



Electrochemical synthesis and surface characterization of (pyrrole+2-methylfuran) copolymer



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ABSTRACT

Electrochemical copolymerization of pyrrole (Py) and 2-methylfuran (2 MF) was performed on platinum and ITO substrates in acetonitrile/lithium perchlorate solution, using cyclic voltammetry method. The electrochemical behavior of the modified electrode surface by polypyrrole, poly(2-methylfuran) homopolymers and (pyrrole+2-methylfuran) copolymer was characterized by cyclic voltammetry, electrochemical impedance spectroscopy (EIS), UV–visible spectroscopy, atomic force microscopy (AFM) and scanning electron microscopy (SEM). The cyclic voltammetry shows anodic and cathodic peaks which are characteristic of the oxidation and the reduction of the formed films. The electrochemical impedance spectroscopy confirmed the results obtained by cyclic voltammetry. AFM and SEM analyses proved as well that the morphology and the electrochemical properties of the polypyrrole film are modified in the presence of 2-methylfuran.

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1. Introduction

Every year, the scope of the technological applications of conducting organic polymers is further extended in a wide variety of areas, due to their ease of synthesis and interesting electrical, electrochemical, structural, mechanical and optical properties [1–3].

Among them, polypyrrole (PPy) is one of the most widely investigated conducting polymers because of its low oxidation potential [4], easy preparation, efficient polymerization at neutral pH, stability in ambient conditions, thickness controllability and good electrical conductivity [5–7]. These properties are favorable to its application in the areas of metallization of dielectrics [8], batteries and supercapacitors [9–11], actuators [12], protection against corrosion [13], development of solar cells [14], biocompatible coatings [15], sensors [16], electro-catalysis and electrochromic display devices [17].

On the other hand, synthesis and characterization of polyfuran (PFu) and its derivatives have attracted less attention because of their supposed less well-defined structures, poor stability, low conductivities, poor mechanical properties and ill-defined electrochemical behavior, which mainly resulted from the high

oxidation potential [18]. PFu conducting polymers can be prepared either by chemical or electrochemical polymerization [19]. Most recently, the furan-based materials were also found to have potential applications in organic electronic devices such as field-effect transistor and photovoltaic applications [20,21], biosensor technology [22] and as humidity sensor [23].

Due to the possibility of modifying the properties of individual polymers, the electrochemical copolymerization was one of the most useful methods because of its several advantages. The copolymer is expected to gain good mechanical properties from its two components through sacrificing some of these qualities. Thus, copolymers of heterocyclic compounds may be useful in practical applications.

This work concerns the synthesis, electrochemical and spectroscopic characterization of polypyrrole, poly(2-methylfuran) and (Py+2 MF) copolymer films obtained from pyrrole and 2-methylfuran on a platinum and ITO electrodes. These modified electrodes can be used in various applications, such as light emitting diodes (LEDs) and photovoltaic cells. Characterization of the obtained films has been carried out using electrochemical impedance spectroscopy (EIS), UV–vis spectroscopy, atomic force microscopy (AFM) and scanning electron microscopy (SEM), in order to have information on their electrochemical properties.

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2. Experimental

Pyrrole (Py) and 2-Methylfuran (2 MF) were purchased from Aldrich (98%), they were stored at 4 °C in the absence of light and used as received without further purification. The acetonitrile, pure product for analysis was obtained from Aldrich. The supporting electrolyte salt lithium perchlorate (LiClO_4) was purchased from Fluka.

The electrochemical depositions were performed in a three-electrode cell using a PGZ 402 VoltaLab coupled to a computer under Voltmaster software which makes it possible to select the electrochemical technique and to fix the desired parameters. The working electrode was a platinum disk ($\varnothing = 1 \text{ mm}$) or ITO substrates (SOLEMS), the auxiliary electrode was a platinum wire, and the reference electrode was a saturated calomel electrode with KCl (SCE).

The pre-treatment of the exposed surface was performed by polishing mechanically to smooth surface finish, using emery paper down to 1200 grade, degreasing with acetone, washing with distilled water and drying.

The electrochemical impedance spectroscopy measurements were carried out using perturbation voltage amplitude of 10 mV in the frequency range between 100 kHz and 50 mHz. The cell used during the impedance measurements was a traditional cell containing the $\text{CH}_3\text{CN}/(\text{LiClO}_4 \text{ 0.1 M})$ solution, without monomer.

The polypyrrole, poly(2-methylfuran) and the (Py+2 MF) copolymer deposited on transparent ITO plates were characterized by UV–vis absorption spectroscopy using a Shimadzu UV-1800 spectrophotometer. MFP 3D atomic force microscope (from Asylum research) was employed for examining the surface topography of the films. The surface morphology of the films was investigated using a XL30 ESEM microscope operating at 5 kV.

3. Results and discussion

3.1. Electropolymerization of pyrrole

Fig. 1 shows successive cyclic voltammograms corresponding to pyrrole (0.01 M) dissolved in $\text{CH}_3\text{CN}/(\text{LiClO}_4 \text{ 0.1 M})$ electrolyte solution and recorded in a potential range between -0.3 and $+1.3 \text{ V}$ vs SCE, with scan rate of 50 mV s^{-1} .

The cyclic voltammograms show during the positive scan

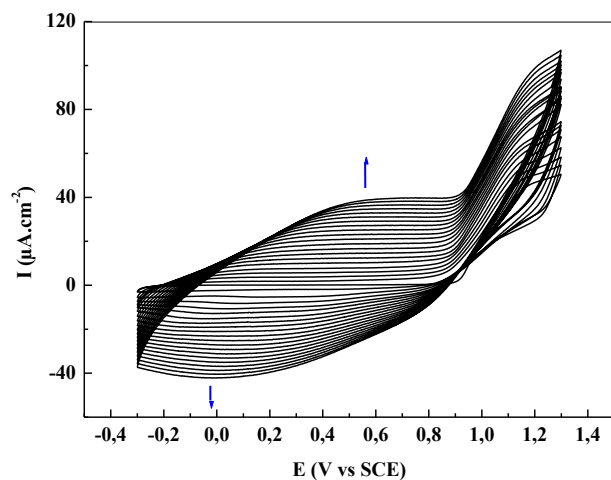


Fig. 1. Cyclic voltammograms relating to polypyrrole (PPy/Pt), in a $\text{CH}_3\text{CN}/(\text{LiClO}_4 \text{ 0.1 M})$ solution, recorded between -0.3 and $+1.3 \text{ V}$ vs SCE at a scan rate of 50 mV s^{-1} .

potential a large anodic wave around 0.5 V and a peak at 1.2 V/SCE, whereas in the reverse scan, a cathodic wave is illustrated at 0 V, this result is in good agreement with literature values [24]. The anodic and cathodic waves correspond respectively to oxidation and reduction of the polypyrrole [25]. The oxidation wave is seen to shift slowly towards higher voltages with repeated scans, whereas the reduction wave is observed to shift to even lower negative voltages. These shifts are accompanied by an increase in the current intensity. It is worth noting the increase in the voltage difference range ($\Delta E_p = E_{pa} - E_{pc}$) with increasing the number of cycles, which is a clear indication of the deposition of polypyrrole film on the electrode surface.

3.2. Electropolymerization of 2-methylfuran

Fig. 2 shows cyclic voltammograms corresponding to the electrochemical polymerization of 2-methylfuran (0.01 M) dissolved in $\text{CH}_3\text{CN}/(\text{LiClO}_4 \text{ 0.1 M})$ electrolyte solution, carried out in a potential range between 0 and 2 V/SCE, with scan rate of 50 mV s^{-1} .

During the positive scan potential, an anodic peak was observed around 1.56 V/SCE and preceded by a shoulder at 0.85 V which disappears during the successive scanning of the potential, corresponding to the oxidation of 2-methylfuran monomer. In the reverse scan, a cathodic peak was obtained at 0.24 V corresponding to the reduction of the formed polymer film. A slight decrease of the current intensities of the anodic and cathodic peaks was observed. This is probably due to the inhibition of the surface by an insulating polymeric film which was carried out on the electrode. Nessark et al. [26,27] also suggested that the current intensity of the anodic peak decreases during cycling and it stabilizes after many cycles when the electrode surface is coated by an insulating film. However, the anodic and cathodic peak potentials were observed to remain the same, indicating that the kinetic process of both the oxidation and the reduction reactions remains unchanged during cycling.

3.3. Electropolymerization of (pyrrole+2-methylfuran) copolymer

Fig. 3 shows cyclic voltammograms of the $\text{CH}_3\text{CN}/(\text{LiClO}_4 \text{ 0.1 M})$ solution, containing Py (0.01 M) and 2 MF for different concentrations (10^{-4} , $5 \cdot 10^{-4}$, 10^{-3} , $5 \cdot 10^{-3}$ and 10^{-2} M), which were obtained at scan rate $v = 50 \text{ mV s}^{-1}$, between -0.3 and 2 V vs SCE.

The cyclic voltammograms (Fig. 3) show during the positive scan

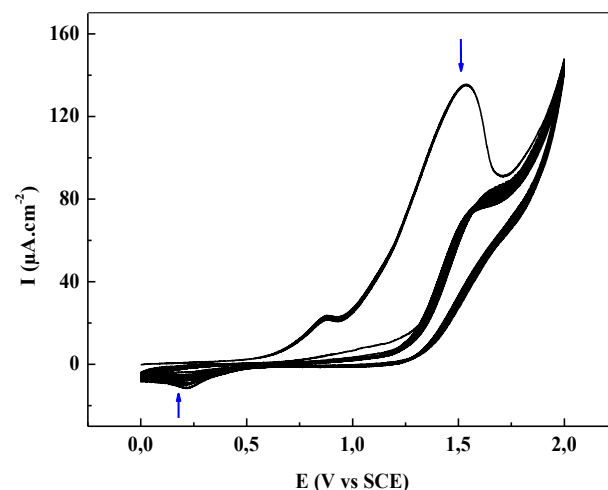


Fig. 2. Cyclic voltammograms relating to poly(2-methylfuran) (P2MF/Pt), in a $\text{CH}_3\text{CN}/(\text{LiClO}_4 \text{ 0.1 M})$ solution, recorded between 0 and 2 V vs SCE, at $v = 50 \text{ mV s}^{-1}$.

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