Synthesis and Characterization of PPY-Au Nanocomposite

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Abstract— The study involves the synthesis of PPY and PPY based gold nanocomposite (PPY-Au-nanocomposite) by polymerization procedure. The UV-Visible analysis was carried to confirm the presence of gold particles in nanocomposite. FTIR analysis was carried to identify chemical bonds in molecule by producing an infrared absorption spectrum. Size, shape and morphology of the synthesized particle were analyzed by X-ray diffraction and TEM analytic techniques. Average size of gold nanoparticles in PPY-Au-nanocomposite was recorded as ~13.5 nm. Further, conductivity of PPY was compared with finally obtained PPY-Au-nanocomposite. Results showed that addition of gold as metal nanoparticles resulted in high conductivity of PPY-Au-nanocomposite as compare to pure PPY.

Key words: Nanocomposite, PPY

I. INTRODUCTION

Nano-composites comprising conducting polymers and metal nanoparticles have received great attention due to the potential possibilities to create suitable materials for electrocatalysis, chemical sensors, and microelectronic devices [1-3]. These nano-composites are also used in DNA testing and cancer treatment [4-5]. Conducting polymers are well known for their excellent electronic properties with conductivity covering the whole range from insulator to metal while retaining the lightweight, mechanical properties and processing advantages of polymers [1-5]. Among the metal nanoparticle, gold nanoparticles have been extensively studied and are believed to have great potential for applications in optics, catalysis, and other fields [6-12]. Further, Polypyrrole (PPY) is a frequently studied conducting polymer due to its application in sensing and catalysis. Polypyrrole is considered among the most promising conductive polymers due to its stability and ease of conversion between conducting and insulating forms. Different chemical and electrochemical methods are generally used in the synthesis of polypyrrole [3, 13]. Despite many interesting applications, the use of polypyrrole is restricted because difficulty in its processing. Several approaches have been explored to improve the ability to process polypyrrole, including the use of emulsion, inverse emulsion, steric stabilizer, and microemulsion methods. Researchers have reported their work on the synthesis of polypyrrole-metal nanocomposites. The sensing and catalytic abilities of the polypyrrole based metal nanocomposites are reported significantly better than those for polymer alone [13]. The present work discusses the synthesis of polypyrrole based gold nanocomposite (PPY-Au-nanocomposite) by polymerization procedure.

II. MATERIALS AND METHODS

A. Materials

Gold Chloride ($HAuCl_4$) was obtained from Alfa Aesar. Polyvinyl pyrrolidone (PVP) and ferric chloride ($FeCl_3$) were purchased from Lambachemie and Hydrazine hydrate (H_6N_2O) was obtained from RFCL.

B. Synthesis of Gold nanoparticles

Gold nanoparticles were produced by reduction of chloroauric acid ($HAuCl_4$). One gram PVP and 5 ml of $HAuCl_4$ was dissolved in dissolve in 50 ml distilled water. This solution was continuously stirred rapidly and a reducing agent Hydrazine hydrate (H_6N_2O) was added. This causes Au^{3+} ions to be reduced to neutral gold atoms. Gradually, the color of the solution changes from faint yellowish color to grey to purple to deep purple and finally precipitated in wine red color. The fast stirring of solution was responsible for formation of uniform gold nanoparticles.

C. Synthesis of Polypyrrole (PPY)

One milliliter of pyrrole was added via syringe in 100 ml of distilled water stirred solution containing 9.74 g $FeCl_3$ $.6H_2O$ at room temperature. The Fe^{3+}/pyrrole molar ratio was 2.5. The solution was stirred for 24 hrs with magnetic stirrer, which gives rise to the formation of a black precipitates. The resulting black precipitate was filtered in vacuum. The precipitate was washed with copious amount of triply distilled water until the washing was cleared. The polypyrrole so obtained was soft jet black powder, dried in desiccators overnight and again dried in an oven at 25 ^oC.

D. Synthesis of PPY-Au-nanocomposite

The 9.74 g of $FeCl_3$ $.6H_2O$ was added in 100 ml distilled water put in volumetric flask. 10 ml gold solution was added in volumetric flask and finally added 1 ml of polypyrrole via syringe. Stirred the reaction mixture by magnetic stirrer for 24 hrs, the reaction mixture was filtered in vacuum. A black colored precipitate was obtained. Obtained precipitated was washed with distilled water. The material so obtained was first dried in desiccators overnight and then in oven at 25 ^oC. The obtained powder was soft and black in color.

Further, the composite powder was pressed to form pellets of 10 mm diameter and 2 mm thickness by applying pressure of 90MPa in hydraulic press. The pellets of PPY and its nanocomposites were coated with silver paste on either side. These pallets were used for microstructure and electric conductivity analysis. UV-visible study of gold nanoparticles and PPY-Au-nanocomposite particles was conducted to detect the presence of gold particles. FT-IR analysis was carried to identify chemical bonds in molecule by producing an infrared absorption spectrum. X-ray diffraction (XRD) analysis was used to reveal crystallographic structure of Au particles, PPY and PPY-Au nanacomposite. Transmission Electron Microscope (TEM) analysis was used to identify the size of gold nanoparticles in PPY-Au nanoparticles. Further, conductivity of PPY-Au nanoparticles was determined by using two probe method.

III. RESULTS AND DISCUSSIONS

A. UV-Visible Analysis

The UV-Vis spectra of gold particles and PPY-Au-nanocomposite are shown in Fig. 1a–b. Because PPY absorbs wavelengths close to those absorbed by Au, it was difficult to detect the presence of Au nanoparticles with UV-Vis spectroscopy. The absence of absorption peaks corresponding to Au nanoparticles in the resulting nanocomposite can be explained on the basis of strongly acidic environment of the solutions and reduced oscillator strength. This may also be due to overlap of the absorption of polymers matrix and metal nanoparticles. Absorption peak was not appear even in pH neutralized solutions. Therefore, it seems that the main reason for the absence of peaks corresponding to Au nanoparticles was due to overlapping. The UV-Vis spectra of polypyrrole-gold (Fig. 1b), obtained during initial stages of polymerization, clearly shows a broad peak of around 450 nm, confirming the formation of Au nanoparticles.

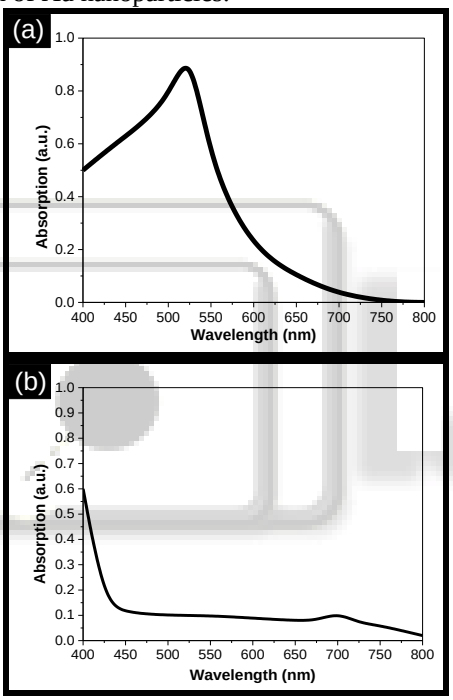

Fig. 1: UV-Visible spectra of (a) gold nanoparticles (b) PPY-Au nanocomposite

B. Fourier Transform Infrared Spectroscopy (FT-IR) Analysis

FT-IR identifies chemical bonds in a molecule by producing an infrared absorption spectrum. The FT-IR spectra PPY and PPY-Au-nanocomposite are shown in Fig. 2. The Spectrum of the PPY-Au-nanocomposite clearly exhibit some shifts as compare to the characteristic absorption peaks of PPY and evidence the insertion of gold in polymer matrix. In the FT-IR spectra of both PPY and PPY-Au-nanocomposite, absorption peaks corresponding to bipolaron bands were observed at 904 and 1170 cm^{-1} in good agreement with spectroscopic characterization of polypyrrole. The shift in position of the peak at 1536 cm^{-1} (for PPY) and 1642 cm^{-1} (for PPY-Au-nanocomposite), corresponding to –C-N stretch to higher wave numbers in the spectra, indicates an effective charge transfer between the Au and polypyrrole, as mentioned in previous reports.

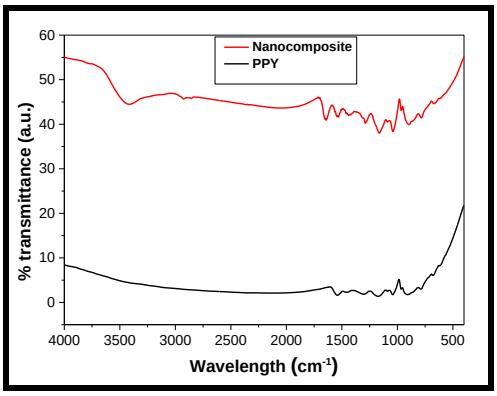

Fig. 2: FT-IR spectra of PPY-Au-nanocomposite and PPY

C. X-ray Diffraction (XRD) Analysis

XRD analysis of pure PPY and PPY-Au-nanocomposite was conducted and results are shown in Fig. 3. The XRD pattern of PPY showed a broad hump around 23-32° which was clearly manifesting the amorphous nature of the sample. However, the PPY-Au-nanocomposite exhibited sharp XRD peak (confirming the presence of Au particles) along with broad hump (for PPY). As shown in Fig. 3, peaks due to (111), (200) (220) and (310) Braggs Differaction of Au nanoparticles was clearly observed in PPY-Au-nanocomposite sample.

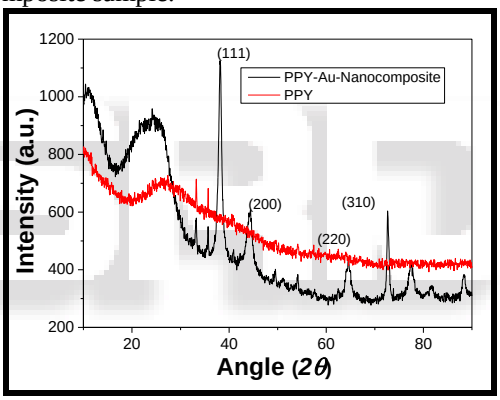

Fig. 3: XRD pattern of pure PPY and PPY-Au-nanocomposite

The Scherer's formula was used to calculate the particle size of PPY-Au-nanocomposite. The particle size was calculated as follows:

$$t = \frac{\lambda\, k}{\beta \cos\theta}$$

Here, t is thickness of crystallite, k is constant dependent on crystallite shape (here, 0.89), λ is x-ray wavelength (1.54 Å), β is integral breadth and θ is Bragg's angle. The corresponding results are summarized in Table 1.

[2θ]	Plane	Crystallite size (nm)
38.1886	(111)	14.93
44.4246	(200)	16.76
64.5947	(220)	10.20
72.6453	(310)	12.12
Average		13.50

Table 1: Size measurement of PPY-Au-nanocomposite

D. TEM Analysis

Transmission Electron Microscopic of the PPY-Au-nanocomposite was conducted as shown in Fig. 4. Most of the gold nanoparticles were found attached to the PPY matrix. The average size of the Au nanoparticles in the PPY-

Au-nanocomposite was ~10 nm. The size of Au particles in PPY-Au-nanocomposite, calculated from TEM and from XRD was in close agreement with each other.

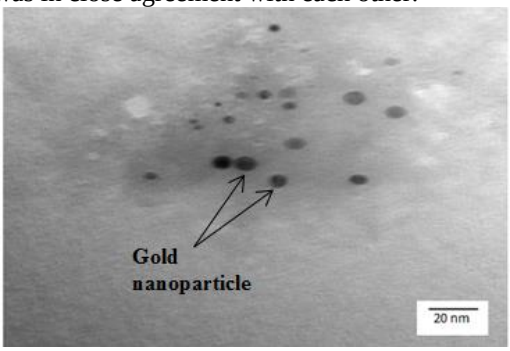

Fig. 4: TEM Image of PPY-Au nanocomposite

E. Electric Conductivity Study

Electric conductivity of pure PPY and PPY-Au-nanocomposite were obtained by using two probe methods. The value of the conductivity of pure PPY was obtained as 2.30×10^{-8} S/cm^{-1} and for PPY-Au-nanocomposite was found ~ 1.87×10^{-7} S/cm^{-1}. Au is a conductor, the higher conductivity of the PPY-AU-nanocomposite than PPY was attributed to the presence of Au in the PPY-Au-nanocomposite. It was clearly evident from the conductivity values that the dispersion of gold nanoparticles decreased the resistivity of the nanocomposite as compare to the pure PPY due to metallic nature of gold nanoparticles.

IV. Conclusions

The current research work describes a procedure for synthesis of PPY-Au-nanocomposite comprising of well-dispersed Au nanoparticles. The UV-Visible, FITR and XRD confirmed the presence of gold nano particles in PPY–Au–nanocomposite matrix. TEM results of nanocomposite showed well attachment of the nanoparticles with the polypyrrol matrix. The average particle size of Au nanoparticles in nanocomposite was calculated using Scherer formula/TEM analysis was around 10 nm. These nanoparticles were dispersed in PPY polymer matrix in different ratio. The PPY-Au-nanocomposite showed higher electric conductivity than pure PPY and was attributed to the presence of gold in PPY matrix.

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