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Article

Electrical Conductivity and Organic Vapour Sensing Properties of Novel Nanocomposite Comprising Polypyrrole, Tin Oxide Nanoparticles and Single-Walled Carbon Nanotubes

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ABSTRACT

In this study, a novel ternary nanocomposite comprising polypyrrole (PPy), tin oxide (SnO_2) nanoparticles and single-walled carbon nanotubes (SWCNTs) has been synthesized by the chemical oxidative polymerization method in an aqueous medium using anhydrous ferric chloride as an oxidant. The successful preparation of PPy/SnO_2/SWCNTs nanocomposite was established by several advanced characterization techniques such as Fourier transform infrared spectroscopy, X-ray diffraction, scanning electron microscopy and thermogravimetric analysis. The initial direct current electrical conductivity of PPy/SnO_2/SWCNTs was calculated by a four-in-line probe device and found to be 2.53 Scm^{-1} at room temperature. At the same time, PPy/SnO_2/SWCNTs also exhibited exceptional conductivity retention ability under the isothermal as well as cyclic ageing conditions. The vapour sensing performance of the PPy/SnO_2/SWCNTs based sensor-pellet was examined for some organic compounds such as ethanol, methanol, acetone, toluene, benzene, chloroform and n-hexane. The percent sensing response of the sensor was found to be 80.1%, 70.0%, 45.2%, 20.0%, 14.9%, 9.4%, and 6.1% toward ethanol, methanol, acetone, toluene, benzene, chloroform, and n-hexane, respectively.

Key words: Polypyrrole, Tin oxide nanoparticles, Single-walled carbon nanotubes, Nanocomposite, Sensing, Electrical conductivity.

1. INTRODUCTION

There has been a rapid development in the area of conducting polymers in the past few decades. They have been found to show immense potential for developing gas/vapour sensors as their electrical conductivity can be easily altered at ambient temperatures upon exposure to reductive or oxidative molecules of the analyte gas [1-3]. The conducting polymers being studied nowadays mainly are polypyrrole (PPy), polyaniline, polythiophene, polyacetylene, poly(phenylene vinylene) and poly(3,4-ethylenedioxythiophene) [1-8]. In their un-doped state, they are usually semiconductors or insulators. They find application in a variety of energy and electrical device applications as they possess unique tunable electrical properties, are easy to synthesize as well as are structurally diverse and flexible [4].

Among the aforementioned polymers, PPy has been used wisely as it is environmentally stable, economical and has high electrical conductivity as well as superior adsorption property [7-9]. Studies have revealed that these properties can be improved to a great extent by doping PPy with inorganic nanoparticles such as SiC, MoS_2, tin oxide (SnO_2), and CeO_2. which is carried out by several approaches such as hydrothermal method, sol-gel, solvothermal, co-precipitation, and microwave technologies [6,7,10-12].

Carbon nanotubes (CNTs), discovered by Iijima in 1991 [13], possess distinct geometry, large surface area, brilliant electronic, mechanical and structural properties and thus have been used in a variety of applications in fields such as nano-electronics, gas/vapour sensors and field emission as well as energy storage devices [14,15]. Multi-walled CNTs (MWCNTs) and single-walled CNTs (SWCNTs) have attracted considerable interest for fabricating gas sensors and a number of published reports are already available on the fabrication of gas/vapour sensing devices employing nanocomposites of PPy and CNTs [16,17].

SnO_2, a typical n-type semiconductor possesses a wide band gap of 3.7 eV and has high thermal stability, opaque, sensitive as well as reflecting, and also displays low photocatalytic activity [18,19]. It is commonly employed as a vital functional material for fabricating gas sensors, optoelectronic devices, transparent conductive electrodes and catalyst supports [20-23].

Hieu *et al.* [22] synthesized a highly sensitive PPy/SWCNTs nanocomposite based thin film sensor for detecting NH_3 at room temperature using the spin coating and chemical polymerization method. The effects of the thickness of the film, annealing temperature and SWCNT content were studied in order to optimize the sensing performance of the sensor. The thin film sensor showed a response of 26% and 276% at 10 and 800 ppm concentrations of ammonia, respectively. The response and recovery time was calculated to be 22 s and 38 s, respectively. Ayad and Suhail [23] synthesized a

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nanocomposite of PPy and functionalized SWCNTs and employed it for sensing NO_2 using the electrochemical polymerization method. The response of this film based sensor toward NO_2 gas was evaluated by monitoring the variation in the electrical resistance at several temperatures (20, 50, 100, 150, and 200°C). The nanocomposite exhibited greater sensitivity than that of pure PPy.

Du *et al.* [24] synthesized a bead necklace shaped structured nanocomposite in which singly stranded single-walled CNT were impregnated in several PPy/phenylalanine (PA), PPy@PA, nanoparticles by the simple emulsion polymerization technique and employed this nanocomposite for selectively sensing ammonia vapours at room temperature. At ambient temperature, the sensor exhibited a high sensitivity of 100 ppb and its performance was unaffected even under the effect of humid conditions (0–70%) relative humidity. The effective sensitivity was 0.1 ppm as well as the reaction time of the ammonia gas was about 500 s and the recovery time was found to be 600 s.

Hamouma *et al.* [25] synthesized cheap and conducting nanocomposites (Pap@CNT-NH_2@PPy) by sonochemically polymerizing pyrrole on paper strips of cellulose which were decorated with aminophenyl-modified multiwalled CNTs (CNT-NH_2). This composite served as a highly efficient ammonia sensor in the concentration range of 0.005-0.05 ppm and showed a brilliant sensing response of 525% to 0.1 ppm level of NH_3 at ambient temperatures and was also found to be stable for a long duration of time. The limit of detection was 0.04 ppb. The calculated response time was 138 s and 465 s, respectively. Scandurra *et al.* [26] synthesized an electrochemical device which served as a H_2O_2 sensor based on nanocomposites of Gold/Nafion blends with PPy/MWCNTs. The sensitivity of the developed sensor was found to be 1.47 $\mu A\ \mu M^{-1} cm^{-2}$ and the response time was calculated to be 80 s.

Kaur and Kumar [27] synthesised nanowires using PPy SnO_2 nanocomposites which were employed for sensing of ammonia vapours at room temperature. The composite showed maximum sensitivity of 26% at room temperature which was greater than that showed by PPy nanowires (18%) at 100 ppm of ammonia. Jun *et al.* [28] used the single nozzle electrospinning method for synthesizing a gas sensor based on PPy-coated SnO_2 tube-in-tube structure. This structure was highly selective and sensitive (0.05 ppb) towards sensing dimethyl methylphosphonate (DMMP). They used a mass flow controller for exposing the electrode of the sensor to vapours of DMMP (0.01 ppb to 100 ppm) and the resistance values were recorded in real time.

Hsieh *et al.* [29] synthesized PPy/SnO_2/graphene nanoribbon (GNR) ternary nanocomposites by *in situ* chemical oxidative polymerization. The response value of the PPy/SnO_2/GNR sensor comprising 3-wt% of SnO_2 nanoparticles toward 1 ppm of NH_3 was found to be 92.7, which was three times greater than that exhibited by a pure PPy sensor. The fabricated composite sensor was capable of sensitively and selectively detecting NH_3 vapours between 0.6 and 2.0 ppm concentrations at room temperature. Bano *et al.* [30] synthesized nanoparticles of SnO_2 by co-precipitating along with PPy-chitin matrix by using the chemical oxidative polymerisation method. The synthesized composite showed enhanced photocatalytic activity toward RhB dyes under visible light which increased upon increasing the SnO_2 content. The electrocatalytic properties of the nanocomposites were also studied by cyclic voltammetry for detecting methanol (0.001 M to 0.5 M) at a scan rate of 10 mV/s.

Husain *et al.* [31] synthesized nanocomposites of PPy/SnO_2 by the *in-situ* polymerization methods. The prepared composite was studied for its direct current (DC) conductivity as a function of temperature in the temperature range 30–190°C and it was seen that there was an increase in the conductivity upon increasing the concentration of SnO_2. Among all the prepared nanocomposites, the one comprising 30 wt % PPy/SnO_2 showed the highest sensitivity and conductivity. A novel PPy/ZnO/SWCNTs nanocomposite was synthesized by Husain *et al.* [17] and employed for ammonia vapour sensing at room temperature. The fabricated sensor-pellet showed excellent sensitivity, reversibility and selectivity towards ammonia with a detection limit of 50 ppm.

To the best knowledge of authors, there are no reports on synthesis, characterization or application of PPy/SnO_2/SWCNTs nanocomposite. Thus, inspired by unique properties of PPy, SnO_2 and SWCNTs, we have synthesized a novel nanocomposite (PPy/SnO_2/SWCNTs) comprising all three materials. The as-synthesized PPy/SnO_2/SWCNTs was characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM) and thermogravimetric analysis (TGA) techniques. PPy/SnO_2/SWCNTs was converted into a pellet for the investigation of electrical and vapour sensing properties by a four-in-line probe conductivity measuring device.

2. EXPERIMENTAL

2.1. Materials

Pyrrole (99%) (Sigma-Aldrich), SnO_2 nanoparticles average particle size 30–50 nm and SWCNTs (Platonic Nanotech Pvt. Ltd., Jharkhand, India), anhydrous ferric chloride ($FeCl_3$) (Fischer Scientific, India) were used as received. Double-distilled water was used in synthesis and other experiments.

2.2. Synthesis of PPy/SnO_2/SWCNTs Nanocomposite

PPy/SnO_2/SWCNTs nanocomposite was synthesized employing the *in situ* oxidative polymerization method in the aqueous state, where $FeCl_3$ was used as the oxidant [11,16,17]. The first step in preparing the PPy/SnO_2/SWCNTs was adding 100 mg of SWCNTs to 100 ml doubly distilled water. Then this mixture was agitated for 40 min. In the next step, to this mixture 400 mg SnO_2 nanoparticles were added and then this resultant mixture was ultrasonicated for 90 min. Then to this suspension comprising SWCNTs and SnO_2, 2.0 ml (0.029 mol) of pyrrole was added and this mixture was kept for sonication for 4 h. $FeCl_3$ solution was prepared by adding 4.9 g (0.03 mol) of it to 100 ml doubly distilled water. Then into the suspension containing pyrrole, SnO_2 and SWCNTs, dropwise addition of $FeCl_3$ solution was done with continuous stirring. After 10 h, a black coloured slurry was obtained which was washed thoroughly using double distilled water and methanol, followed by filtration. The filtered nanocomposite was kept for drying in an air oven at 80°C for 20 h. Finally, dried material was converted into very fine powder for the characterization and preparation of pellet.

2.3. Characterization and Instrumentation

The bonding in the molecular structure of PPy/SnO_2/SWCNTs was studied using FTIR spectroscopy and the spectra was recorded in the range 400–4000 cm^{-1} employing a Perkin Elmer −1725 instrument. The SEM study was performed by a JSM-6150 (JEOL, Japan). The amorphous character and the crystal structure of the samples were examined by a XRD Bruker D8 diffractometer with CuKα radiation at 1.540 Å within the range of $5° \leq 2\theta \leq 80°$ operating at a voltage of 40 kV. The TGA of the sample was performed by a Shimadzu 60H in the temperature range 30–800°C. The electrical conductivity and sensing experiments were conducted employing a four-in-line probe instrument attached with an oven which was PID controlled and manufactured by Scientific Equipment, Roorkee, India. The following equation was used in calculating the DC electrical conductivity:

$$\sigma = \left(ln2\left[2S/W\right]\right)/\left(2\pi S\left[V/I\right]\right) \quad (1)$$

where I, V, W, S and σ represented the current (A), voltage (V), the thickness of the pellet (cm), probe spacing (cm) and electrical conductivity (Scm^{-1}), respectively [32-38].

For carrying out the conductivity and sensing experiments pellets were used, which were made by a hydraulic pressure machine operating at 65 kN pressure for 80 s. In order to prepare the pellets, 250 mg of material was used.

Isothermal ageing experiments were carried out by heating the pellet at 50°C, 70°C, 90°C, 110°C and 130°C in a PID controlled air-oven and conductivity was recorded at a particular temperature at an interval of 5 min. While cyclic ageing experiments were carried out by recording the conductivity at particular temperature (50°C, 70°C, 90°C, 110°C and 130°C) for five successive cycles [32-35].

3. RESULTS AND DISCUSSION

3.1. FTIR Studies

The FTIR spectrum of PPy/SnO_2/SWCNTs is shown in Figure 1. All the characteristic peaks of PPy [3,7,8,11,16,17] and one characteristic peaks SnO_2 [33] were observed in the spectrum. The peak at 3405.67 cm^{-1} is attributed to the N-H stretching vibrations of PPy. The peaks at 2920.18 cm^{-1} and 2851.72 cm^{-1} are related to the =C-H stretching vibrations. The peak at 1576.52 cm^{-1} is attributed to C=C stretching vibrations. The peak at 1463.22 cm^{-1} indicates about C-C stretching and the peaks that appeared at 1315.21 cm^{-1} and 1218.31 cm^{-1} are due to the C=N bending and C-N vibrations, respectively. The =C-H bending vibration is appeared at 1044.74 cm^{-1}, while the peak at 795.49 cm^{-1} is related to the stretching vibrations of C-H bonds. The peak at 935.31 cm^{-1} indicates about =C-N^{+}-C stretching vibration. This peak at 935.31 cm^{-1} confirms that pyrrole was effectively oxidized to PPy and doped by $FeCl_3$ into positively charged species that act as charge carriers, i.e. polarons/bipolarons. At the same time, the peak at 660.21 cm^{-1} indicates about Sn-O-Sn symmetric and anti-symmetric vibrational mode of SnO_2 [33].

3.2. XRD Studies

The XRD spectrum of PPy/SnO_2/SWCNTs is shown in Figure 2. The characteristic peaks of SnO_2 were detected at 2θ=26.66°, 33.74°, 37.76°, 51.68°, 54.40°, 57.70°, 62.54°, 63.96°, 65.68°, 71.76° and 78.52° which correspond to the (110), (101), (200), (211), (220), (002), (310), (112), (301), (202) and (321) crystal planes, respectively [16,33]. While, the peaks observed at 2θ=24.04° and 41.02° indicate about the (002) and (100) planes of SWCNTs, respectively [39].

3.3. SEM Studies

The surface morphology of PPy/SnO_2/SWCNTs was studied by SEM. The SEM image of PPy/SnO_2/SWCNTs is shown in Figure 3. The tube-like structures observed in the SEM image tell about the presence of SWCNTs. The presence of SnO_2 can be observed in the round shape structures. Most importantly, no free SnO_2 or SWCNTs were seen, which confirm that pyrrole was successfully and thoroughly polymerized on the surface of SnO_2 and SWCNTs or we can also say that SnO_2 and SWCNTs were successfully incorporated into the PPy matrix.

3.4. TGA

The TGA curve of PPy/SnO_2/SWCNTs is shown in Figure 4. The first weight loss at about 90°C is owing to loss of water in form of moisture [11,17]. While second and third weight losses at about 300°C and 600°C are related to the removal of dopants and degradation polymer matrix [11,17]. The total weight loss was found to be about 55.69% which is much lower than pristine PPy (92.65%) reported by Husain *et al.* [11]. Thus, the incorporation of SnO_2 and SWCNTs significantly increased the thermal stability of PPy/SnO_2/SWCNTs. The greater

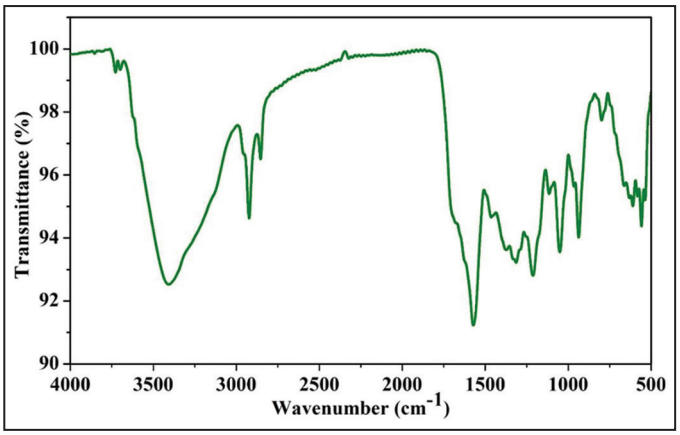

Figure 1: The FTIR spectrum of PPy/SnO_2/SWCNTs. FTIR: Fourier transform infrared spectroscopy, PPy: Polypyrrole, SnO_2: Tin oxide, SWCNTs: Single-walled carbon nanotubes.

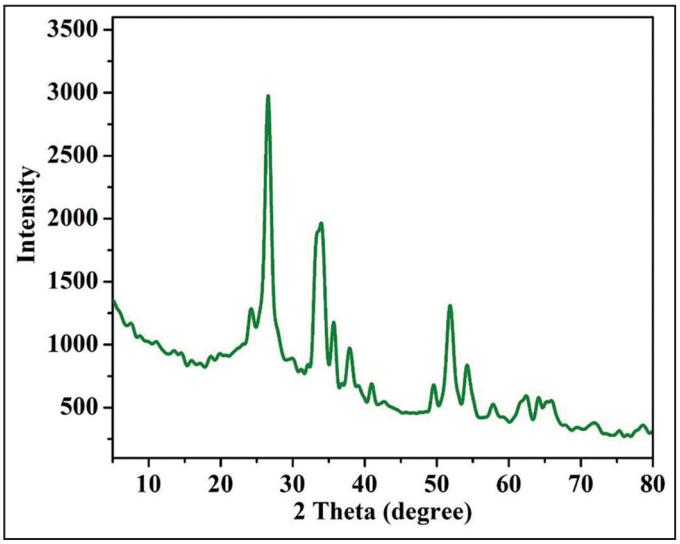

Figure 2: The XRD spectrum of PPy/SnO_2/SWCNTs. XRD: X-ray diffraction, PPy: Polypyrrole, SnO_2: Tin oxide, SWCNTs: Single-walled carbon nanotubes.

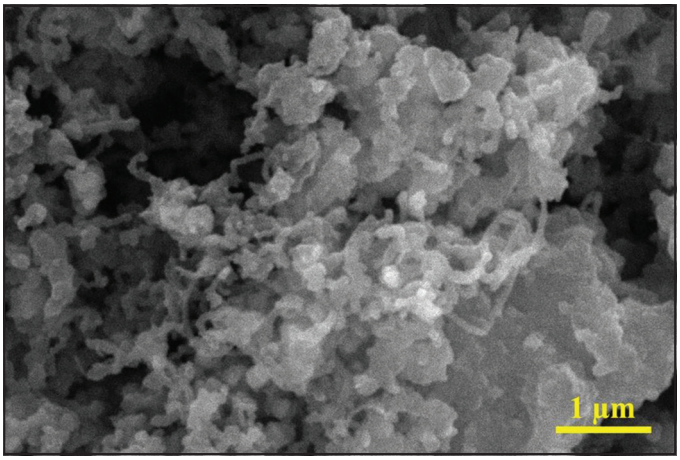

Figure 3: The SEM image of PPy/SnO_2/SWCNTs. SEM: Scanning electron microscopy, PPy: Polypyrrole, SnO_2: Tin oxide, SWCNTs: Single-walled carbon nanotubes.

thermal stability is the reason of greater conductivity retention ability of PPy/SnO_2/SWCNTs at higher temperatures [3,11].

3.5. Electrical Conductivity Studies

The electrical conductivity of conducting polymers depends on the number as well as the mobility of charge carriers. The polarons and bipolarons which are very similar to the holes in the case of semiconductors, act as the charge carriers. The electrical properties are significantly affected by any type of interaction that can increase or decrease the quantity as well as mobility of charge carriers. Therefore, electrical properties are generally controlled by both type and amount of dopants along as well as fillers [1-3,11,33-36].

The conductivity studies were carried out in terms of initial conductivity at room temperature along with conductivity retention properties under the isothermal as well as cyclic ageing conditions. The initial conductivity of PPy/SnO_2/SWCNTs was found to be 2.53 Scm^{-1} at room temperature. While the initial conductivity of pristine PPy was found in the range of 0.152 Scm^{-1} as reported by Husain *et al.* [11]. Sultan *et al.* [3] also reported initial conductivity of pristine PPy which was 0.466 Scm^{-1}.

The much improved electrical conductivity of PPy/SnO_2/SWCNTs than pristine PPy may be due to; the vast surface area provided by SWCNTs and SnO_2 nanoparticle on which polymerization of pyrrole occurred. As a consequence, an extended π-conjugated system of PPy chains has been created through which charge carriers of PPy move freely. Furthermore, there may be some synergistic/electronic interactions of PPy chains with SWCNTs and SnO_2 which might increase the number of charge carriers of PPy. SWCNTs also can provide its charge carriers of very high mobility which also may reinforced with the charge carriers of PPy. Thus, significantly increased charge carriers density and reduction in hopping/tunnelling distance between metallic regions cause significant boost in the electrical conductivity of PPy/SnO_2/SWCNTs [11,35,38].

3.5.1. DC electrical conductivity retention under isothermal ageing condition

The thermal stability of PPy/SnO_2/SWCNTs was determined by DC electrical conductivity retention under isothermal aging conditions. We used the following equation to calculate relative electrical conductivity ($\sigma_{r,t}$) at a particular temperature:

$$\sigma_{r,t} = \frac{\sigma_t}{\sigma_o} \quad (2)$$

Where σ_t and σ_o denote the DC electrical conductivity (Scm^{-1}) at time t and zero, respectively [2,11,32-36].

Pristine conducting polymers are good semiconductor at room temperature. However, at higher temperature, their electrical conductivity drop significantly even totally lost sometimes due to degradation of polymer chains. On the other hand, nanocomposites of conducting polymer are very good semiconductor/conductor even at higher temperatures because of greater thermal stability imparted by nanofillers, better polymer chain alignment and boost in the π-conjugated system provided by synergistic/electronic interaction working at molecular levels between polymer chains and filler inorganic nanoparticles.

Husain *et al.* [11] reported that pristine PPy showed decent conductivity retention ability only at 50°C and 70°C. At higher temperatures (90°C, 110°C and 130°C), conductivity decreased significantly because of the removal of moisture, loss of dopant, and degradation of PPy chains.

However, PPy/SnO_2/SWCNTs showed excellent conductivity retention ability at 50°C, 70°C, 90°C, 110°C and 130°C. Furthermore, the electrical conductivity of PPy/SnO_2/SWCNTs increased with increasing temperature indicating very good semiconductor such as behavior (Figure 5).

3.5.2 DC Electrical Conductivity Retention under Cyclic Ageing Condition

The thermal stability of PPy/SnO_2/SWCNTs in terms of DC electrical conductivity retention was also investigated under cyclic ageing conditions. We used the following equation to calculate relative electrical conductivity (σ_r):

$$\sigma_r = \frac{\sigma_r}{\sigma_{50}} \quad (3)$$

Where σ_T and σ_{50} correspond to the DC electrical conductivity at temperature T (°C) and 50°C, respectively [2,11,32-36].

Husain *et al.* [11] reported that pristine PPy showed boost in conductivity with respect to increasing temperature for first cycle only. After first cycle, the conductivity of PPy was not retained and decreased drastically due to loss of dopants and degradation of PPy

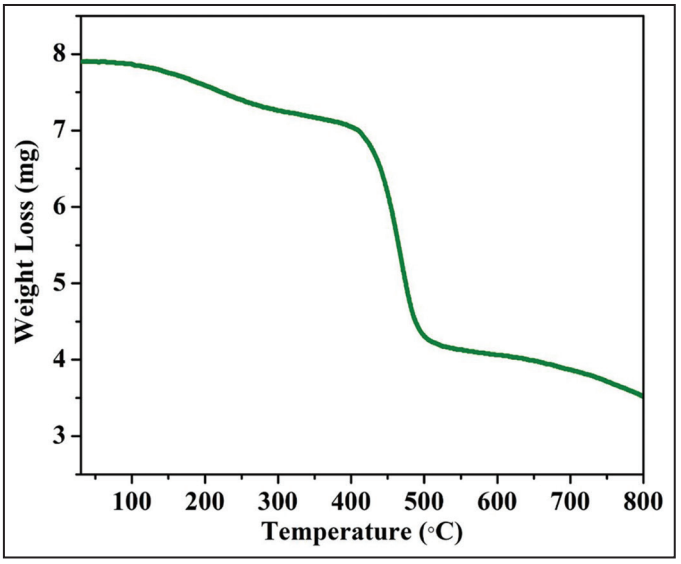

Figure 4: The TGA curve of PPy/SnO_2/SWCNTs. TGA: Thermogravimetric analysis, PPy: Polypyrrole, SnO_2: Tin oxide, SWCNTs: Single-walled carbon nanotubes.

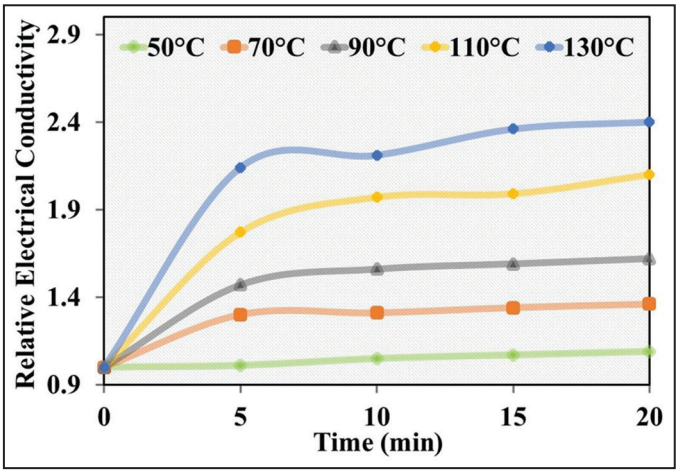

Figure 5: Relative electrical conductivity of PPy/SnO_2/SWCNTs under isothermal aging condition. PPy: Polypyrrole, SnO_2: Tin oxide, SWCNTs: Single-walled carbon nanotubes.

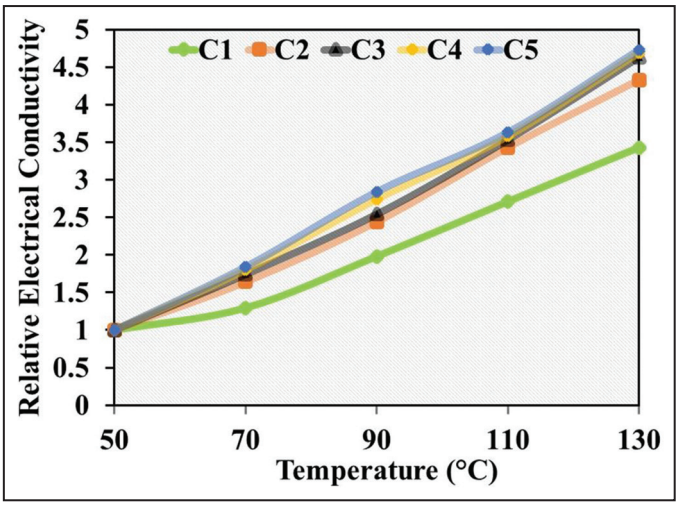

Figure 6: Relative electrical conductivity of PPy/SnO_2/SWCNTs under cyclic ageing condition. PPy: Polypyrrole, SnO_2: Tin oxide, SWCNTs: Single-walled carbon nanotubes.

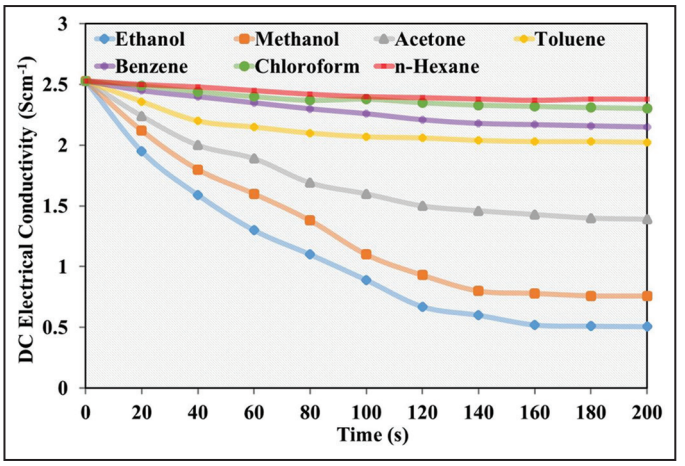

Figure 7: The change in conductivity of PPy/SnO_2/SWCNTs sensor in different organic vapours with respect to time. PPy: Polypyrrole, SnO_2: Tin oxide, SWCNTs: Single-walled carbon nanotubes.

chains. However, PPy/SnO_2/SWCNTs showed boost in conductivity with respect to increasing temperature because of greater mobility of charge carriers at greater temperatures (Figure 6). However, the boost in the conductivity in first cycle was lower than the next three cycles. Most importantly, boost in the conductivity with respect to increasing temperatures exhibited similar patterns for second, third, fourth, and fifth cycles showing very good conductivity retention ability of PPy/SnO_2/SWCNTs.

Thus, PPy/SnO_2/SWCNTs can be a potential candidate as a semiconductor in various application due to high initial electrical conductivity along with excellent conductivity retention properties under both isothermal and cyclic ageing conditions.

3.6. Organic Vapour Sensing Studies

The organic vapour sensing performance of PPy/SnO_2/SWCNTs based sensor-pellet was tested against ethanol, methanol, acetone, toluene, benzene, chloroform and n-hexane at room temperature. All the sensing experiments were carried out at normal laboratory conditions. The experimental set-up used in the sensing can be seen elsewhere. First, sensor-pellet was attached with four-in-line probe tightly. After that, probe was kept in a beaker having analyte sample for producing vapours. The sensor was kept in the beaker continuously for 200 s and change in the conductivity of sensor was recorded. The conductivity of the sensor started to drop in the atmosphere of analyte vapours with respect to time (Figure 7).

The % sensing response (S) of the sensors was calculated by the following formula:

$$S = \frac{\Delta\sigma}{\sigma_i} \times 100 \quad (4)$$

Where: σ_i and $\Delta\sigma$ correspond to the initial DC electrical conductivity and change in the conductivity of the sensor in the analyte vapours in 200 s, respectively [2,11,35].

The percent sensing response of the sensor was found to be 80.1%, 70.0%, 45.2%, 20.0%, 14.9%, 9.4%, and 6.1% toward ethanol, methanol, acetone, toluene, benzene, chloroform, and n-hexane, respectively (Figure 8).

Thus, PPy/SnO_2/SWCNTs can be a promising material for selective detection of various organic compounds tested here due to different sensing response toward different organic vapours.

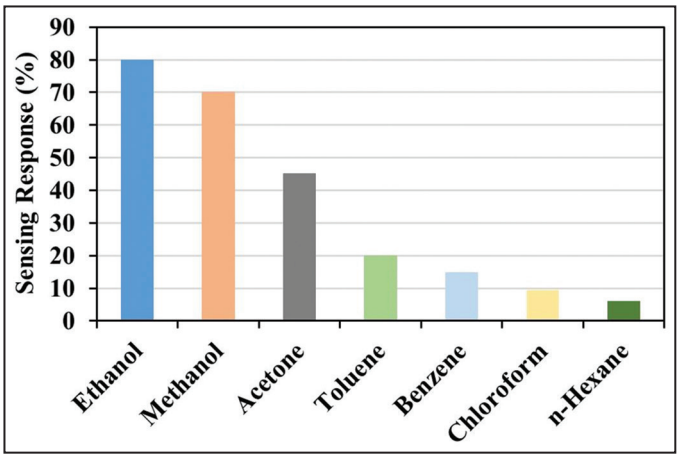

Figure 8: The sensing response (%) of PPy/SnO_2/SWCNTs sensor towards different analyte vapours at room temperature. PPy: Polypyrrole, SnO_2: Tin oxide, SWCNTs: Single-walled carbon nanotubes.

4. CONCLUSIONS

Herein, we have synthesized a novel ternary nanocomposite comprising PPy, SnO_2 nanoparticles and SWCNTs by the chemical oxidative polymerization method in an aqueous medium using anhydrous $FeCl_3$ as an oxidant. The as-prepared material was successfully characterized by FTIR, XRD, SEM and TGA. PPy/SnO_2/SWCNTs showed excellent electrical conductivity retention properties under both the under the isothermal as well as cyclic ageing conditions besides high initial DC electrical conductivity of 2.53 Scm^{-1} at room temperature. The organic vapour sensing properties of the PPy/SnO_2/SWCNTs based sensor-pellet was tested against some organic compounds such as ethanol, methanol, acetone, toluene, benzene, chloroform, and n-hexane. The sensing response of the sensor was found to be 80.1%, 70.0%, 45.2%, 20.0%, 14.9%, 9.4%, and 6.1% toward ethanol, methanol, acetone, toluene, benzene, chloroform, and n-hexane at room temperature, respectively.

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7. CONFLICTS OF INTEREST

There are no conflicts of interest to declare.

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