Synthesis and Characterization of Polypyrrole (PPy) by In-situ Polymerization Technique

M.S. Phalak1,*, R.B. Waghulade2, Y.R. Toda3

1 Department of Applied Physics, Government College of Engineering, Jalgaon – 425 001, Maharashtra, India.
2 Department of Physics, D.N.C.V.'s Arts, Commerce & Science College, Jalgaon – 425 001, Maharashtra, India.
3 Thin Film Research Lab, Dept. of Physics, Pratap College, Amalner – 425 001, Maharashtra, India.

Abstract

This work reports synthesis of polypyrrole nano powder by chemical in-situ polymerization of pyrrole in aqueous solution and ammonium persulphate solution which acts as oxidant. It is characterized by X-ray diffraction (XRD), Fourier infra-red spectroscopy (FTIR) and scanning electron microscopy (SEM). The XRD spectrum reveals that the materials are amorphous in nature. FTIR analysis confirms that all peaks are characteristic of PPy. SEM analysis showed that the powder has a uniform granular morphology and the size varies from ~500 nm to 1 μm. The micrograph of polypyrrole reveals the presence of globular particles. The formed particles are irregular in nature. The results show that the fibers are chemically formed as spherical nanostructures.

1. Introduction

Current progress on the use of polymer materials revealed that the polymer has encounter a range of applications which are not restricted only being used as passive materials due to its unusual properties, but also being used as active materials that hold conducting, electromagnetic and optical properties [1]. Besides, polymer materials have also comprehensively its potential uses in the areas of magnetic applications [2] and possibly in the area of energy storage devices [3]. The study of polymers that linked to the electronics and electrical properties are being investigated progressively more to date [4]. The common feature in the structure of conducting polymers is poly-conjugation of the π-system of their backbone [5]. In the last two decades, intensive studies have been addressed to generate the electrical conductivity in polyaromatic backbone polymers. Among them, polyaniline (PANI), Polypyrrole (PPy) and polythiophene (PT) are considered as potential candidates for development of conducting polymer type materials [6]. Among those conducting polymers, polypyrrole (PPy) is especially promising for commercial applications because of its good environmental stability, facile synthesis and higher conductivity than many other conducting polymers. PPy can often be used as biosensors [7, 8], gas sensors [9, 10], anti electrostatic coatings [11], solid electrolytic capacitor [12, 13], wires [14], polymeric batteries, electronic devices and functional membranes, etc. [15-17] and electro-chromic windows and displays, and packaging.

The preparation of conducting polymer is mostly carried out either through chemical reaction or electrochemical method. The synthesis of polymer by either of these methods depends upon the intended application of the polymer. The chemical route usually involves a polymerization reaction of monomer which carried out in the reactor [18]. It is a simple, fast and the chief synthesis method since no special instruments are required. The polymerization reaction is ended with resulting in a fine powder obtained after oxidative polymerization of the monomer by chemical oxidants in aqueous or non-aqueous solvents taken place during reaction [19]. Hence, large quantities of polypyrrole (PPy) can be made through such preparation method.

PPy can be easily prepared by chemical oxidative polymerization [20], electrochemical [21] and micro emulsion polymerization technique [22]. Subject to the progression of method development which are available, the chemical in situ polymerization was found to be the most striking and preferred method for synthesis of PPy.

2. Experimental Methods

2.1 Materials Used

Pyrrole monomer (S.D. Lab, Mumbai), ammonium persulphate (NH4)2S2O8 (Fisher Scientific), hydrochloric acid (HCl), acetone, methanol, all of analytical grade were procured and used as received without further purification. Double distilled water was used throughout this work.

2.2 Preparation of Material Powder

Fig. 1 shows schematic representation of synthesis of PPy nano powder. In this work, synthesis of PPy was done by in situ polymerization. The 0.1 M pyrrole was dissolved in 1 M HCl (used as a protonic acid) and stirred for 10 minutes to get pyrrole hydrochloride. To this solution, 0.1 M ammonium persulphate which acts as oxidant was added drop by drop with continuous stirring for 1 hr at 0 – 5 °C for polymerizing. The suspension was left for 20 hr for polymerization. Finally, the suspension was filtered and washed with distilled water repeatedly and dried in vacuum at 60 °C for 8 hr. The powder thus obtained was crushed into fine powder in an agate mortar.

Fig. 1 Flowchart showing the various steps involved in synthesis of PPy

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3. Results and Discussion

3.1 X-Ray Diffraction

X-ray diffraction study of synthesized PPy powders was carried out using BRUKER AXS48 (Germany) advanced model X-ray diffraction with CuKα, λ=1.54056 Å radiation in the 2θ range 20°-80°. The scanning speed of the specimen was maintained 0.5°/min. Fig. 2 shows the XRD pattern of synthesized PPy powder. The 2θ peak observed at 72.17° which correspond to the 420 planes of reflections. PPy only exhibits a broad characteristic peak at 2θ= 25°, implying an amorphous structure [23]. The XRD spectrum reveals that the materials are amorphous in nature. The observed peaks are matching well with ASTM reported data of pure PPy.

![Fig. 2 XRD of synthesized PPy](image)

3.2 FTIR Analysis

Fig. 3 shows the FTIR spectra of PPy powder. The peaks at 790 cm⁻¹, 921 cm⁻¹ are attributed to C–H wagging. The characteristic peaks at 1549.95 cm⁻¹ and 1469.55 cm⁻¹ correspond to the C = C stretching, whereas peaks at 1631.60 cm⁻¹ and 1302.46 cm⁻¹ represent to respectively, C = N and C – N bonds. The occurrence of small peaks at 3432.66 cm⁻¹ is assigned to presence of N-H stretching vibrations. All these peaks are the main characteristic of PPy [24-26].

![Fig. 3 FTIR spectra of synthesized PPy](image)

3.3 Scanning Electron Microscopy

The microstructure of a PPy thin film is shown in Fig. 4. The powder has a uniform granular morphology and it is observed that the PPy shows uniform porous nature and smaller and bigger voids everywhere, and the size varies from ~500 nm to 1 μm. The micrograph of polypyrrole reveals the presence of globular particles. The particles formed are irregular in nature. One can clearly observe that the fiber is chemically coated by the spherical nanostructures [27]. A higher porosity usually led to a better response owing to a higher rate of gas absorption.

![Fig. 3 SEM micrograph of synthesized PPy](image)

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