical properties that have attracted more and more attention. Conductive PANI (PANI) has been studied extensively because of its ease synthesis, environmental stability, electrical and other properties. SnO_2 has been attracted much attention as cathodes in rechargeable battery, selective gas sensors such as H_2S, ammonia because of their high surface area and redox activity. One of the important aspects of the SnO_2 is its layered lamellar structure. Many studies have been conducted to form PANI - SnO_2 composites structurers controlling internal morphology has still remained a challenge. In this paper, authors report synthesis, characterization, AC conductivity, dielectric constant, dielectric loss of polyamine - SnO_2 nanocomposites and gas sensing properties by employing 100 ppm of H_2S gas at room temperature.

2. Experimental Methods

2.1 Materials Method

Materials and Methods: All the reagents were analytical grade only and were used as received. Aniline monomer was distilled under reduced pressure and kept below 0 - 5 ^{0}C prior to use. Aniline monomer, hydrochloric acid (HCl), ammonium per sulphate [(NH4) 2S2O8] and Tin Nitrate were purchased from Merck chemicals ltd.

2.2 Synthesis of Tin oxide

Tin oxide nanoparticles were synthesized by self – propagating low temperature combustion method, employing tin oxalate as precursor. The precursor is prepared by dissolving equimolar quantity of ammonium persulphate and oxalic acid in distilled water.

2.3 Synthesis of Polyaniline

Synthesis of PANI was carried out by in - situ chemical oxidation polymerization Technique. Aniline (0.1M) was mixed in 1M hydrochloric acid and stirred for 15 min to form aniline hydrochloride. To this solution, add 0.1M of ammonium persulphate, which acts as an oxidizer was slowly added drop - wise with continuous stirring at 0 - 5^{0}C for 4 hours to get it completely polymerized. The precipitate was filtered, washed with deionized water, acetone and finally dried in an oven at 60^{0}C for 24hrs to achieve a constant mass. In this way, polyaniline (PANI) is synthesized.

2.4 Synthesis of Polyaniline - SnO_2 composites

Synthesis of PANI - SnO_2 nanocomposites was carried out by In - situ chemical oxidation polymerization technique. Aniline (0.1M) was mixed in 1M hydrochloric acid and stirred for 15 - 20 min to form Aniline hydrochloride. SnO_2 powder is added in the mass fraction to the above solution with vigorous stirring in order to keep the SnO_2 homogeneously suspended in the solution. To this solution, add 0.1M of ammonium persulphate (APS), which acts as an oxidizer was slowly added drop - wise with continuous stirring at 0 - 5^{0}C for 5to6 hours to be completely polymerized. The precipitate was filtered, washed several times with deionized water and acetone. Finally dried in hot air oven for 24hrs to achieve a constant mass. In this way, PANI - SnO_2 nanocomposites with various weight percentages of SnO_2 were synthesized. Later, the synthesized samples were made into a fine powder with the help of agate mortar.

3. Results and Discussion

X - Ray diffraction studies were performed using Philips X - ray diffractometer with CuK_α as the radiation source. The morphology of the nanocomposites in the form of powder were investigated using scanning electron microscope (SEM) Model - EVO - 18 (Special Edison, Zeiss, Germany). The DC conductivity of PANI and PANI - SnO2 nanocomposites were studied by using Keithley 2400 Electrometer. For DC conductivity studies, the samples were prepared in the pellet form (10 mm diameter and thickness varying up to 2 mm) by applying pressure of 10 tons in a Universal testing machine. The pellets were coated with silver paste on either side. Temperature dependent electrical conductivity was measured from 30^0C to170^0C using Keithley 2400 electrometer. AC conductivity has been studied by employing impedance analyzer LCR meter in the frequency range 50Hz to 5 MHz.

3.1 Structural Study

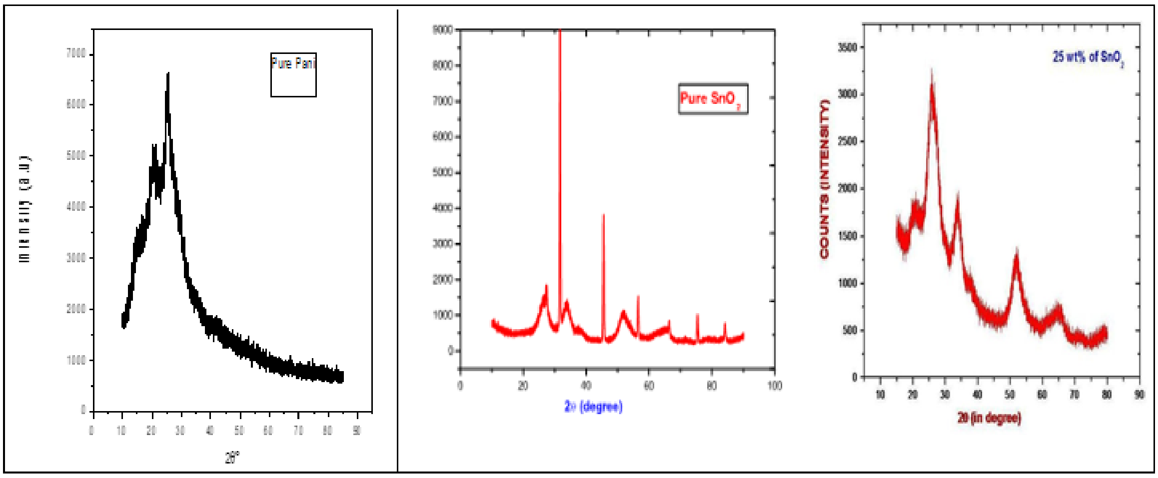

Figure: 1a Figure: 1b

Figure 1: X - ray diffraction pattern of (a) Pure PANI (b) Pure SnO_2 and PANI/SnO_2 nanocomposites.

The Figure 1 (a) shows X - ray diffraction pattern of PANI. which is amorphous in nature with a board peak centered on 2θ= 25.53° which corresponds to (200) diffraction planes of pure PANI.

Figure 1 (b) represents an X - ray diffraction pattern of Pure SnO_2 and PANI - SnO_2 nanocomposite, where well defined broad peaks are observed, which indicates good crystallinity of the materials. These nanoparticles have shown good crystallinity because of existence of sharp peaks in XRD pattern. The Crystallite size of the synthesized PANI - SnO2 nanoparticles was calculated using Debye Scherer's formula given by $D=0.9\lambda/\beta cos\theta$, where D is the average crystallite size, λ is the wavelength of X - ray (1.5405A) and β is the full width half maximum in radians. The average crystallite size is found to be 30nm This clearly says that the formation of SnO_2 is dispersed in PANI nanocomposite.

3.2 Morphological Study

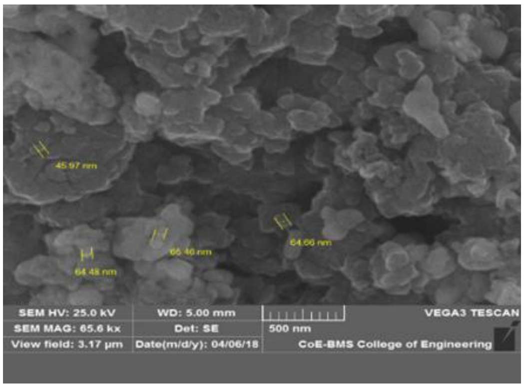

Figure 2 (a): Pure PANI

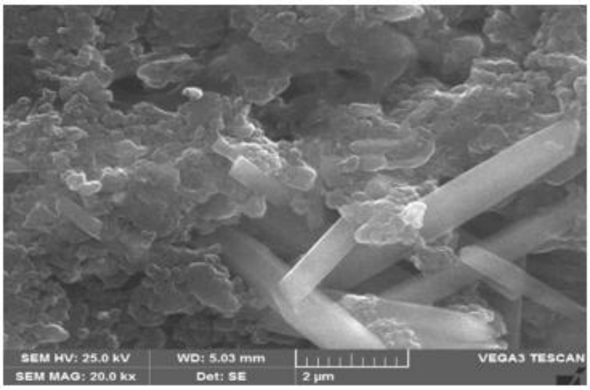

Figure 2 (b): PANI - SnO2 composites

Figure 2 (a) shows that scanning electron micrograph (SEM) image of pure PANI. It is found to have highly agglomerated chainlike structure. the average size was calculated by using linear intercept formula and it is found to be 30nm.

Figure 2 (b) shows the higher resolution SEM image of pure SnO_2 and it is seen to be spongy and nano rods like structure. the average grain size was found to be 30nm. The grains are found to be well interconnected with each other which indicate that they have enough binding energy to combine with neighbors' grains or molecules.

3.3 Electrical Studies

3.3.1 AC Conductivity

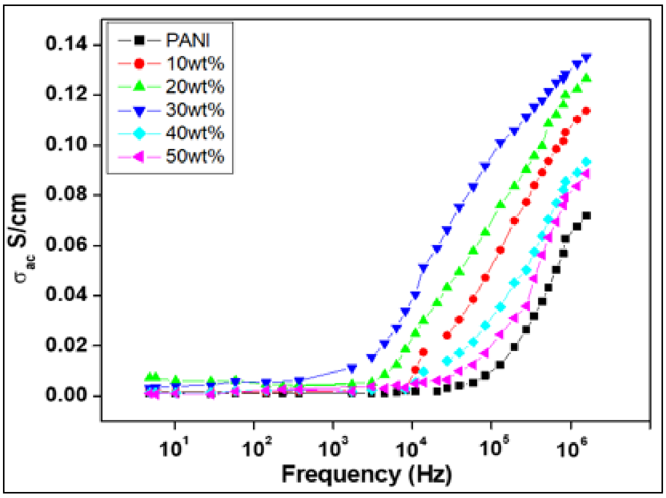

Figure 4: The plot of AC Conductivity with frequency for PANI/SnO_2 nanocomposites

The A. C conductivity as a function of frequency for Pure PANI and for the nanocomposites of PANI - SnO_2 of different weight percentages at room temperature are shown in the Figure 4. The electrical property of AC conductivity (σ) as a function of frequency has been determined, using dielectric data using the following equation.

$$\sigma = \varepsilon' \varepsilon_{\circ} \omega tan\delta$$

Where ε_0 is the permittivity of free space = 8.85 10^{-12} Fm^{-1}, ω is the angular frequency and ε' is the dielectric constant.

From Figure 4, it is observed that in all the cases, σ (AC conductivity) remains constant up to 1000 Hz afterwards it increased for higher frequencies. The nanocomposite of PANI - SnO2 at 30 wt. % shows high conductivity due to interfacial polarization. But for the other wt. % of PANI - SnO2 conductivity value is low because of dipole polarization. This behavior of the nanocomposites may be due to the variation in the distribution of SnO_2 nanoparticles in PANI.

3.3.2 Gas sensor

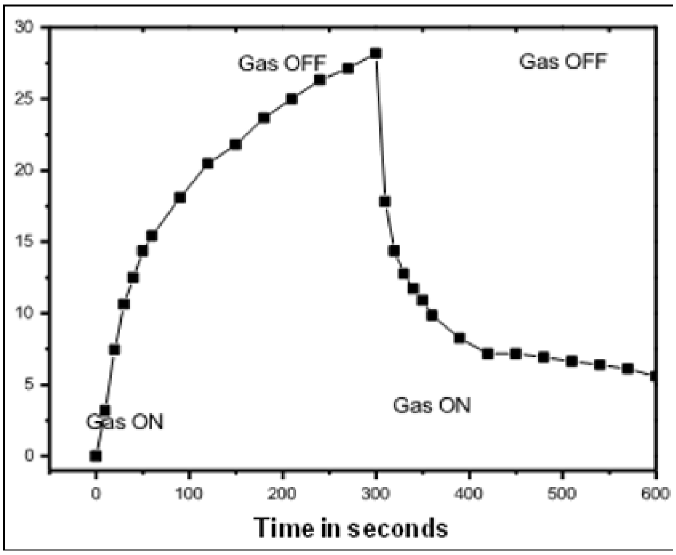

Figure 5: Gas response of PANI/SnO_2 nanocomposites

Figure 5 Shows the variation of resistance as function of time of PANI - SnO_2 nanocomposites. It is observed from the above figure PANI - SnO_2 nanocomposites shows highest responses at 280 seconds when 100 ppm of H2S gas is injected into gas chamber, it reaches a saturation value, the chamber is open and fresh air was injected, there after gradually decreases. Therefore, response time was around

Sensing parameters	PANI/SnO2 nanocomposite
Response temperature	303K
Response time (sec)	11

4. Conclusion

PANI - SnO_2 were successfully synthesized by in - situ polymerization method. The XRD pattern confirms size and crystallite of nanocomposites. The average crystalline size of the PANI were estimated 30nm by XRD technique. The SEM image of PANI and PANI - SnO2 shows nano rods like structure. DC electrical conductivity of pure PANI and PANI - SnO2 composites was carried out from 320K temperature to 450K and increases with increase in the temperature indicates semiconductor behavior of PANI sample. From AC conductivity it is observed that, the conductivity of all composites is higher than that of pure PANI and highest for 30wt% of PANI - SnO_2 nanocomposites and response of PANI - SnO2 nanocomposites medium for employing 100 ppm H_2S gas at room temperature.

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