

Polypyrrole-silver Nanocomposite: Synthesis and Characterization

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Abstract: Polypyrrole-Silver (PPy-Ag) nanocomposite has been successfully synthesized by the chemical oxidative polymerization of pyrrole with iron (III) chloride as an oxidant, in the presence of a colloidal suspension of silver nanoparticles. Turkevich method (Citrate reduction method) was used for the synthesis of silver nanoparticles (Ag NPs). The silver nanoparticles were characterized by UV-Visible spectroscopy which showed an absorption band at 423 nm confirming the formation of nanoparticles. PPy-Ag nanocomposite was characterized by Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR) and X-ray diffraction (XRD) techniques for morphological and structural confirmations. TEM and SEM images revealed that the silver nanoparticles were well dispersed in the PPy matrix. XRD pattern showed that PPy is amorphous but the presence of the peaks at 2θ values of 38.24° , 44.57° , 64.51° and 78.45° corresponding to a cubic phase of silver, revealed the incorporation of silver nanoparticles in the PPy matrix. A possible formation mechanism of PPy-Ag nanocomposite was also proposed. The electrical conductivity of PPy-Ag nanocomposite was studied using two probe method. The electrical conductivity of the PPy-Ag nanocomposite prepared was found to be 4.657×10^{-2} S/cm, whereas that of pure PPy was found to be 9.85×10^{-3} S/cm at room temperature (303 K). The value of activation energy (E_a) for pure PPy was 0.045 eV while it decreased to 0.034 eV for PPy-Ag nanocomposite. The synthesized nanocomposite powder can be utilized as a potential material for fabrication of gas sensors operating at room temperature. Copyright © 2016 IFSA Publishing, S. L.

Keywords: Polypyrrole, Silver nanoparticles colloid, TEM, FTIR, XRD, Electrical conductivity.

1. Introduction

Polypyrrole (PPy) is the most extensively studied conducting polymer amongst the known conducting polymers because of its high conductivity, improved stability, mechanical properties, and high flexibility in its preparation [1, 2]. PPy has applications in sensors, biosensors, microactuators, polymeric batteries, antielectrostatic coatings, electrochromic devices, electronic devices, functional electrodes,

optical switching devices, and so on [2, 3]. Metal nanoparticles especially gold, silver, platinum, and copper have been the focus of great interest due to their unique electronic, optical, catalytic and thermal properties, as well as their important applications in various fields [4-6]. Conducting polymer-based nanocomposites loaded with metallic nanoparticles provide exciting systems to explore the possibility of constructing device functionality [7]. Hence, metal nanoparticles and conducting polymers have evoked

an interest in a number of researchers to synthesize nanocomposite materials [8-13].

PPy-Ag nanocomposites show superior performance in sensing optoelectronics and catalytic capabilities as compared to those of pure PPy [14, 15]. Therefore, the preparation of PPy-Ag nanocomposites becomes an innovative challenge for researchers. Various methods for synthesis have been investigated for the preparation of these nanocomposites. For example, Jing et al. reported the usage of one-step UV-induced polymerization for the synthesis of PPy-coated Ag nanoparticles (NPs) [16], Chen et al. reported a one-step process to fabricate Ag-PPy Coaxial Nanocables in the presence of PVP, produced by redox reaction between AgNO₃ and Pyrrole [17], Fujii et al. demonstrated a one-step versatile synthetic route for synthesis of PPy-coated Ag nanocomposite particles [18]. Babu et al. prepared polymer-silver nanocomposites modified cotton fabrics. It has been shown that fabrics modified by conducting polymer silver composites provided conductivity and reduced the microbial attack [19].

In the present work, we report a novel synthesis of PPy-Ag nanocomposite. Ag NPs were synthesized by chemical reduction method. The PPy-Ag nanocomposite was successfully synthesized by in situ chemical oxidative polymerization of pyrrole in the presence of a colloidal suspension of Ag NPs. The Ag NPs were characterized by UV-Visible spectroscopy (UV-Vis). The synthesized PPy-Ag nanocomposite was characterized by TEM, SEM, FTIR, and XRD. The electrical conductivity of PPy-Ag nanocomposite was studied using two probe method.

2. Experimental

2.1. Materials and Reagents

Pyrrole (Spectrochem Pvt. Ltd, Mumbai) was double distilled and stored in a refrigerator before use. Ferric Chloride (anhydrous) and trisodium citrate were procured from S. D. Fine Chem. Ltd., Mumbai. Silver Nitrate was purchased from Research Lab Fine Chem. Industries, Mumbai. These chemicals were of AR grade and were used as received. All solutions of reacting materials were prepared in distilled water.

2.2. Synthesis of Polypyrrole

PPy was synthesized by chemical oxidative polymerization technique. 1 M pyrrole solution was prepared using distilled water and cooled in an ice bath (~ 0 °C) with continuous stirring for 30 minutes. The precooled aqueous solution of FeCl₃ (1 M) was added dropwise to the pyrrole solution. The oxidant to monomer ratio was 2.5:1. The polymerization was conducted for a period of 5 h under constant stirring at ~ 0 °C. This mixture was allowed to settle for 24 h

to ensure complete polymerization. The obtained precipitate was filtered under reduced pressure. The product was washed with distilled water successively to remove the unreacted monomer, oxidant, and ferrous and/or ferric contamination and was then dried under vacuum at room temperature to a constant weight.

2.3. Synthesis of Silver Nanoparticles

The silver colloid was prepared by using Turkevich method (citrate reduction method) [20]. In a typical experiment, 50 ml of 1 mM AgNO₃ was heated up to boiling temperature. To this solution, 5 mL of 10 mM trisodium citrate was added dropwise, at about 1 drop per second. During this process, the solutions were mixed vigorously and the boiling temperature was also maintained. Slowly, the colour of the solution turned golden yellow, indicating the formation of Ag NPs [21-23].

2.4. Synthesis of PPy-Ag Nanocomposite

The PPy-Ag nanocomposite was synthesized by in situ chemical oxidative polymerization. In a typical synthesis experiment, 100 ml of Ag NPs colloidal solution was cooled in an ice bath (~ 0 °C) and stirred by a magnetic stirrer for 30 minutes, and 1 ml of pyrrole was then added dropwise to this solution. Until the pyrrole was thoroughly dissolved, the mixture was stirred. The aqueous solution of FeCl₃ (1 M) was precooled, added dropwise to the above solution. The obtained mixture was allowed to react for 5 h under constant stirring. The color of the solution changed from golden yellow to grey and then finally to black. This mixture was allowed to settle for 24 h. The product was filtered under reduced pressure, washed with distilled water until the filtrate became colourless and dried under vacuum at room temperature to a constant weight [24, 25].

2.5. Instruments and Analysis

The UV-Vis absorption spectrum of Ag NPs was recorded by UV-EQ-825 (Equiptronics). The morphological examination of PPy and PPy-Ag nanocomposite was carried out using scanning electron microscope (JEOL, JSM-7600F) at an accelerating voltage of 10 kV. The TEM images were recorded with a JEM-2100F transmission electron microscope with 100 kV of accelerating voltage. The diffraction ring patterns (SAED) were examined in its high resolution (HRTEM) mode. The chemical structure was examined by FTIR spectroscopy (Perkin-Elmer Ltd. system 2000) in the range of 400–4000 cm⁻¹. The XRD pattern was recorded using Panalytical X'Pert (Philips) in the range of 2θ = 20° to 80°.

2.6. Electrical Characterization

The fine powder of PPy and PPy-Ag nanocomposite was pressed into a pellet (diameter 10 mm and thickness 0.6 mm) at 7 ton/cm² pressure. The pellets were then coated with conductive silver paste and dried under vacuum, before taking an electrical measurement. The electrical conductivity was measured by two probe method in the temperature range of 303 K to 343 K. The apparatus used for conductivity measurement using two probes was specially fabricated in our laboratory. Electrical conductivity was measured using the following formula:

$$\sigma = \frac{I}{V} \times \frac{t}{A}, \quad (1)$$

where

σ = conductivity in S/cm;

I / V = slope obtained from the I-V plot;

t = thickness in cm;

A = area in cm².

3. Results and Discussion

3.1. UV-Vis Spectroscopy

The UV-Vis absorption spectrum of a colloidal solution of Ag NPs is shown in Fig. 1.

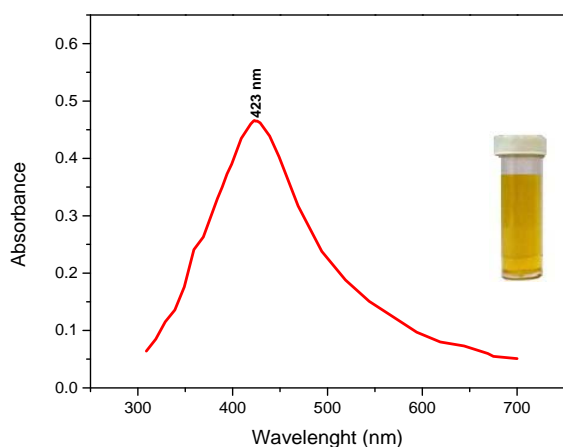


Fig. 1. UV-Vis absorption spectra of colloidal solution of Ag NPs.

The Turkevich method for preparing silver colloids uses trisodium citrate, which serves the dual role of a reductant and a stabilizer. The distinctive golden yellow color of colloidal silver is due to the conduction of electrons on the metal surface which undergo a collective oscillation when excited by light of specific wavelengths. This phenomenon is known as plasmon resonance [5, 27]. The observation of a

broad surface plasmon absorption peak at approximately 423 nm confirms the formation of Ag NPs. [21, 28].

3.2. Morphology

Fig. 2 shows TEM images with different magnifications of PPy-Ag nanocomposite. Ag NPs are in a broad size distribution from 20 to 70 nm with an average size of ~ 40 nm. The TEM features clearly reveal that these particles are non-uniform and are dispersed, which is probably due to clustering of the nanoparticles. The Ag NPs seem to be embedded in the PPy matrix. Moreover, these nanoparticles have a little aggregation in the PPy particles [29].

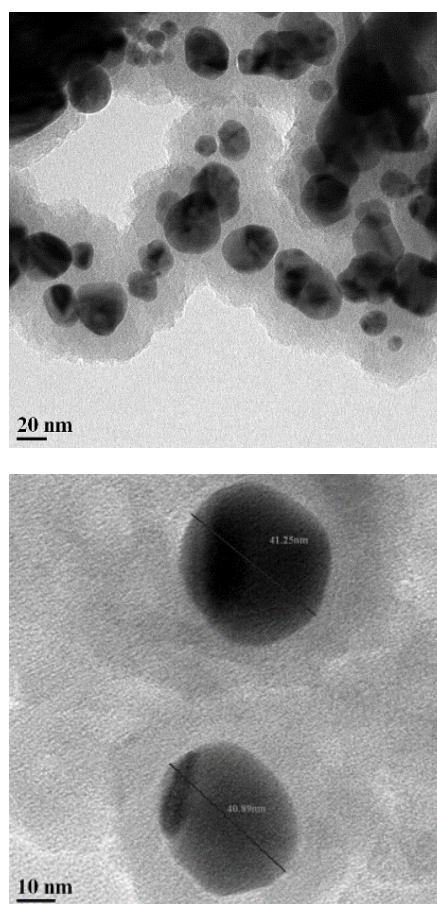


Fig. 2. TEM images of PPy-Ag nanocomposite.

Fig. 3 shows the Selected Area Electron Diffraction (SAED) pattern of the PPy-Ag nanocomposite. The electron diffraction pattern showed characteristic concentric ring-like diffraction pattern associated with silver of crystalline nature. The diffraction rings could be indexed on the basis of the FCC structure of silver. Four strong diffraction rings arise due to reflections from (1 1 1), (2 0 0), (2 2 0) and (3 1 1) lattice planes of silver, respectively [30].

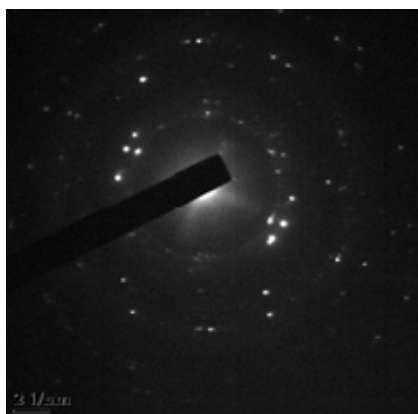


Fig. 3. SAED pattern of the PPy-Ag nanocomposite.

Fig. 4 shows SEM images with different magnifications of PPy-Ag nanocomposite which suggests that the product exhibits a uniform granular morphology and the average grain size is from about 180 nm to 300 nm. Such globules are seemingly growing one over the other to form a continuous structure. [31, 32]. The uniform granular morphology is preferred for gas sensing applications and it promotes adsorption as well as desorption of the gas molecules [33, 34].

It is considered that the PPy-Ag nanocomposite is formed through the process as shown in Fig. 5.

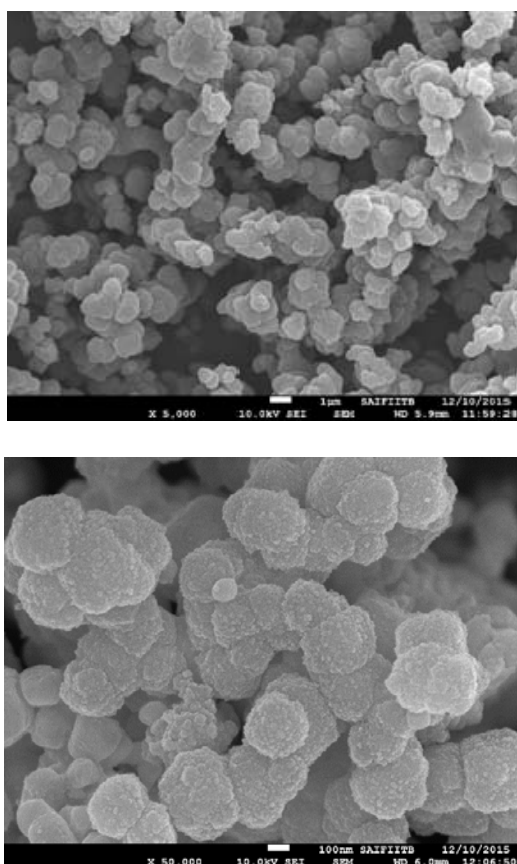


Fig. 4. SEM images of PPy-Ag nanocomposite.

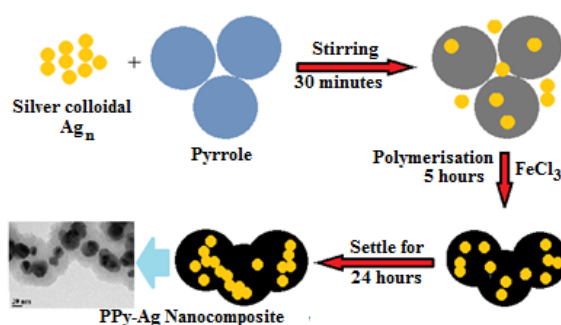


Fig. 5. Mechanism of formation of PPy-Ag nanocomposite.

Firstly, a colloidal solution of nanoparticles (Ag_n) is prepared. (Silver nitrate provides Ag^+ ions that are reduced by sodium. Citrate ions simultaneously act as a reducing agent and a stabilizer.) After the addition of pyrrole in the colloidal solution, the pyrrole molecules are adsorbed onto the citrate-capped Ag NPs through electrostatic interactions. Finally to induce the polymerization of pyrrole, an oxidant is added and the PPy-Ag nanocomposite can be obtained. In this process, the Ag NPs are aggregated inside the PPy [24].

3.3. FTIR Result

Fig. 6 shows the FTIR spectra of the PPy and PPy-Ag nanocomposite. FTIR spectra of PPy shows the main characteristic peak at 675 cm^{-1} corresponding to C-H wagging [35]. The peaks around $725\text{--}1110\text{ cm}^{-1}$ represent the C-H in plane and C-H out plane deformation in PPy units [36]. The characteristic peaks for PPy observed at 1463 cm^{-1} and 1551.33 cm^{-1} , are attributed to the vibration of pyrrole ring. The peak at 1307 cm^{-1} corresponds to C-H deformation [32]. The broad peaks in the range of $3400\text{--}3500\text{ cm}^{-1}$, especially the small peak at 3425 cm^{-1} was ascribed to stretching of N-H and C-H bonds [35, 37]. In Fig. 6 (b) a strong peak at 1383 cm^{-1} may originate from the absorption assignable to doped NO_3^- in PPy, or the interactions at the interface of Ag NPs and PPy [38]. From the spectra obtained, we can also observe that the positions of the peaks of the PPy-Ag nanocomposite are shifted to a higher wavenumber when compared with pure PPy. This may be due to the compound formed between Ppy and silver [24]. It was found that the positions of all peaks were identical in both PPy-Ag nanocomposite and were well matched with the literature available.

3.4. XRD Result

Fig. 7 represents XRD pattern of the synthesized PPy powder. The broad peak was observed at 25.45° . This peak is a characteristic of the amorphous nature of PPy [39, 40] and it is indicative of the short range arrangement chains of PPy [41].

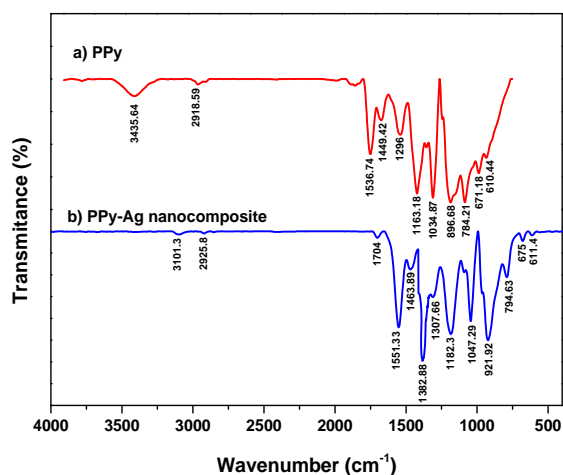


Fig. 6. FTIR Spectra of PPy and PPy-Ag nanocomposite.

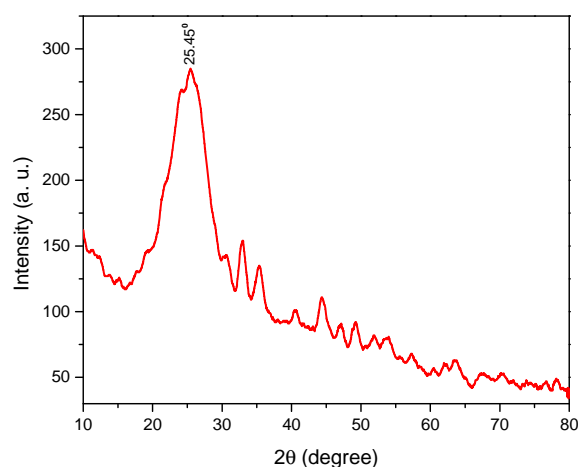


Fig. 7. XRD pattern of pure PPy.

The XRD of PPy-Ag nanocomposite is shown in Fig. 8. The peak observed at 23.34° is due to PPy. All characteristic peaks of the face centered cubic (FCC) structure of Ag, namely (1 1 1), (2 0 0), (2 2 0) and (3 1 1) at $2\theta = 38.24, 44.57, 64.51,$ and 77.45° respectively (JCPDS file no. 04-0783) were observed, which are in agreement with the reported values. This confirms the existence of metallic Ag in the black powder [15, 42]. No other peaks were observed which in turn indicated that no impurities were present which confirmed that the adopted synthesis method gave pure PPy-Ag nanocomposite [25].

The average crystallite size from a sharp peak at 38.24° , Ag (1 1 1) diffraction line for PPy-Ag nanocomposite is estimated by using Scherrer's formula:

$$D = \frac{K\lambda}{\beta \cos \theta}, \quad (2)$$

where D is the average size of the crystallite, K is the shape factor (equals 0.89 for unknown shape), λ is the wavelength of the X-ray radiation, β is the peak

full line width at half of maximum (FWHM) in radian and θ is the diffraction peak position [43]. The average crystallite size of the silver is found to be about 39.67 nm which is consistent with the result of the TEM.

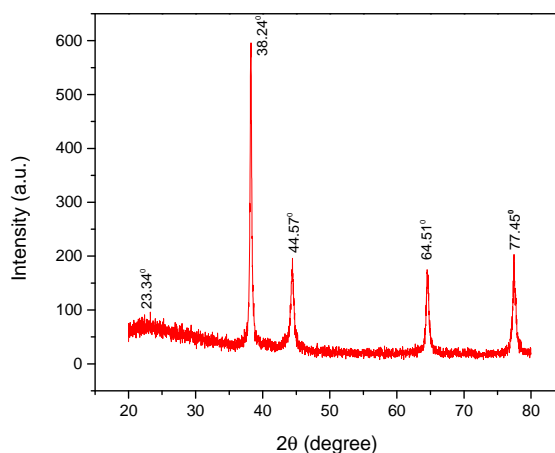


Fig. 8. XRD pattern of PPy-Ag nanocomposite.

3.5. Conductivity Measurement

In order to determine the current conduction mechanism in pure PPy and PPy-Ag nanocomposite, the variation of DC electrical conductivity was studied in the temperature range of 303 K to 343 K. Linear relationship of the I-V curve was found. The electrical conductivity of the PPy-Ag nanocomposite prepared was found to be 4.657×10^{-2} S/cm, whereas that of the pure PPy was found to be 9.85×10^{-3} S/cm at room temperature. The enhancement in conductivity appears due to electronic tunneling through the Ag NPs [44].

Fig. 9 shows DC electrical conductivity versus $1000/T$ of PPy and PPy-Ag nanocomposite at different temperatures. The $\ln \sigma$ versus $1000/T$ plots are found to be linear. This linearity of the plots indicates that the standard band model can be applied for semiconduction in PPy and PPy-Ag nanocomposite [45].

The conductivity-temperature relation is given by Arrhenius equation [44]:

$$\sigma = \sigma_0 \exp\left(-\frac{E_a}{k_B T}\right), \quad (3)$$

where σ is the conductivity, σ_0 is the conductivity at infinite temperature constant, E_a is the activation energy, k_B is the Boltzmann constant, T is the temperature (K). The activation energy has been calculated using equation (3) and the slope of the straight line plot in Fig. 9. The value of activation energy (E_a) for pure PPy is 0.045 eV while it decreases to 0.034 eV for PPy-Ag nanocomposite, confirming electronic tunneling through the Ag NPs.

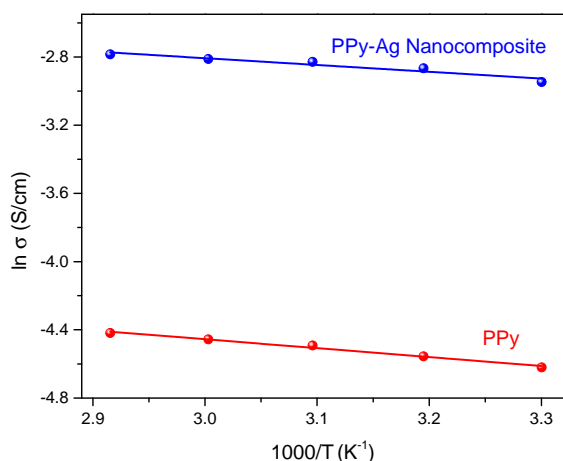


Fig. 9. Variation of DC conductivity with temperature of PPY and PPY-Ag nanocomposite.

4. Conclusion

In this work, the PPY-Ag nanocomposite was successfully synthesized via in situ chemical oxidative polymerization of pyrrole, in the presence of a colloidal suspension of Ag NPs. The results of TEM, SEM, FTIR and XRD measurements indicated that the PPY-Ag nanocomposite was successfully prepared. Ag NPs with an average size of ~ 40 nm were embedded in the PPY matrix. The electrical conductivity of PPY-Ag nanocomposite was studied using the two probe method and was observed to be 5 times higher than that of pure PPY. It was also observed that the activation energy (E_a) decreased from 0.045 eV to 0.034 eV for PPY-Ag nanocomposite, as compared to that of pure PPY. The synthesized nanocomposite can be used as a potential material for fabrication of gas sensors operating at room temperature.

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