**Synthesis and Characterization of Polypyrrole-Antimony (III) Oxide Hybrid Polymer Nanocomposites**

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Abstract-The PPy-Sb₂O₃ nanocomposites with Sb₂O₃ (< 250 nm) nanoparticles various weight percents were prepared by mechanical mixing. Fourier transform infrared (FTIR) spectroscopy, ultraviolet-visible (UV–Vis) spectroscopy, X-ray diffraction (XRD), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), scanning electron microscopy (SEM), and energy dispersive X-ray analysis spectroscopy (EDAX) were used to characterize the PPy-Sb₂O₃ nanocomposites. The FTIR results indicated that there are some interactions between PPy and Sb₂O₃ nanoparticles. Such an interaction is likely caused by the formation of the coordinate bonding between the lone pair electron of atom in PPy chain with orbit of Sb atom of Sb₂O₃, indicating a reduction in the strength of PPy-Sb₂O₃ interactions as the wt % increases, which may lead to the broader size distribution of Sb₂O₃ nanoparticles dispersed in nanocomposites.

The UV-vis results of PPy-Sb₂O₃ nanocomposites interactions is significantly increased by increasing the Sb₂O₃ wt%, leading to reduce the energy level interval of benzenoid ring and hence result in a red shift. The XRD result indicates that PPy has been successfully anchored on the surface of Sb₂O₃ nPs through the mechanical mixing method. The morphology and elemental composition analysis were characterized by using SEM and EDAX. This result indicates high interaction between PPy and metal oxides.

1. INTRODUCTION

Hybrid inorganic-organic nanocomposite materials have attracted more and more attention due to creating new materials which combine different functions and characteristics of individual materials. Different inorganic materials including carbon nanotubes, metal, metal oxides and nanosheets have been investigated in polymer matrices [1]. Nanocomposite materials have attracted a lot of interest due to their probable commercial exploitation as sensors, batteries, toners in copying machines, quantum electronic devices, smart windows and materials for electromagnetic shielding, etc. Nanocomposite materials made from nanoparticles of oxides and conducting polymers are the most interesting and challenging areas of research in recent times [2]. The conducting polymers, such as polyiophene, polypyrrole (PPy) and polyaniline have been exhaustively studied due to their outstanding mechanical and electrical properties, which afford applications in actuators, sensors and electrochromic devices [3]. Among the conducting polymers, PPy has attracted considerable attention because it is easy synthesis, it has relatively good quality environmental stability and its surface charge characteristics are can be customized by changing the dopant species into the material during the synthesis [4,5]. In the present work, we report the fabrication of conductive PPy-Sb₂O₃ nanocomposites using mechanical mixing method. The chemical structures of the PPy-Sb₂O₃ nanocomposites are characterized by Fourier transform infrared (FT-IR) spectroscopy and optical parameters are by using UV-vis characterization. The thermal stability of the PPy-Sb₂O₃ nanocomposites is performed by thermogravimetric analysis (TGA) and differential scanning calorimetric (DSC). Scanning electron microscope (SEM) is used to characterize the dispersion of Sb₂O₃ nPs and the morphology of the PPy-Sb₂O₃ nanocomposites. The effects of the Sb₂O₃ nPs on the crystallization structures of the PPy are also studied. In this present work, the inorganic-organic hybrid nanocomposite containing polypyrrole as the organic part and antimony (III) oxide as the inorganic part have been used for studying structural, optical and thermal properties. Such types of nanocomposite have shown to possess small grain size and high stability. To the best of our knowledge, this is the first...
ever attempt made to synthesis and investe of these nanocomposites with special properties.

2. EXPERIMENTAL

2.1. Materials

All of the chemical reagents used in this experiment were A.R. grade. The monomer pyrrole (PPy) and Dodecylbenzene Sulfonic acid (DBSNa) as dopant was purchased from Aldrich Chemical and purified by distillation under reduced pressure, stored in refrigerator before use. Antimony (III) oxide nanopowder, <250 nm nanoparticle (98% purity) from Aldrich Chemical, Ammonium peroxydisulfate (99%, Merck), Ethonal (99% purity, Merck), Acetone (99% purity, Merck) were purchased from Merck chemical. The water used throughout the work is distilled water.

2.2. The preparation of polypyrrole (PPy)

The polypyrrole was synthesized by chemical oxidation polymerization under static condition in a lower temperature. About 900 ml of de ionized water was taken in a flask and an arrangement for mechanical stirring. Dodecylbenzene sulfonic acid solution (DBSNa) as a dopant was dissolved in above 900 ml of deionized water and the solution was well stirred in the flask. Monomer pyrrole was added in the above suspension solution and keeps stirring for 30 min. After 30 min ammonium peroxydisulfate (NH\textsubscript{4}\textsuperscript{2+}) solution was well stirred in the flask. Monomer pyrrole was added drop wise slowly to the good degree of polymerization is achieved the suspension solution was dark black in color. The entire solution mixture was continuously stirred well at 0 -5°C for 24 h. Experimental setup as shown in Fig.1.

2.3. Synthesis of polypyrrole-Sb\textsubscript{2}O\textsubscript{3} nanocomposites

PPy-Sb\textsubscript{2}O\textsubscript{3} nanocomposites were synthesized using different wt% of Sb\textsubscript{2}O\textsubscript{3} with respect to polypyrrole which are referred as PPy-Sb\textsubscript{2}O\textsubscript{3} nanocomposites. Pure PPy was synthesized following the same procedure without Sb\textsubscript{2}O\textsubscript{3} nanoparticles. The molar ratio of polymer (PPy) and metal Oxide Sb\textsubscript{2}O\textsubscript{3} was 1:0.25 to prepare PPy-Sb\textsubscript{2}O\textsubscript{3} (25%) nanocomposites by using mechanical mixing method. Similarly the samples were prepared in the different weight % of Sb\textsubscript{2}O\textsubscript{3} nanoparticles like PPy-Sb\textsubscript{2}O\textsubscript{3} (50%) and PPy-Sb\textsubscript{2}O\textsubscript{3} (100%) by the ratio 1:0.50 and 1:1 respectively.

3. CHARACTERIZATION

FT-IR spectra of the pure PPy, PPy-Sb\textsubscript{2}O\textsubscript{3} nanocomposites, Sb\textsubscript{2}O\textsubscript{3} nPs samples were recorded at room temperature using FT-IR Spectrometer Make: Perkin Elmer; Model: Spectrum RX 1; Range: 400 cm\textsuperscript{-1}-4000 cm\textsuperscript{-1}; Resolution: 4. The sample was prepared in the pellet form by mixing the polymer powder with KBr by the ratio 1:10 and pressing it in the Perkin Elmer hydraulic device using 15 ton pressure.

4. RESULT AND DISCUSSION

4.1 FTIR spectral analysis

The FTIR spectra of pure PPy, PPy-Sb\textsubscript{2}O\textsubscript{3} (25-100%) nanocomposites and pure Sb\textsubscript{2}O\textsubscript{3} nPs are shown in Fig.2. The main transmittance peaks of PPy are appeared at 3402.05 cm\textsuperscript{-1} and 1547.23 cm\textsuperscript{-1} could be corresponded to the N-H stretching vibration and symmetric stretching vibration of C-C bond in the PPy ring, respectively. The band at 1397.53 cm\textsuperscript{-1} is assigned to N-H bending vibration bond. The transmittance...
peaks appeared at 1184.22 cm\(^{-1}\) and 905.96 cm\(^{-1}\) was attributed to the in-plane bending vibration of C-H bond and the C-H out-of-plane bending vibration indicating the polymerization of pyrrole respectively [6]. The FTIR spectrum of the PPy-Sb\(_2\)O\(_3\) (25\%) nanocomposite demonstrated the peaks at 3396.57 cm\(^{-1}\), 1545.98 cm\(^{-1}\), 1381.84 cm\(^{-1}\), 1185.86 cm\(^{-1}\), and 908.86 cm\(^{-1}\) that are considered to arise from pyrrole ring stretching, N-H stretching vibration, C-C symmetric stretching vibration, N-H bending vibration, C-H in plane bending vibration and C-H out-of-plane bending respectively. The transmittance peaks and corresponding stretching vibration of pure PPy, PPy-Sb\(_2\)O\(_3\) nanocomposites was shown in Table.1. These results confirmed the presence of PPy moieties in the nanocomposites. Interestingly, all peak positions shifted towards higher values after Sb ions adsorption. The delocalized \(\pi\) electrons in PPy matrix, which are involved in the skeletal vibration of PPy ring, are affected by the doping ions in the polymer matrix. Different types of dopants in the PPy backbone may disturb the conjugate structure of PPy and this limit the extent of charge delocalization along the polymer chains, leading to red shift. However, as for PPy-Sb\(_2\)O\(_3\) nanocomposites, except the peaks of PPy, the broad band between 500 and 950 cm\(^{-1}\) is attributed to the Sb-O bond, suggesting that the Sb\(_2\)O\(_3\) was embedded in PPy matrix. The results indicated that there are some interactions between PPy and Sb\(_2\)O\(_3\) particles. Such an interaction is likely caused by the formation of the coordinate bonding between the lone pair electron of Sb atom in PPy chain with orbit of Sb atom of Sb\(_2\)O\(_3\), indicating the strength of PPy-Sb\(_2\)O\(_3\) interactions, as the wt \% increases, which may lead to the broader size distribution of Sb\(_2\)O\(_3\) particles dispersed in nanocomposites. Compared to pure PPy, the characteristic peaks of PPy-Sb\(_2\)O\(_3\) nanocomposites slightly shifted to higher wavenumber, indicating the strong interaction at the interface. Besides, the characteristic peaks of PPy-Sb\(_2\)O\(_3\) are well maintained in the nanocomposites, indicating that PPy has been successfully compounded with Sb\(_2\)O\(_3\) without changing chemical composition. Comparing to the corresponding peaks of pure PPy, the peaks of PPy-Sb\(_2\)O\(_3\) shifted towards lower wavenumber. This shifting of absorption bands may be due to the action of hydrogen bonding between the hydroxyl groups on the surface of Sb\(_2\)O\(_3\) nPs and the amine groups in the PPy molecular chains. Similar observations of absorption shifting peaks of PPy-Sb\(_2\)O\(_3\) towards are obtained in lower wavenumber. This result indicates that the PPy-Sb\(_2\)O\(_3\) nanocomposites have been successfully synthesized and the observed shift indicates the interaction between PPy and nPs.

### 4.2. UV-vis absorption spectral analysis

The UV-vis spectra of pure PPy (a), PPy-Sb\(_2\)O\(_3\) (b,c, d) nanocomposites and pure Sb\(_2\)O\(_3\) (e) are shown in Fig.3. The absorption reveals that there is different composition and morphology in ranging Sb\(_2\)O\(_3\) concentration from 25-100\% in PPy-Sb\(_2\)O\(_3\) nanocomposites. However, as the characteristic absorption bands of pure PPy are obtained in the wavelengths range of 250-300 nm, 450-450 nm and 900-1000 nm. UV-vis analysis was also conducted to analyze the PPy nanocomposites (b, c & d).

### Table 1 FTIR data of pure PPy and PPy-Sb\(_2\)O\(_3\) nanocomposites

| Sample Name | N-H stretching vibrations (cm\(^{-1}\)) | C-C ring symmetric stretching vibrations (cm\(^{-1}\)) | N-H bending vibrations (cm\(^{-1}\)) | In-plane C-H bending Vibrations (cm\(^{-1}\)) | Out-plane C-H bending Vibrations (cm\(^{-1}\)) |
|-------------|----------------------------------------|-----------------------------------------------------|-------------------------------------|----------------------------------------|-------------------------------------|
| Pure PPy    | 3402.05                                | 1547.23                                             | 1397.53                             | 1184.22                                | 905.96                              |
| PPy-Sb\(_2\)O\(_3\) (25\%) | 3396.57                                | 1545.98                                             | 1381.84                             | 1185.86                                | 908.86                              |
| PPy-Sb\(_2\)O\(_3\) (50\%) | 3410.94                                | 1548.74                                             | 1387.97                             | 1187.47                                | 911.61                              |
| PPy-Sb\(_2\)O\(_3\) (100\%) | 3373.71                                | 1544.73                                             | 1395.43                             | 1181.12                                | 916.23                              |

Fig.2 FTIR spectra of pure PPy (a) and PPy-Sb\(_2\)O\(_3\) nanocomposites (b, c & d)

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Fig. 3 UV-vis spectra of pure PPy (a), PPy-Sb₂O₃ nanocomposites (b, c, d) and pure Sb₂O₃ nPs

Table 3 Crystallographic parameters of pure PPy, PPy-Sb₂O₃ nanocomposites and pure Sb₂O₃ nPs

| Sample Name          | Wavelength (nm) | Absorption Band | Band gap (eV) |
|----------------------|-----------------|-----------------|---------------|
|                      | Band: 1 | Band: 2 | Band: 3 | Band: 1          | Band: 2          | Band: 3          |
| Pure PPy             | 260   | 473   | 931   | 0.1826          | 0.1581          | 0.2017          | 3.46          |
| PPy-Sb₂O₃ (25%)      | 343   | 360   | 379   | 0.3393          | 0.4155          | 0.3951          | 3.04          |
| PPy-Sb₂O₃ (50%)      | 342   | 359   | 379   | 1.0751          | 1.4434          | 1.3211          | 1.76          |
| PPy-Sb₂O₃ (100%)     | 343   | 359   | 380   | 1.7405          | 1.7130          | 1.8569          | 1.49          |
| Pure Sb₂O₃           | 340   | 359   | 380   | 1.8181          | 2.0666          | 2.5913          | 1.71          |

Among the various cases, the highest band gap energy was obtained for pure PPy and then the band gap was decreased with increasing Sb₂O₃ concentration which is clearly shown in the Table 2. The PPy-Sb₂O₃ interactions is significantly increased by increasing the Sb₂O₃ wt%, leading to reduce the energy level interval of benzenoid ring and hence, result in a red shift. The FTIR spectra of the nanocomposites shown in Fig.3 also support this conclusion. Upon doping PPy exhibits unusual electronic structure due to electron–phonon coupling. Polaron and bipolaron states appear within the band gap, which gives rise to the broad band at wave length 900-1000 nm in the case of pure PPy. Generally, the optical band gap in a semiconductor is determined by plotting absorption coefficients (α) as (αhv)^1/m vs. hv where ‘m’ represents the nature of the transition and hv is the photon energy. Now ‘m’ may have different values, such as 1/2, 2, 3/2 and 3 for allowed direct, allowed indirect, forbidden direct and forbidden indirect transitions respectively.

The optical absorption coefficient (α) near the absorption edge for direct interband transitions is given by the equation (1).

\[
\alpha = A(h\nu - E_g)^{1/m} \tag{1}
\]

where ‘A’ is the absorption constant for a direct transition. For allowed direct transition one can plot (αhv)^2 vs. hv as shown in Fig.4 and extrapolate the linear portion of it to α=0 value to obtain the corresponding band gap.
4.3. X-Ray diffraction studies

Fig. 5 shows the XRD patterns of pure PPy, PPy-Sb$_2$O$_3$ nanocomposites (25-100%) and pure Sb$_2$O$_3$ nPs which is a evidence of crystalline nature of the samples. The XRD pattern of pure PPy shows a broad peak and sharp peak appeared at 23.04$^\circ$ and 44.36$^\circ$ respectively, which indicates the crystalline nature. XRD curve of PPy shows that the PPy prepared in the absence of Sb$_2$O$_3$ nPs is amorphous in nature. The crystallite sizes of the PPy were estimated from X-ray line broadening using Scherer's formula. It can be seen clearly from the XRD patterns of Sb$_2$O$_3$ nPs, that the Sb$_2$O$_3$ nPs showed a single-phase in nature. There was no secondary phase detected and the high intensity of the peaks revealed the crystalline nature of the as Sb$_2$O$_3$ nPs. Obviously, the diffraction peaks of the Sb$_2$O$_3$ nPs appear in the PPy-Sb$_2$O$_3$ nanocomposites from the Fig. 5 (b,c,d) the intensity of these peaks becomes stronger with increasing the nanoparticle loadings, while the two original peaks of PPy show a reduced intensity at 2$\theta$=23.04 and 44.36$^\circ$. The XRD pattern also confirm the presence of antimony in the PPy-Sb$_2$O$_3$ (25-100%) nanocomposites and pure Sb$_2$O$_3$ the crystallize size as-calculated, where the average crystallize size are 97 nm (25%), 170 nm (50%), 176 nm (100%) and 152 nm (pure Sb$_2$O$_3$). The strain and dislocation density of pure PPy, PPy-Sb$_2$O$_3$ nanocomposites (25-100%), pure Sb$_2$O$_3$ nPs data are seen in Table 3. The parameters are slightly changed with the addition of Sb$_2$O$_3$ nPs. Furthermore, these results revealed the amorphous nature of PPy in the nanocomposites, suggesting that the addition of Sb$_2$O$_3$ nPs retain the crystallization of the PPy molecular chains. This may be because when PPy is adsorbed on the surface of the Sb$_2$O$_3$ nPs. The increasing trend intensity indicating that the Sb$_2$O$_3$ greatly increased due to the adsorption of PPy molecular chains on the surface of the Sb$_2$O$_3$ nPs. In order to study the effect of the addition of Sb$_2$O$_3$ nPs in PPy matrix, a careful analysis of the position of the XRD peak indicates that, there is a shifting in peak's position towards lowering 20 value, but in this case, the crystallinity of Sb$_2$O$_3$ nPs was found to be disturbed in the PPy-Sb$_2$O$_3$ nanocomposites. However, in the present work the crystallinity of Sb$_2$O$_3$ is not disturbed by PPy.
molecular chain on the surface of Sb$_2$O$_3$ nPs as can be seen from Fig. 6. The shifting of the peak’s position clearly indicates that PPy-Sb$_2$O$_3$ nanoparticles are incorporating into the PPy polymer matrix. The broad weak diffraction peak of PPy still exists, but its intensity decreases. This indicates a strong effect of the Sb$_2$O$_3$ nPs on the structures of crystalline of the formed PPy and the interaction between PPy backbone and Sb$_2$O$_3$ nPs.

This result indicates that, PPy has been successfully anchored on the surface of Sb$_2$O$_3$ NPs through the mechanical mixing method. However, the characteristic peak intensities of PPy-Sb$_2$O$_3$ nanocomposite gradually decreased with increasing the weight percentage of Sb$_2$O$_3$, indicating the incorporation of Sb$_2$O$_3$ into the polymer matrix. Previous literature also support that the parent work that the introduction of Sb$_2$O$_3$ will affect the crystalline behavior of PPy [10-12].

Fig. 5 X-Ray diffraction patterns of pure PPy (a), PPy-Sb$_2$O$_3$ nanocomposites (b, c, d) and pure Sb$_2$O$_3$ nPs (e)
3 28.48 0.047 210 3.1314 174 0.0003 3.03*10^-14
4 32.20 0.047 713 2.7776 175 0.0003 3.09*10^-14
5 35.26 0.047 107 2.5433 177 0.0002 3.14*10^-14
6 46.10 0.047 733 1.9673 183 0.0002 3.37*10^-14
7 54.30 0.047 47 1.6880 189 0.0001 3.60*10^-14
8 57.28 0.094 220 1.6071 96 0.0003 9.26*10^-15
9 59.20 0.047 140 1.5595 194 0.0001 3.77*10^-14
10 64.38 0.047 53 1.4459 199 0.0001 3.98*10^-14
11 68.90 0.047 87 1.3617 203 0.0001 4.19*10^-14
12 74.10 0.071 183 1.2784 140 0.0001 1.96*10^-14
13 76.32 0.047 123 1.2467 214 0.0001 4.61*10^-14

| Peak No | 20  | FWHM | Intensity | d Spacing Value | Crystallize size (nm) | Strain | Dislocation density |
|---------|-----|------|-----------|-----------------|----------------------|--------|--------------------|
| 1       | 13.80 | 0.047 | 253       | 6.4117          | 170                  | 0.0007 | 2.89*10^-14       |
| 2       | 27.72 | 0.118 | 2957      | 3.2155          | 69                   | 0.0009 | 4.80*10^-15       |
| 3       | 28.42 | 0.071 | 430       | 3.1379          | 115                  | 0.0005 | 1.33*10^-14       |
| 4       | 32.10 | 0.094 | 1037      | 2.7861          | 87                   | 0.0006 | 7.72*10^-15       |
| 5       | 35.06 | 0.047 | 273       | 2.5573          | 177                  | 0.0002 | 3.13*10^-14       |
| 6       | 46.02 | 0.094 | 1130      | 1.9760          | 91                   | 0.0004 | 8.42*10^-15       |
| 7       | 54.56 | 0.047 | 980       | 1.6806          | 190                  | 0.0001 | 3.61*10^-14       |
| 8       | 58.84 | 0.047 | 67        | 1.5681          | 193                  | 0.0001 | 3.76*10^-14       |
| 9       | 59.14 | 0.071 | 180       | 1.5609          | 128                  | 0.0002 | 1.65*10^-14       |
| 10      | 64.10 | 0.071 | 90        | 1.4516          | 131                  | 0.0002 | 1.74*10^-14       |
| 11      | 67.22 | 0.047 | 70        | 1.3916          | 202                  | 0.0001 | 4.11*10^-14       |
| 12      | 74.04 | 0.047 | 263       | 1.2793          | 211                  | 0.0001 | 4.47*10^-14       |
| 13      | 76.24 | 0.047 | 103       | 1.2478          | 214                  | 0.0001 | 4.61*10^-14       |

Table 3 Crystallographic parameters of pure PPy, PPy-Sb2O3 nanocomposites and pure Sb2O3 nPs

4.4. Thermogravimetric analysis

The thermogravimetric analysis of pure PPy, PPy-Sb2O3 (25-100%) nanocomposites and pure Sb2O3 nPs is shown in Fig.6. To investigate the weight loss of the as-synthesized pure PPy, PPy-Sb2O3 (25-100%) nanocomposites and pure Sb2O3 nPs samples, the thermogravimetric analysis has been carried out in a nitrogen atmosphere. In order to see the effect of temperature on the thermal behavior of the polymer thermogravimetric analysis of PPy-Sb2O3 nanocomposites has been carried out from 25-500 °C temperature. To investigate the thermal properties and the interaction between PPy and Sb2O3, TGA analysis has been carried out through decomposition curve. From the Fig.6 (a) pure PPy undergoes two-step decompositions are observed. The first one is appeared at 110 °C which is due to the removal of adsorbed water resulting with a weight loss of 53.99%. The second step of decomposition starts from 200 °C and goes up to 450 °C with about 26.31% weight loss. Finally the residual mass and residual temperature of pure PPy is 19.70 and 497.8 °C. Degradation of PPy-Sb2O3 (25%) nanocomposite takes place in five steps. The first three steps of weight loss observed at 200 °C is due to the removal of adsorbed water and the remaining second step (between 200 °C and 450 °C) of weight loss is due to the breakdown of the polymer backbone in the nanocomposites as shown in Fig. 7 (b).

Fig.6 TGA spectra of pure PPy (a), PPy-Sb2O3 nanocomposites (b,c,d) and pure Sb2O3 nPs (e)

| Pure PPy | Mass Change | Mass | Residual Mass | Residual Temp |
|----------|-------------|------|---------------|--------------|
| Stage: 1 | -53.99      | 19.70| 497.80        |
| Stage: 2 | -26.31      | -    | -             |
| Stage: 3 | -           | -    | -             |
| Stage: 4 | -           | -    | -             |
| Stage: 5 | -           | -    | -             |

| PPy-Sb2O3 (25%) | Mass Change | Mass | Residual Mass | Residual Temp |
|-----------------|-------------|------|---------------|--------------|
| Stage: 1        | -25.36      | 19.70| 497.80        |              |
| Stage: 2        | -16.31      | -    | -             |              |
| Stage: 3        | -           | -    | -             |              |
| Stage: 4        | -           | -    | -             |              |
| Stage: 5        | -           | -    | -             |              |
4.5. Differential scanning calorimetric analysis

Differential scanning calorimetric spectrum of pure PPy, PPy-Sb$_2$O$_3$ (25-100%) nanocomposites and pure Sb$_2$O$_3$ nPs were shown in Fig.8. Fig. 7 (a) shows the DSC of pure PPy have broad endothermic peak appeared at around 370.3 °C, this peak reveals the removal of water molecules from the pure PPy molecules. DSC spectrum of pure PPy have sharp exothermic peaks appeared, exothermic peak was shown at about 95.4 °C, which was the complex peak, area of this peak is 756.5 J/g; the onset and endset temperature of the complex peak is 65.2 °C, 110.3 °C respectively, this is presumably due to the polymer decomposition [15].
The DSC spectrum of PPy-Sb$_2$O$_3$ (25-100%) nanocomposites is shown in Fig. 7 (b-d). The exothermic peaks appeared in the PPy-Sb$_2$O$_3$ spectrum which named the complex peaks. From PPy-Sb$_2$O$_3$ (25-100%) nanocomposites spectrum which have complex peaks and the area of this complex peaks are 5.481 J/g, 17.150 J/g, and 94.200 J/g. To compare these nanocomposites, the area of the complex peaks is increased with increasing the Sb$_2$O$_3$ concentration. The peaks indicating that, the polymer decomposition was found to be present in all ratios (25-100%) of nanocomposites. The peaks indicating that the polymer decomposition was found to be present in PPy-Sb$_2$O$_3$ (25-100%), but that was clearly absent in pure Sb$_2$O$_3$ nPs samples. Despite the degradation of PPy-Sb$_2$O$_3$ (25-100%) nanocomposites samples indicating the gradual enhancement of thermal stability of the polymer chain with increasing the amount of Sb$_2$O$_3$. The exothermic peak disappeared for pure Sb$_2$O$_3$ sample, indicating strong interaction of the oxide with the polymer chain [16].

4.6. Scanning electron microscopic studies

Scanning electron microscopy (SEM) images of the pure PPy, PPy-Sb$_2$O$_3$ (25-100%) nanocomposites and pure Sb$_2$O$_3$ nPs are shown in Fig. 8 (a-e). The micrographs of pure PPy powder (Fig.8a) show big globular clusters of polymers. The surface morphology of pure PPy changed completely, when it was converted to the nanocomposites with Sb$_2$O$_3$ (Fig.8b-d), which established the interaction of Sb$_2$O$_3$ surface with the polymer chain. The white colour is Sb$_2$O$_3$ nPs and light coloured shell is PPy in the nanocomposites. The prepared nanocomposite exists as relatively loose aggregates of PPy-Sb$_2$O$_3$ with crystallize size of 100–250 nm which is observed from SEM study. The amorphous polypyrrole matrix can restrict the further growth of Sb$_2$O$_3$ nanocrystals and avoid their further aggregation in the chemical reaction process. According to above results, it can be summarized that, the parameter modulation of PPy in presence of Sb$_2$O$_3$ nPs affects not only the final morphology but also the structure of Sb$_2$O$_3$ nPs within the PPy-Sb$_2$O$_3$ nanocomposites.
Succeeded in controlling PPy-Sb$_2$O$_3$ morphology, the investigation was turned to explore, the formation mechanism of the PPy-Sb$_2$O$_3$ morphology through characterizing the intermediates obtained in different reaction stages. The change in the surface morphology has been observed with increasing composition of Sb$_2$O$_3$ (25-100 wt %) in PPy-Sb$_2$O$_3$ nanocomposites. The complex, stringy, interconnected network is a general feature of the morphology of PPy-Sb$_2$O$_3$ nanocomposites. At higher (100 wt %) of nanocomposites, the connected pathway become more and more dense morphology are observed due to excess doping as the PPy-Sb$_2$O$_3$ is approached. At this higher percentage of PPy-Sb$_2$O$_3$ nanocomposites; the morphology appears almost foam like with PPy-Sb$_2$O$_3$ network surrounded by Sb$_2$O$_3$. Thus Sb$_2$O$_3$ provides large conduction island thereby, reducing the conduction path through the nanocomposites [17].

4.7. Energy dispersive X-ray analysis

Fig. 9(a-e) shows the EDAX spectrum of the pure PPy, PPy-Sb$_2$O$_3$ (25-100%) nanocomposites and pure Sb$_2$O$_3$ nPs. The corresponding chemical composition is listed in Table 5. Fig. 9a illustrates the element weight (%) of C, O, and S of pure PPy sample was 69.32%, 24.24% and 6.44% respectively. It is seen that C, O, S and Sb elements are detected in the PPy-Sb$_2$O$_3$ (25-100%) nanocomposites, which indicates that O and Sb-ions have been doped into the PPy matrix successfully. The spectrum of pure PPy, PPy-Sb$_2$O$_3$ (25-100%) nanocomposites and pure Sb$_2$O$_3$ nPs shows the carbon molecules weight % are 69.32%, 40.66%, 30.02%, 17.87% and 14.80% which is element composition changes were obtained for oxygen and sulfur molecules weights, and the weight % of antimony ion are 35.72 %, 49.98 %, 62.05 % and 85.20 %, for this chemical composition increasing trend was appeared. As shown in Fig.9b an element like carbon, sulfur, oxygen and antimony of PPy-Sb$_2$O$_3$ nanocomposite samples which compare due to pure PPy, the element contents of carbon, oxygen and sulfur was decreased.
Antimony alone increased with increasing the concentration of Sb$_2$O$_3$ nanoparticles [18].

The FTIR analysis is carried out for pure PPy, Sb$_2$O$_3$ nPs and PPy-Sb$_2$O$_3$ (25-100%) nanocomposites systematically. The characteristic bands were observed for the corresponding materials. From the results, one can conclude that, the wavenumber region is shifted to higher values after Sb ion absorption. The results indicate that the coordination bond formed between the long pair of electrons of the atom in the PPy chain with the orbit of Sb atom of Sb$_2$O$_3$, indicating the strength of PPy-Sb$_2$O$_3$ nanocomposites have been synthesized successfully and the observed shift which indicates the interaction between PPy and Sb$_2$O$_3$ nPs. UV-visible spectra results indicates that the absorption bands which are correspond to the transition of $\pi-\pi^*$. From the tauc plot, one can conclude that the band gap energy is calculated for each case of materials. Among the materials, the band gap energy (3.46 eV) is obtained for pure PPy. The band gap is decreased with increasing Sb$_2$O$_3$ concentration and this indicates the PPy-Sb$_2$O$_3$ interactions are significantly increased by increasing the Sb$_2$O$_3$ concentration loading to reduce the energy level intervals. X-Ray diffraction studies suggest the crystallographic nature of the materials and from the report, we analyze the XRD patterns of pure PPy, Sb$_2$O$_3$ nPs and PPy-Sb$_2$O$_3$ nanocomposites is in a systematic manner. The amorphous peak of pure PPy was appeared in addition to sharp peak, but the other patterns indicates the crystallinity was greatly improved with the addition of Sb$_2$O$_3$ form the nanocomposites. From the crystallite size calculations, the average crystallite size of PPy-Sb$_2$O$_3$ (25 wt%), PPy-Sb$_2$O$_3$ (50 wt%) and PPy-Sb$_2$O$_3$ (100 wt%) are 97 nm, 170 nm and 176 nm respectively. From this, one can inferred that the lowest average crystallite size is observed for PPy-Sb$_2$O$_3$ (25%). The strain and dislocation density calculation and the data suggest the crystallographic nature and defects of the materials. Thermogravimetric results of the pure PPy, Sb$_2$O$_3$ nPs and PPy-Sb$_2$O$_3$ (25-100%) nanocomposites suggest that thermal behavior and stability of the materials and the number of stages of decomposition may vary depending upon the materials. In this report, the lowest number of stages (2) is observed for pure PPy. The residual mass of PPy-Sb$_2$O$_3$ (25-100 wt%) is increased, when the composition increase from PPy-Sb$_2$O$_3$ (25 wt%) to PPy-Sb$_2$O$_3$ (100 wt%). This is because, the increase of loading amount of Sb$_2$O$_3$ in the matrix of PPy, but the residual temperature is almost constant for all the cases. From the differential scanning calorimetric analysis, one can reveal the stages in which the molecules of various categories eliminating from the surface. The exothermic and endothermic peaks suggests that the polymer decomposition is found in the case of PPy-Sb$_2$O$_3$ nanocomposites. These kinds of analysis help us to estimate the thermal stability of pure PPy, Sb$_2$O$_3$ nPs and PPy-Sb$_2$O$_3$ (25-100%) nanocomposites. In this report, the surface morphological analyses of pure PPy, Sb$_2$O$_3$ nPs and PPy-Sb$_2$O$_3$ (25-100%) nanocomposites are carried out successfully. Actually, the particles are not in spherical in size for all the cases. Instead, the particles are agglomerated initially and the some square shaped particles are also found and also there are some surface modifications, due to the agglomeration of the particles, so that the core shell like structure is formed on the surface of PPy matrix and this can be seen clearly from the morphological data. From the EDAX analysis, the elemental composition of pure PPy, Sb$_2$O$_3$ nPs and PPy-Sb$_2$O$_3$ (25-100 wt%) is estimated clearly. From the increasing trend of antimony, one can inferred the loading amount of Sb$_2$O$_3$ in the matrix of PPy. This kind of analysis helps us to gain more information about the structure and behavior of the materials.

| Sample Name | Weight (%) |
|-------------|------------|
|             | Carbon     | Oxygen | Sulfur | Antimony | Total |
| Pure PPy    | 69.32      | 24.24  | 6.44   | -        | 100   |
| PPy-Sb$_2$O$_3$ (25%) | 40.66      | 19.60  | 4.02   | 35.72    | 100   |
| PPy-Sb$_2$O$_3$ (50%) | 30.02      | 17.35  | 2.65   | 49.98    | 100   |
| PPy-Sb$_2$O$_3$ (100%) | 17.86      | 18.24  | 1.85   | 62.05    | 100   |
| Pure Sb$_2$O$_3$ | 14.80      | -      | -      | 85.20    | 100   |

Table 5: Element analysis data of pure PPy, PPy-Sb$_2$O$_3$ nanocomposites and pure Sb$_2$O$_3$ nPs

The elements contents of PPy-Sb$_2$O$_3$ (25-100%) nanocomposites show a higher Sb contents due to the formation of a large amount of Sb$_2$O$_3$.

5. Conclusion

From the EDAX analysis, the elemental composition of pure PPy, Sb$_2$O$_3$ nPs and PPy-Sb$_2$O$_3$ (25-100 wt%) is estimated clearly. From the increasing trend of antimony, one can inferred the loading amount of Sb$_2$O$_3$ in the matrix of PPy. This kind of analysis helps us to gain more information about the structure and behavior of the materials.
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