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One-pot synthesis of porphyrin@polypyrrole hybrid and its application as an electrochemical sensor

Shruti Peshoria, Anudeep Kumar Narula*

Molecular Chemistry Laboratory, University School of Basic and Applied Sciences, Guru Gobind Singh Indraprastha University, Sector-16 C, Dwarka, New Delhi 110078, India

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ABSTRACT

In this work, hybrid of polypyrrole with *meso*-(tetracarboxyphenyl)porphyrin (TCPP@ppy) was prepared by a simple one-pot chemical oxidative polymerization method using ammonium persulfate (APS) as the oxidizing agent. Fourier transform infrared spectroscopy (FT-IR) revealed its successful formation with the presence of N–H, C═O and other characteristic peaks of polypyrrole and *meso*-(tetracarboxyphenyl)porphyrin (TCPP) which was also corroborated by ultraviolet–visible (UV–Vis) spectroscopy and X-ray diffraction (XRD) studies with a red shifted Soret band of TCPP and a broad peak at $2\theta = 23.9^{\circ}$ including distinctive TCPP peaks respectively. This denoted that interaction took place between various components. High-resolution transmission electron microscopy (HR-TEM) micrographs of the hybrid depicted TCPP molecules to be encapsulated by ppy matrix. Cyclic voltammetry studies illustrated high electroactivity for the hybrid and ppy. Electrochemical sensor was developed using TCPP@ppy for the detection of cadmium (II) ions (Cd^{2+}) by differential pulse voltammetry technique, thus exhibiting its application as a metal ion sensor.

1. Introduction

Polypyrrole is a well investigated conducting polymer matrix in the field of electrochemical sensors [1], supercapacitors [2], solar devices [3] and drug-delivery systems [4] due to its good redox properties, electrical conductivity, facile synthesis and functionalization, low cost, stability, biocompatibility and environment friendly nature [5]. The electrochemical metal sensing properties of polypyrrole have also been successfully explored taking the advantage of the interaction between nitrogen and metal ions thus improving its prospects to be used in their detection [6]. Electrochemical metal ion sensors have generated much interest in this area as they provide fast and sensitive detection with accuracy, reproducibility, easy operation, portability and low cost [7], thus overcoming the disadvantages of high costs, difficult procedure and immobility associated with other analytical techniques like atomic absorption spectroscopy (AAS), flame atomic absorption spectrometry, inductively coupled plasma mass spectrometry (ICP-MS), X-ray fluorescence etc. [1c]. Since, toxic heavy metals like cadmium (Cd), mercury (Hg), lead (Pb) and arsenic (As) are harmful and have deleterious effects on the living world [7e] with the major concerns being, toxicity at trace concentration, bio accumulative and non-biodegradable nature [8], it is necessary to develop such sensors. Cd^{2+} is primarily responsible for renal and kidney dysfunction and is a carcinogen causing lung cancer, bone degeneration and blood problems [9]. Further, in order to improve the sensitivity and selectivity of these sensors, polypyrrole has also been functionalized with other ligands like Mahmoudian et al. fabricated a composite of polypyrrole with platinum nanoparticles for electrochemical sensing of Hg^{2+} [1c], Dai et al. synthesized nanocomposites of polypyrrole and graphene oxide which was further functionalized with phytic acid for simultaneous electrochemical sensing of Cd^{2+} and Pb^{2+} [10] and Joseph et al. developed electrochemical Pb^{2+} sensor using polypyrrole functionalized with iminodiacetic acid [11]. Another chemical entity to be used in optical and electrochemical metal ion sensors is the free base porphyrin which is considered to have excellent metal coordinating property [12]. For example, Liu et al. prepared visual detector for Cd^{2+} using cotton fiber functionalized with 5,10,15,20-tetrakis(1-methy-4-pyridinio)porphyrin tetra(p-toluenesulfonate) [13], Buntem et al. incorporated *meso*-tetra(p-carboxyphenyl)porphyrin in silica glass to make optical metal ion sensor [12], Zhao et al. fabricated optical Cd^{2+} sensor employing 5,10,15,20-tetrakis(4-N-methylpyridyl)porphyrin p-toluenesulfonate [14] and Cui et al. developed electrochemical sensor for Pb^{2+} by making DNA functionalized iron-porphyrinic metal–organic framework with *meso*-tetra(p-carboxyphenyl)porphyrin [15]. Therefore, in the present work it was considered significant to synthesize a hybrid containing polypyrrole and *meso*-(tetracarboxyphenyl)porphyrin (TCPP),

* Corresponding author.
E-mail address: aknarula@ipu.ac.in (A.K. Narula).

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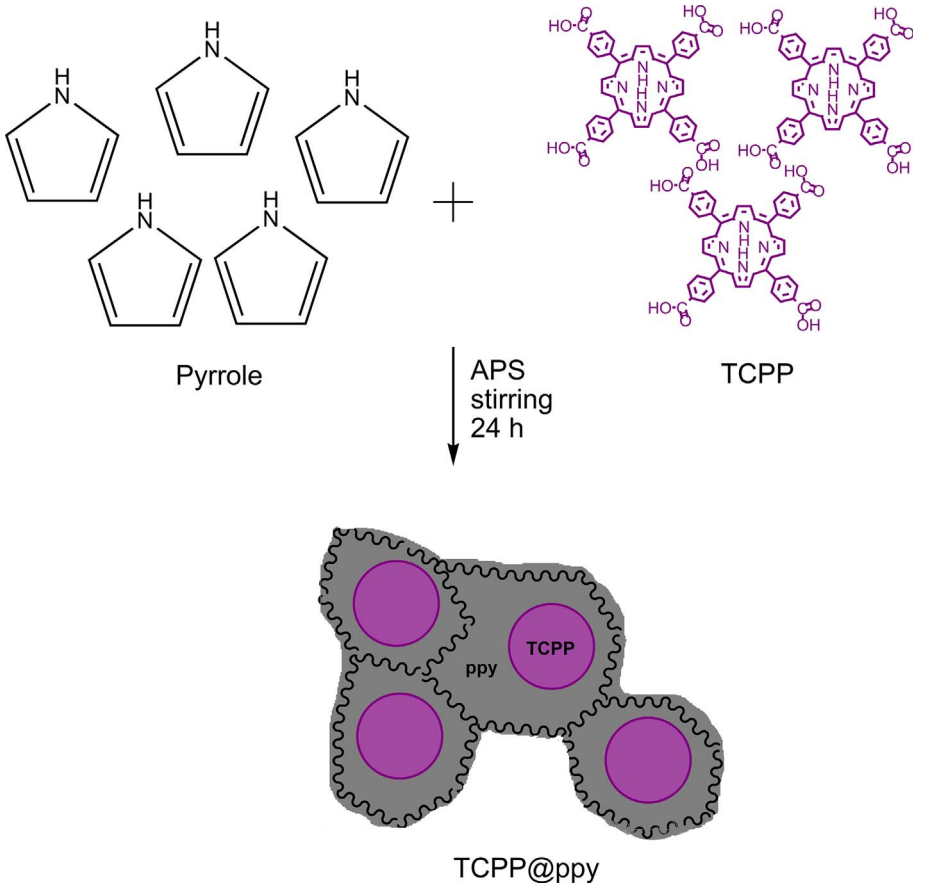

Fig. 1. Schematic showing preparation of TCPP@ppy hybrid.

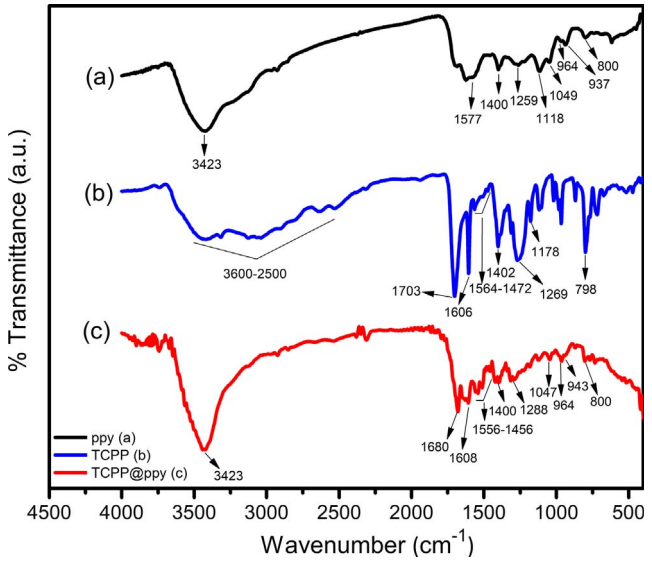

Fig. 2. FT-IR spectra of (a) ppy, (b) TCPP and (c) TCPP@ppy.

with the negatively charged TCPP acting as a bulky dopant during polypyrrole formation, its characterization and to explore its application as an electrochemical sensor for Cd^{2+}, using differential pulse voltammetry (DPV).

2. Experimental

2.1. Materials and methods

Pyrrole monomer (99%, Spectrochem) was distilled before use and *meso*-(tetracarboxyphenyl)porphyrin (TCPP) (> 97.0%) was purchased from TCI Chemicals, other chemicals viz. cadmium chloride monohydrate, $CdCl_2{\cdot}H_2O$ (CDH), lead(II) acetate trihydrate, Pb $(CH_3COO)_2{\cdot}3H_2O$ (CDH), copper(II) acetate monohydrate, Cu $(CH_3COO)_2{\cdot}H_2O$ (Fisher Scientific), zinc(II) acetate dihydrate, Zn $(CH_3COO)_2{\cdot}2H_2O$ (CDH), nickel(II) chloride hexahydrate, $NiCl_2{\cdot}6H_2O$ (S.D. Fine Chem. Limited), cobalt(II) acetate tetrahydrate, Co $(CH_3COO)_2{\cdot}4H_2O$ (Fisher Scientific), mercuric chloride, $HgCl_2$ (CDH), ammonium persulfate, $(NH_4)_2S_2O_8$ (APS) (CDH), sodium acetate trihydrate, $CH_3COONa{\cdot}3H_2O$ (Fisher Scientific), acetic acid glacial, CH_3COOH (SRL), potassium ferrocyanide, $K_4Fe(CN)_6{\cdot}3H_2O$ (CDH), potassium ferricyanide, $K_3Fe(CN)_6$ (SRL) were of AR grade and used as such. Nafion D-520 dispersion (5% w/w in water and 1-propanol, Alfa Aesar) and ethanol, C_2H_5OH were also used as such. Deionized water with resistivity of 18.2 MΩ cm at 25 °C was used for all experiments. 0.1 mM stock solution of Cd^{2+} and 10 mM stock solutions of other salts (Ni^{2+}, Zn^{2+}, Cu^{2+}, Hg^{2+}, Pb^{2+} and Co^{2+}) were used for making further dilutions.

2.2. Characterization details

The morphological studies were carried out using HR-TEM on FEI Tecnai G2 F30 at 300 kV and scanning electron microscopy (SEM) on SEM ZEISS EVO18 at a voltage of 20 kV. FT-IR spectra were recorded using Shimadzu IRAffinity-1S in KBr pellet mode with 45 scans at a resolution of 4 cm^{-1} from 4000 to 400 cm^{-1}. UV–Vis spectra were obtained on a Hitachi U-2900 UV–Visible spectrophotometer. X-ray diffraction (XRD) was carried out on a Rigaku Ultima IV X-ray diffractometer with Cu Kα radiation (λ = 1.541 Å) at an operating voltage and current of 40 kV and 30 mA respectively. Electrochemical measurements were done by cyclic voltammetry and DPV in a three electrode cell assembly connected to a potentiostat (CorrTest two channel

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