

Electrochemical synthesis and characterization of a new conducting polymer: Polyrhodanine

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Abstract

An antimicrobial drug, rhodanine (Rh), was electrochemically polymerized on a Pt electrode using cyclic voltammetry (CV). The high quality and homogeneous polyrhodanine (pRh) films with a dark-purple color were obtained. The chemical structure characterization was investigated by Fourier transform infrared spectroscopy (FTIR) and UV–vis spectroscopy techniques. Further, thermogravimetric analysis (TGA) and differential thermal analysis (DTA) techniques used to investigate thermal properties of the film. It is found that thermal stability of pRh films is relatively high. It is also observed that tetrahydrofurane (THF) and *N*-methyl-2-pyrrolidone (NMP) are good solvents for the polymer.
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1. Introduction

The study of high quality conducting polymer films is one of the main goals in the research and development of inherently conducting polymers because of their potential applications. Conducting polymers can be synthesized from a number of compounds. Due to their unique properties such as electrical conductivity, good environmental and thermal stability, “conventional” polymers (e.g. polyaniline, polypyrrole and polythiophene) are the most widely studied ones. Further researches have been aimed at increasing the variety as well as developing the properties of these polymers.

A great number of conductive polymers was developed and tested for diverse applications. One of these application areas is their use for corrosion protection of metals in aqueous environments [1,2]. They may function as organic inhibitors due to their great number π bonds, reduce the number of active sites on the metal surface through adsorption, and act as a barrier slowing the transport of corrosive agents [3,4].

Many applications have recently been appeared in literature. The incorporation of catalyst micro particles into a conducting polymer film represents an attractive field of researches [5–8]. Conducting polymers have been studied extensively for the

development of the biosensors [9–14] and are known for their ability to compatible with biological molecules in neutral aqueous solutions [15]. In addition, they have the ability to efficiently transfer the electric changes produced by biochemical reactions to electronic circuits [16]. Finally, they may serve as the immobilizing matrices for bio-molecules and provides a suitable environment for their immobilization [9].

Rhodanine (Rh) and its derivatives possess a wide variety of biological activities such as anticonvulsant [17], antibacterial [18], antidiabetic [19], antiviral [20], antimicrobial [21,22], antihistaminic [23] and inhibition of HVC [24,25], JSP-1 [26], etc. Rhodanine and its some derivatives have higher sensitivity and selectivity for the analysis of some noble metal ions [27,28]. They were also tested as corrosion inhibitor and reported to be effective [29,30].

Polymerization of rhodanine and its derivatives has yet been reported. The goal of this study is to investigate and characterize the electropolymerization of rhodanine. Chemical structure of rhodanine is given in Fig. 1.

2. Experimental

2.1. Electrochemical synthesis

All the chemicals were purchased from Merck and used without any further purification. The solutions were prepared

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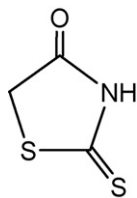


Fig. 1. Chemical structure of rhodanine.

with bi-distilled water. Electrochemical measurements were carried out using a CHI 604 electrochemical analyzer (serial number 6A721A) under computer control. Platinum in dimension of $1\text{ cm} \times 1\text{ cm}$ and the $\text{Ag}/\text{AgCl}_{(\text{g})}$, KCl (3 M) electrodes were used as counter electrode and reference electrode, respectively. The potentials values were given versus this reference electrode.

Electrochemical synthesis was carried out on a platinum sheet (with 2 cm^2 surface area) from 0.01 M monomer containing 0.3 M ammonium oxalate solution using cyclic voltammetry (CV) technique by potential cycling between 0.00 and 1.40 V at a scan rate 100 mVs^{-1} .

2.2. Characterization

IR spectra were recorded in KBr pellets in the $450\text{--}4000\text{ cm}^{-1}$ frequency range using a Perkin-Elmer RX I FTIR spectrophotometer (serial number 55148).

The polymer was tested for solubility in dichloromethan (DCM), tetrahydrofuran (THF), acetone, *N*-methyl-2-pyrrolidone (NMP), ethanol (EtOH) solvents and also in acidic (HCl) and basic (NaOH) solutions.

Conductivity measurements were made as described elsewhere [31].

The UV–vis adsorption spectra were recorded using UV-2101PC UV–vis Spectrophotometer. The solution of the sample in 0.1 M NaOH was used for recording the spectra.

Thermal analyses data were obtained under nitrogen atmosphere using Pyris Diamond TG/DTA Perkin-Elmer thermal analysis and Pyris 7.0 data-processing system at a heating rate of $10\text{ }^{\circ}\text{C}/\text{min}$ over a temperature range of $20\text{--}700\text{ }^{\circ}\text{C}$.

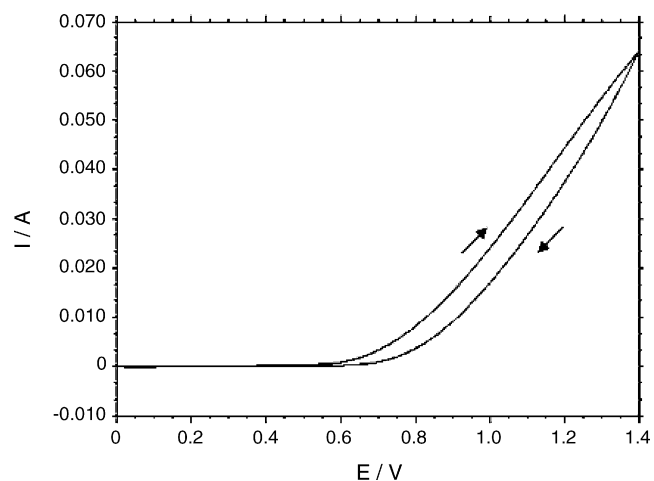
Surface photographs of polymer films were also taken after different cycles.

3. Results and discussions

3.1. Electrochemical synthesis

The cyclic voltammogram of Pt in 0.3 M ammonium oxalate solution is given in Fig. 2. At the forward scan, the current is constant around zero between 0.00 and 0.60 V potential range. Then the anodic current increases up to 1.40 V.

Fig. 3 shows the CV obtained in 0.01 M monomer containing 0.3 M ammonium oxalate solution. In the first cycle, two oxidation peaks observed at the forward scan. These oxidation peaks are assigned to the monomer oxidation. At the reverse scan, there is only one weak peak at around 0.15 V. This peak is due to the reduction of the platinum oxide created during the

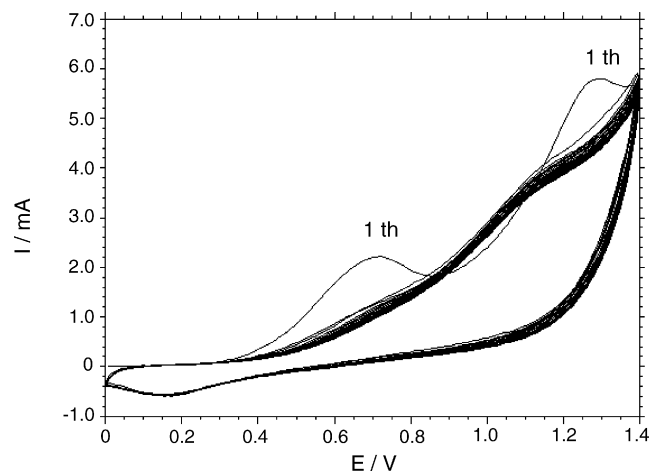
Fig. 2. The cyclic voltammogram recorded for Pt electrode in 0.3 ammonium oxalate, scan rate: 100 mVs^{-1} .

positive sweep. In the second cycle, monomer oxidation is accompanied by the formation of polymer film on the metal or oxide electrode. When the cycle numbers increased, the color of the electrode surface changed slightly from colorless to transparent-yellow and then to dark purple at further cycling. This behavior can be attributed to the re-oxidation of pRh film around 1.10 V. CV results are indicating the formation of the polymer film on the platinum surface.

The photographs of the electrochemically synthesized pRh films on platinum surface after 10 cycles (a) and 50 cycles are given in Fig. 4. It can be clearly seen that yellow-colored polymer film was formed on platinum surface after 10 cycles (Fig. 4a). The film color changed to dark purple with increasing cycle number. In this study, 50 whole cycles were applied for the formation of pRh film (Fig. 4b). The obtained films were found to be very smooth, homogenous and adherent.

3.2. FTIR spectroscopy

To clarify the structure of pRh films *ex situ* FTIR spectroscopy was employed. The FTIR spectra of pRh film

Fig. 3. The voltammograms recorded during film growth on Pt electrode in 0.3 M ammonium oxalate + 0.01 M rhodanine (50 cycles), scan rate: 100 mVs^{-1} .

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