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Original Research Article

Mechanical properties of polypyrrole/SnO_2 nanocomposites and its LPG sensing application

Md Shakeel Ahmed[1], Ameena Parveen[2,*], Sriram Manjunath[3]

[1] *Department of Physics, Government First Grade College, Hulsoor, Karnataka 585416, India*
[2] *Department of Physics, Government Degree College, Yadgir, Karnataka 585201, India*
[3] *Department of Physics, Government First Grade College, Bellari, Karnataka 583101, India*
*** Corresponding author:** Ameena Parveen, Aparveen981@gmail.com

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ABSTRACT: Polypyrrole/SnO_2 nanocomposites were created using in-situ polymerization techniques. The nanocomposites were described using scanning electron microscopy (SEM) and transmission electron microscopy (TEM), and their thermal properties were studied using a Differential Scanning Calorimeter (DSC). The DC conductivity of the samples was measured as a function of temperature from 30 ℃ to 1900 ℃, and it was observed that increasing the concentration of tin oxide particles improves conductivity due to polaron hopping and composite chain length extension. The tensile strength of PPy nanocomposites doped in PVA thin film up to 6 wt% indicates 64.2 MPa, which may be related to the homogenous distribution of PPy nanocomposite in PVA. The study reveals that because 50 wt% of the nanocomposites have the highest conductivity and sensitivity, these nanocomposites may be useful in future applications.

KEYWORDS: polypyrrole; scanning electron microscopy; conductivity; sensitivity

1. Introduction

Polypyrrole is a type of conducting polymer that has gained considerable attention and usage in various fields due to its unique properties and applications. This compound has an intriguing structure, exceptional conductivity, and versatility, making it a promising material for use in electronics, energy storage, sensing devices, and even biomedical applications. In this essay, we will discuss the characteristics, synthesis methods, and potential applications of polypyrrole. Polypyrrole is a conjugated polymer consisting of pyrrole monomers linked together through carbon-nitrogen bonds, forming a long chain structure. The presence of alternating single and double bonds along the backbone of the polymer results in the delocalization of electrons, leading to its electrical conductivity. Additionally, the polymer can be doped with various salts or acids to enhance its conductivity further. Polypyrrole also exhibits good mechanical flexibility and stability, making it ideal for various applications[1–4]. Due to their unusual mix of electrical and optical properties and processing advantages, Alan J. Heeger and Hideki Shirakawa conducted extensive research on the new generation of “synthetic metals” in 1976[5]. The electrical conductivity of conjugated polymers is achieved through the delocalization of the electrons, which allows charge mobility along the polymer chain’s backbone. Conducting polymers have been synthesized using an oxidizing or reducing method, as well as chemical or electrochemical doping[6–8].

The synthesis of polypyrrole involves two main methods: chemical and electrochemical polymerization. In the chemical method, oxidative polymerization is carried out using an oxidizing agent, such as ferric chloride, in the presence of pyrrole monomers. This process typically occurs in a solvent or in an aqueous solution, resulting in the formation of a black, insoluble powder. On the other hand, electrochemical polymerization involves the electrodeposition of polypyrrole onto an electrode surface. This method offers better control over the polymerization process, as the reaction occurs at a lower temperature and allows for the preparation of thin films[9–12]. Due to its electrical conductivity, polypyrrole has found extensive use in organic electronics. It serves as a highly efficient electrode material in various electronic devices, such as supercapacitors, batteries, and solar cells. The conductive nature of polypyrrole allows for excellent charge transport, ensuring improved performance in energy storage and conversion devices. Additionally, polypyrrole is also being explored for use in gas sensors, as its conductivity changes in the presence of different gases, making it suitable for detecting toxic or hazardous substances[13].

One of the key advantages of tin oxide is its high sensitivity towards reducing gases such as carbon monoxide and methane. When exposed to these gases, tin oxide undergoes a chemical reaction that results in a change in its electrical conductivity. This change can be measured and used to detect the presence and concentration of the target gas[14]. Furthermore, tin oxide sensors are known for their low cost and ease of fabrication. They can be produced using simple techniques such as screen printing or thin film deposition, making them suitable for large-scale production. Additionally, tin oxide sensors exhibit good stability and long-term reliability, making them suitable for continuous monitoring applications[10]. In recent years, researchers have been exploring ways to enhance the performance of tin oxide sensors by modifying their structure and composition. For example, doping tin oxide with other metal oxides or nanoparticles can improve its selectivity for specific gases or chemicals. Additionally, nanostructuring tin oxide films can increase their surface area, leading to improved sensitivity. To begin with, the synthesis of polypyrrole-tin oxide nanocomposites can be achieved through various methods. One common method involves the chemical deposition of polypyrrole onto the surface of tin oxide nanoparticles. This can be done by oxidative polymerization of pyrrole monomers in the presence of tin oxide nanoparticles, resulting in the formation of a conductive polymer layer on the nanoparticles. Another method involves the in-situ polymerization of pyrrole in the presence of tin oxide nanoparticles, where the nanoparticles serve as a template for the growth of polypyrrole. These synthesis methods can be further modified and optimized to control the size, shape, and composition of the nanocomposites, enabling fine-tuning of their properties[15].

Once synthesized, polypyrrole-tin oxide nanocomposites exhibit several enhanced properties compared to their individual components. Firstly, the incorporation of tin oxide nanoparticles into the polypyrrole matrix enhances the electrical conductivity of the nanocomposites. This is attributed to the high electron mobility and good charge transfer ability of tin oxide nanoparticles, which facilitate efficient charge transport within the nanocomposites. Additionally, the presence of polypyrrole in the nanocomposites provides improved mechanical properties, stability, and processability, making them suitable for various applications[16,17].

2. Experimental

2.1. Synthesis of tin oxide nanoparticles

Analytical Reagent grades of $SnCl_4.5H_2O$ and ethanol were successfully used to make tin oxide nanoparticles. To begin, a transparent sol solution was made by dissolving 3.50 gm. of tin chloride pentahydrate in 100 mL ethanol while swirling dynamically. Drop by drop, 4 mL of aqueous ammonia solution was added to the aforementioned solution under steady stirring. For purification, the gel was filtered and washed with ethanol before being dried for 4 h at 60 °C. The resultant powder was calcined for 2 h at 400 °C, yielding ash-colored tin oxide nanoparticles[18–20].

2.2. Synthesis of PPy-SnO_2 nanocomposites

1M aniline solution dissolved in 100 mL of HCl was placed in a 1000 mL beaker and agitated for 5 min before adding 0.5 g (10 wt%) of SnO_2 nanoparticles and stirring with a magnetic stirrer for around 15 min before adding ammonium per sulphate drop by drop. Even after the ammonium per sulphate was completely added, stirring was continued for another 10 min, and the precipitate was allowed to settle for 30 min–40 min. To remove contaminants, the precipitate was filtered and washed numerous times with distilled water. Finally, the precipitate was rinsed with acetone and allowed to dry at room temperature before being ground for 15 min with a mortar and pestle. In the same manner, nanocomposites with 10, 20, 30, 40, and 50 wt% of SnO_2-doped polypyrrole were prepared[21].

3. Preparations of pellet

The powders of polypyrrole and polypyrrole/SnO_2 nanocomposites generated from the synthesis processes outlined earlier were crushed and finely processed in an agate mortar in the presence of an acetone medium. The powder is then squeezed in a hydraulic press to generate pellets with diameters of 10 mm and thicknesses ranging from 1 mm to 2 mm. To improve connections, the pellets of polypyrrole and its metal oxide nanocomposites are covered with silver paste on both sides of the surfaces for temperature-dependent conductivity and sensor experiments[22].

4. Characterizations

The surface morphology of the prepared nanocomposites was examined in powder using a scanning electron microscopy (SEM) Model-EVO-18 (Special Edison, Zeiss, Germany), a transmission electron microscope (TEM), and a differential scanning calorimetry (DSC) instrument: DSC Q20 V24.10 Build 122. Mechanical properties studies were carried out by a Universal Testing Machine (UTM), and through a GPIB interface controlled by a home-made protocol developed in LabView 7.0, stress-strain data is gathered and saved in a data file. The DC conductivity of these nanocomposites was investigated using a Keithley 6514 electrometer, and the sensing capabilities of these nanocomposites were investigated using a laboratory setup.

5. Results and discussion

5.1. Scanning electron microscopy

The scanning electron microscopic image provides valuable insights into the morphology and structure of these nanocomposites. Nanoparticles of tin oxide show in **Figure 1(a)** that the particles are spherical in shape, industrial, and not aggregated with a size of 68 nm. **Figure 1(b)** shows that the SEM morphologies of 30 wt% nanocomposite are highly agglomerated and irregular in shape, and nanoparticles are embedded within the polypyrrole matrix. As observed in the SEM images in **Figure 1(c,d)**, PPy has a typical cauliflower-like or tumour-like form. The presence of SnO_2 in the nanocomposites with a uniform spherical shape provides space factors for PPy orderly growth, resulting in a unique PPy morphology. The particle size of the PPy/SnO_2 nanocomposite is significantly smaller than that of PPy. As a result, the SEM data show that the reactions are astonishingly successful for PPy functionalization[23–25].

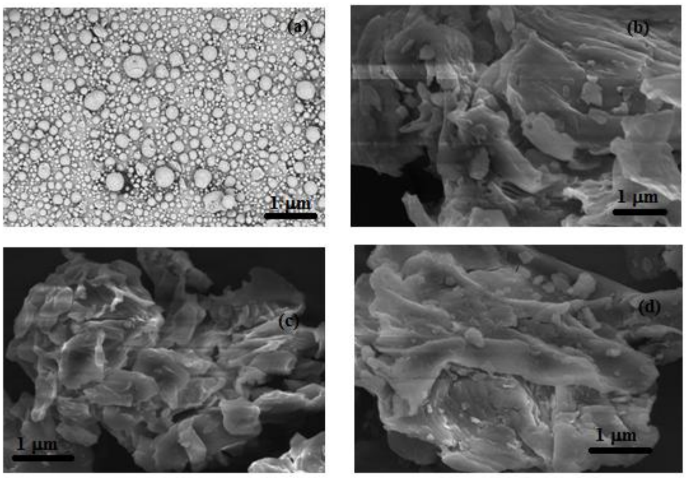

Figure 1. (a) shows a scanning electronic micrograph image of pure polypyrrole and **(b–d)** PPy/tin oxide nanocomposites of 10, 30, and wt%.

5.2. Differential scanning calorimetry

Figure 2 shows the DSC curve of the PPy and PPy/dopant samples with a broad typical endothermic drop, indicating the glass transition temperature of polypyrrole at 99 °C. The shape of the curve suggests that water loss overlaps with the polymer Tg. The incorporation of nanoparticles into polypyrrole matrices has shown significant improvements in their electrical conductivity and mechanical strength. However, understanding the thermal behaviour of these nanocomposites is crucial for their successful integration into practical devices. The absence of any shoulder or melting peak beyond this region shows that the PPy molecules are amorphous and have less sequence. In the case of polypyrrole nanocomposites, DSC can provide valuable insights into their thermal stability and compatibility with other materials. The presence of nanoparticles may affect the polymer's crystallinity or induce changes in its molecular structure. These alterations can be detected through shifts in peak temperatures or changes in enthalpy values observed in DSC curves.

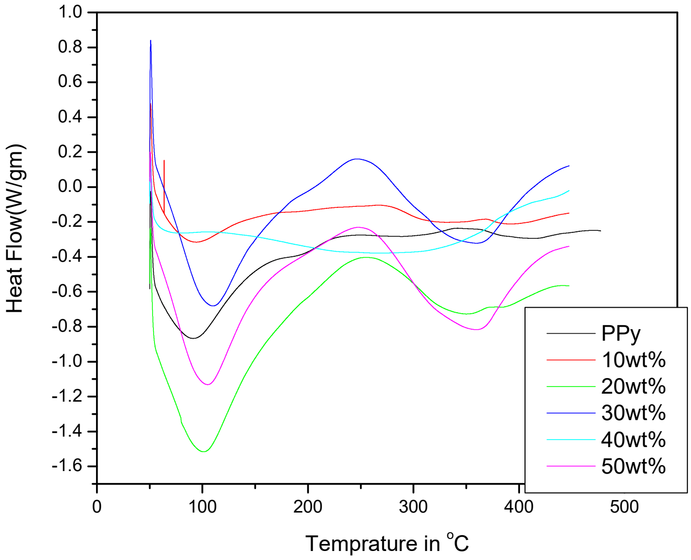

Figure 2. DSC curve against temperature of polypyrrole and PPy/SnO_2 nanocomposites.

Moreover, DSC can also help optimize processing conditions for fabricating polypyrrole nanocomposites by providing information about curing temperatures or reaction kinetics during synthesis. At high temperatures around 100 °C, the DSC curve of the 20 wt% of PPy/SnO_2 nanocomposite trace displays a severe fall. This sharpness means that the crystalline is better. It also features a few more inflection points, one from 330 °C to 360 °C due to the melting of the PPy chain and the other at 360 °C to 400 °C due to phase transition[26–29].

6. DC conductivity

The DC conductivity of polypyrrole tin oxide nanocomposites can be attributed to several factors. Firstly, the presence of tin oxide nanoparticles within the polypyrrole matrix provides additional pathways for electron conduction. These nanoparticles act as conductive bridges, allowing for the efficient transfer of electrons through the material. As a result, the overall conductivity of the nanocomposite increases.

Secondly, the morphology of the nanocomposite plays a crucial role in determining its DC conductivity. The dispersion of tin oxide nanoparticles within the polypyrrole matrix should be uniform to ensure a continuous network of conducting pathways. Achieving this uniform dispersion is a challenging task, as nanoparticles tend to agglomerate, leading to poor conductivity. However, various synthesis techniques, such as in-situ polymerization and electrochemical deposition, have been employed to overcome this challenge and enhance the DC conductivity of the nanocomposite. The temperature-dependent DC conductivity of polypyrrole and polypyrrole/SnO_2 nanocomposites measured at the temperature range of 30 °C to 190 °C, as shown in **Figure 3**. It is observed that the conductivity of the polypyrrole nanocomposites increases with an increase in temperature. The conductivity values of the composites studied are clearly higher than those of pure PPy. The conductivity increases continuously with temperature up to a transition point, indicating semiconductor properties. Temperature increases conductivity due to the migration of charge carriers (polarans) from one localized state to another. The conductivity of 50 wt% of all nanocomposites is higher, indicating that conductivity is determined not only by ion mobility (SnO_2) but also by charge carrier hopping. The conductivity is proportional to it and follows an expression of the type[30,31]:

$$\sigma (T) = \sigma_0 \exp [-T_0/T)^{1/4}]$$

where σ is the conductivity, T is the applied temperature, and σ_0 is the conductivity at the characteristic initial temperature T_0.

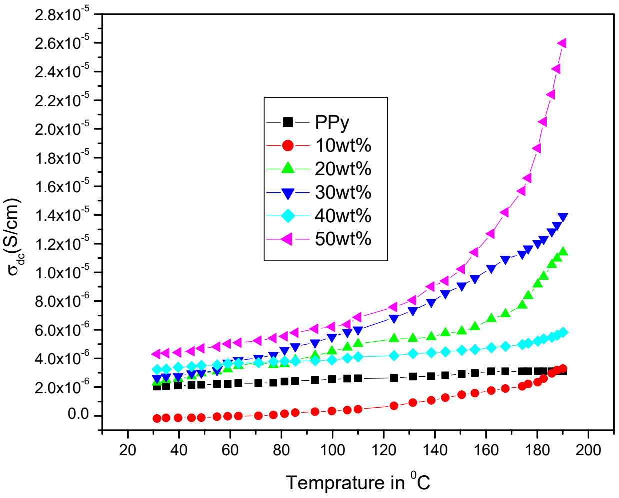

Figure 3. DC conductivity against temperature of PPy/SnO_2 nanocomposites.

Figure 4 shows the tensile strength of PPy nanocomposites doped in PVA in different weight percentages to fabricate the electrically conducting sensor encapsulation thin films. It is found that the tensile strength decreases with an increase in PPy nanocomposites doped in PVA thin film up to 6 wt%, which shows 64.2 MPa. This may be due to the homogeneous distribution of PPy nanocomposite in PVA and well-ordered network formation. Further, it is also noted that the elongation of the PVA is significantly affected by the agglomeration of PPy nanocomposite in the PVA thin film when doped above 6 wt%. The maximum elongation of Ppy nanocomposite in PVA by 6 wt% may be due to the limit of critical concentration[32–34].

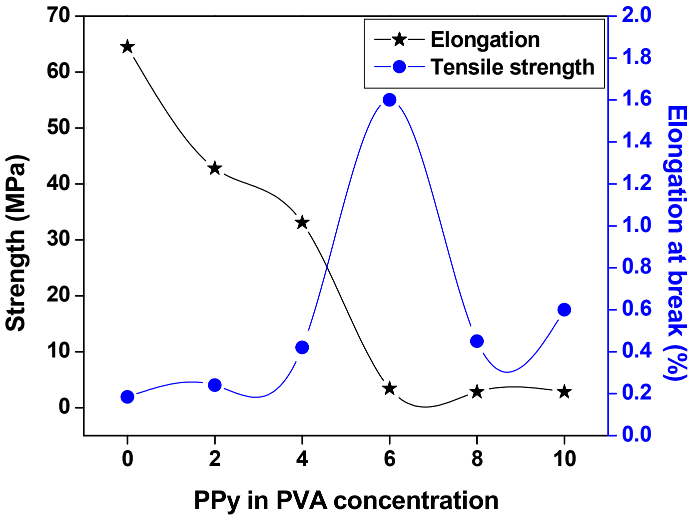

Figure 4. shows the mechanical properties of PPy nanocomposites in PVA thin films.

7. Sensing study

Figure 5 depicts the sensitivity of a Polypyrrole/SnO_2 nanocomposite for LPG sensing. The LPG sensing performance of the Ppy-SnO_2 nanocomposites is primarily attributed to the synergistic effect between the two materials. When exposed to LPG, the surface of the Ppy-SnO_2 nanocomposites undergoes chemical reactions and structural changes, resulting in a change in their electrical properties. These changes can be conveniently measured through electrical resistance variations, making Ppy-SnO_2 nanocomposites an ideal material for LPG sensing applications[35,36]. The polymer recovers to its original size after the gas is removed, restoring the conducting channels. Surface reactions are a possible mechanism for detecting LPG gas by LPG. The surface modification of Ppy-SnO_2 nanocomposites has been reported to enhance the gas-sensing capability of the material. The tin oxides act as a surface coating, increasing the surface area and providing additional active sites for gas adsorption. This leads to a higher sensitivity and faster response time of the Ppy-SnO_2 nanocomposites towards LPG. When compared to other nanocomposites, 30 wt% exhibits a high sensitivity of 86.5%. Surface reactions, consequent charge transfer processes with the underlying SnO_2, and the transport method through the sensing material and shape of the sensing layer all influence overall conduction in a sensor element[37–39].

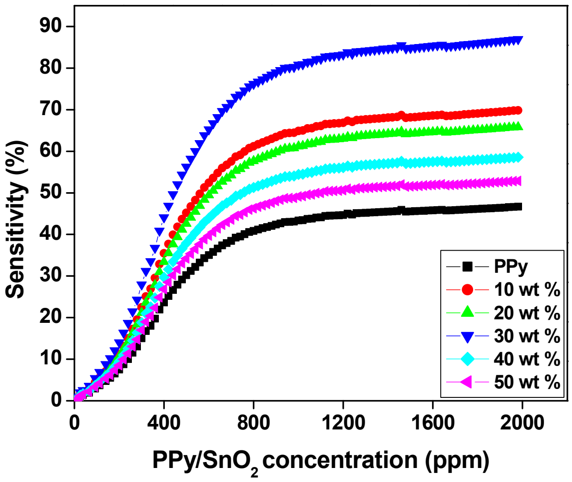

Figure 5. shows the sensitivity of PPy/SnO_2 nanocomposites.

8. Conclusion

The fabrication of conducting PPy/SnO_2 nanocomposites utilizing an in-situ chemical polymerization technique is described in this paper. These composites' conductivity and sensing characteristics were investigated. The influence of SnO_2 concentration on conductivity qualities has been studied. At room temperature, the composites' LPG sensing characteristics were examined, and the better responsiveness of the PPy-SnO_2 nanocomposite was explained in terms of the synergetic interaction of both PPy and SnO_2 particles. It is confirmed that the tensile strength decreases with an increase in PPy nanocomposites doped in PVA thin film up to 6 wt%, which shows 64.2 MPa, which may be due to the homogeneous distribution of PPy nanocomposite in PVA. The development of Ppy-SnO_2 nanocomposites for LPG sensing has emerged as a promising research area. The combination of polypyrrole's excellent electrical conductivity and tin oxide's high sensitivity towards gases has resulted in a material with enhanced gas-sensing properties. Further studies focusing on optimizing the synthesis methods and exploring different dopants or additives are necessary to fully exploit the potential of Ppy-SnO_2 nanocomposites for LPG sensing applications. This research holds great promise for the development of efficient and accurate gas sensors, contributing to the advancement of technology in various industries.

Author contributions

Conceptualization, methodology, writing—original draft preparation, and validation, MSA and AP; formal analysis, SM and AP; investigation, MSA; resources, data curation, and writing—review and editing, SM; visualization, and supervision, AP. All authors have read and agreed to the published version of the manuscript.

Conflict of interest

The authors declare no conflict of interest.

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