Share Your Innovations through JACS Directory

Journal of Advanced Chemical Sciences

Visit Journal at https://www.jacsdirectory.com/jacs

ISSN: 2394-5311

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

M.S. Phalak[1,*], R.B. Waghulade[2], Y.R. Toda[3]

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

ARTICLE DETAILS

Article history:
Received 17 July 2020
Accepted 07 August 2020
Available online 09 September 2020

Keywords:
In-situ Polymerization
Polypyrrole
Nanopowder

ABSTRACT

This work reports synthesize of polypyrrole nano powder by chemical *in-situ* polymerization of pyrrole in aqueous solution and ammonium persulfate 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 the main 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 comprehensive 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 is 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 ($(NH_4)_2S_2O_8$) (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.

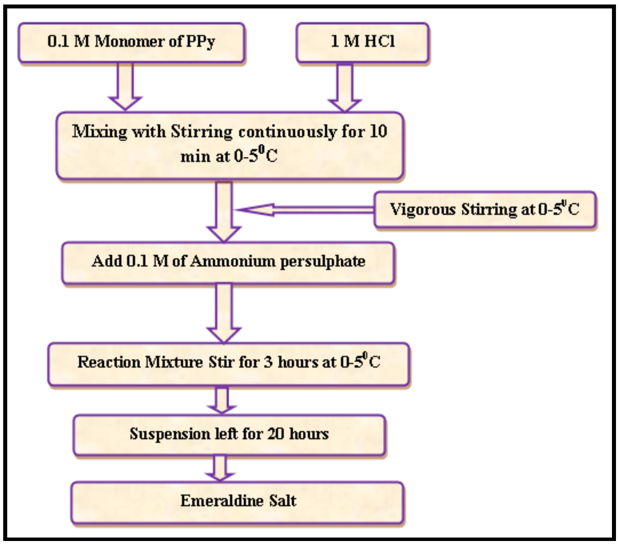

Fig. 1 Flowchart showing the various steps involved in synthesis of PPy

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 of 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 persulfate 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.

---

*Corresponding Author:mrinaliniphalak26@gmail.com(Mrunalini Phalak)

https://doi.org/10.30799/jacs.224.20060203

Cite this Article as: M.S. Phalak, R.B. Waghulade, Y.R. Toda, Synthesis and characterization of polypyrrole (PPy) by in-situ polymerization technique, J. Adv. Chem. Sci. 6(2) (2020) 686–688.

3. Results and Discussion

3.1 X-Ray Diffraction

X-ray diffraction study of synthesized PPy powders was carried out using BRUKER AXSD8 (Germany) advance model X-ray diffraction with CuKα_1 (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.

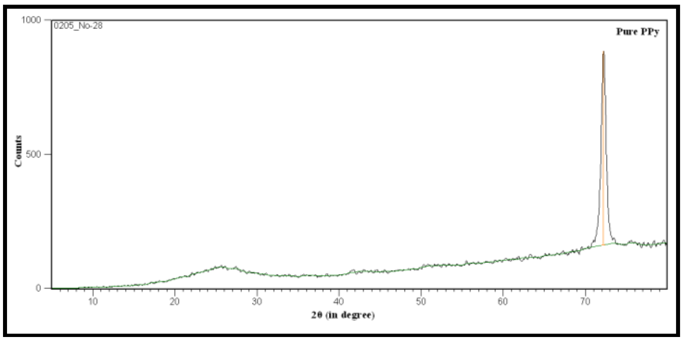

Fig. 2 XRD of synthesized PPy

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].

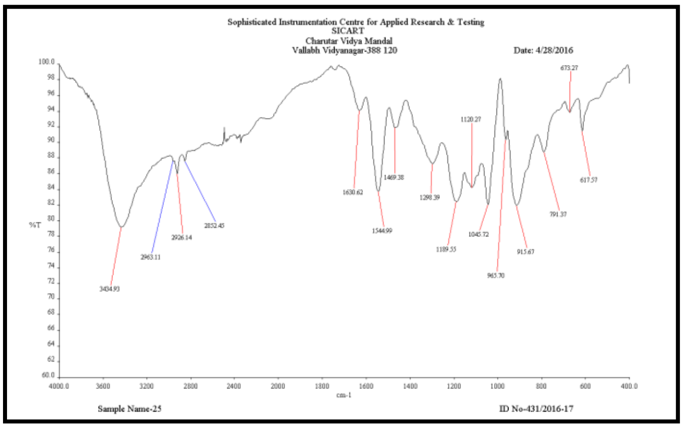

Fig. 3 FTIR spectra of synthesized PPy

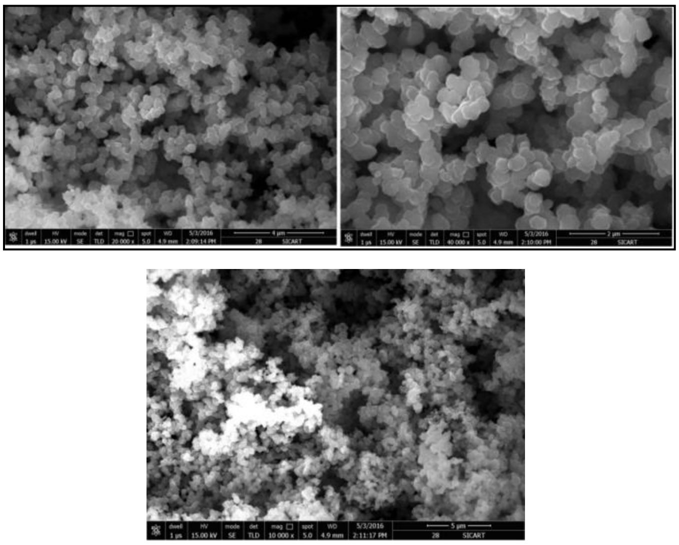

Fig. 3 SEM micrograph of synthesized PPy

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.

4. Conclusion

Nano powder of PPy have been successfully synthesized by the chemical *in-situ* polymerization method and characterized using XRD, FTIR and SEM techniques. XRD spectrum of PPy exhibits only a broad characteristic peak at 2θ= 25° and reveals that the materials are amorphous in nature. FTIR spectra of synthesized PPy powder showed that all peaks are the main characteristic of PPy. The SEM micrograph of synthesized polypyrrole reveals the presence of globular particles of PPy and the size varies from ~500 nm to 1 µm. One can clearly observe that the fiber is chemically synthesized to lead spherical nanostructures.

References

[1] Danming Chao, Jingyu Chen, Xiaofeng Lu, Liang Chen, Wanjin Zhang, Yen Wei, SEM study of the morphology of high molecular weight polyaniline, Synth. Metals 150(1) (2005) 47-51.
[2] M.A. Elita Hafizah, A. Bimantoro, Andreas, A. Manaf, Synthesized of conductive polyaniline by solution polymerization technique, Procedia Chem. 19 (2016) 162-165.
[3] R. Kumar, S. Singh, B.C. Yadav, Conducting polymers: Synthesis, properties and applications, Int. Adv. Res. J. Sci. Eng. Tech. 2(11) (2015) 2393-8021.
[4] Inzelt György, Conducting polymers: A new era in electrochemistry, 2nd Edition, Spinger-Verlag, Berlin, 2012.
[5] T.A. Skotheim, Handbook of conducting polymers, Vol. 1&2, Marcel Dekker, New York, 1986.
[6] Bebi Patil, Ravindra Bulakhe, Chandrakant Lokhande, Super capacitive performance of chemically synthesized polypyrrole thin films: Effect of monomer to oxidant ratio, J. Mater. Sci. Mater. Elect. 25(5) (2014) 2188-2198.
[7] J.C. Vidal, E. Garcia, J.R. Castillo, *In-situ* preparation of a cholesterol biosensor: entrapment of cholesterol oxidase in an over oxidized polypyrrole film electrodeposited in a flow system: Determination of total cholesterol in serum, Anal. Chim. Acta 385(1-3) (1999) 213-222.
[8] T.E. Campbell, A.J. Hodgson, G.G. Wallace, Incorporation of erythrocytes into polypyrrole to form the basis of a biosensor to screen for rhesus (D) blood groups and rhesus (D) antibodies, Electroanal. 11(4) (1999) 215-222.
[9] D. Kincal, A. Kamer, A.D. Child, J.R. Reynold, Conductivity switching in polypyrrole-coated textile fabrics as gas sensors, Synth. Metals 92 (1998) 53-55.
[10] N.T. Kemp, G.U. Flanagan, A.B. Kaiser, H.J. Trodahl, B. Chapman, et al., Temperature dependent conductivity of conducting polymers exposed to gases, Synth. Metals 101(1-3) (1999) 434-435.
[11] S.C. Yang, H. Liu, R.L. Clark, International patent PCT Int. Application, WO 99 22, 380 (Cl.H01 B1/00).
[12] T. Takamatsu, Y. Taketani, International patent Jpn. Kokai. Tokyo Koho JP11 121, 279 [99 121,279]
[13] Y. Kojima, H. Kamikawa, T. Takamatsu, International patent Jpn. Kokai. Tokyo Koho JP11 121, 280 [99 121, 280].
[14] C. Jérôme, D. Labaye, I. Bodart, R. Jérôme, Electrosynthesis of polyacrylic/polypyrrole composites: Formation of polypyrrole wires, Synth. Metals 101(1-3) (1999) 3-4.
[15] T.A. Skotheim, Metal insulator transition in doped conducting polymer, Marcel Dekker, New York, 1986.
[16] T.A. Skotheim, R. Elsenbaumer, J. Reynolds, Handbook of conducting polymers, Marcel Dekker, New York, 1998.
[17] G.G. Wallace, G. Spinks, P.R. Teasdale, Conductive electro active polymers, Technomic, New York, 1997.
[18] Gina Ayu Wardhani, Mas Ayu Elita Hafizah, Andreas, Azwar Manaf, Comparative studies on the synthesis of polypyrrole through polymerization reaction between batch and continuous processes, IOP Conf. Series: Mater. Sci. Eng. 553 (2019) 012054:1-8.
[19] Saville, Paul, Polypyrrole formation and use, Technical memorandum, Defense Research and Development Canada, Canada, 2005.
[20] Salma Bilal, Fouzia Perveen, Anwar-ul-Haq Ali Shah, Chemical synthesis of polypyrrole doped with dodecyl benzene sulfonic acid, J. Sci. Innovat. Res. 4(1) (2015) 33-42.
[21] M.A. Chougule, S.G. Pawar, P.R. Godse, R.N. Mulik, Shashwati Sen, V.B. Patil, Synthesis and characterization of polypyrrole (PPy) thin films, Soft Nanosci. Lett. 1(1) (2011) 6-10.
[22] V. Vernitskaya, Tatyana, Efimov, N. Oleg, Polypyrrole: a conducting polymer; its synthesis, properties and applications, Rus. Chem. Rev. 66(5) (1997) 443-457.
[23] Y.L. Luo, L.H. Fan, F. Xu, Y.S. Chen, C.H. Zhang, Q.B. Wei, Synthesis and characterization of Fe_3O_4/PPy/ P(MAA-co-AAm) tri-layered composite

https://doi.org/10.30799/jacs.224.20060203

Cite this Article as: M.S. Phalak, R.B. Waghulade, Y.R. Toda, Synthesis and characterization of polypyrrole (PPy) by in-situ polymerization technique, J. Adv. Chem. Sci. 6(2) (2020) 686–688.

microspheres with electric, magnetic and pH response characteristics, Mater. Chem. Phys. 120 (2010) 590–597.

[24] Manik A. Chougule, Shailesh G. Pawar, Prasad R. Godse, Ramesh N. Mulik, Shashwati Sen, Vikas B. Patil, Synthesis and characterization of Polypyrrole (PPy) thin films, Soft Nano Sci. Lett. 1 (2011) 6-10.

[25] M.E. Nicho, H. Hu, Fourier transform infrared spectroscopy studies of Polypyrrole composite coatings, Solar Energy Mater. Solar Cells 63 (2000) 423-435.

[26] Sobhan Bahraeian, Khatereh Abron, Fatemeh Pourjafarian, Rohah A. Majid, Study on synthesis of polypyrrole via chemical polymerization method, Adv. Mater. Res. 795 (2013) 707-710.

[27] Naader Alizadeh, Sajad Pirsa, Ahmad Mani Varnosfadaderani, Mohammad Sadegh Alizadeh, Design and fabrication of open tubular array gas sensors based on conducting polypyrrole modified with crown ethers for simultaneous determination of alkylamines, IEEE Sens. Jour. 15(7) (2015) 4130-4136.

https://doi.org/10.30799/jacs.224.20060203

Cite this Article as: M.S. Phalak, R.B. Waghulade, Y.R. Toda, Synthesis and characterization of polypyrrole (PPy) by in-situ polymerization technique, J. Adv. Chem. Sci. 6(2) (2020) 686–688.