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Electrochemical preparation of polypyrrole conducting films

Mária Filkusová*, Renáta Oriňáková

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Abstract

Cyclic voltammetry has been used to investigate the electrochemical polymerization of pyrrole on the surface of a paraffin impregnated graphite electrode (PIGE). Effect of pH and concentration of the electrolyte solution on the electrochemical deposition of polypyrrole (PPy) was studied. The structure of the deposited layers was studied using scanning electron microscope (SEM). Well–adhering black PPy films were obtained.

Keywords: pyrrole, polypyrrole, electrochemical polymerization, paraffin impregnated graphite electrode

Introduction

Polypyrrole (PPy) is one of the most promising polymers for technological and biomedical applications [1]. PPy is one of the most widely used polymers in the research and in the industry, where its use is found in antistatic coating, cell culture substrate, flexible electronics, gas and chemical sensors [2], design of biosensors based on immobilized enzymes, antibodies or DNA [3], batteries and corrosion protection coating [2]. In this paper, we studied the influence of electrochemical polymerization conditions such as electrolyte pH and concentration on the morphology and quality of PPy layers.

Materials and methods

All chemicals were of analytical grade or better quality. Distilled water and freshly prepared solutions were used throughout. Pyrrole (Py) monomer (98+%) was obtained from Sigma–Aldrich. All experiments were carried out at room temperature.

Mária Filkusová*

Department of Physical Chemistry, Faculty of Science, Comenius University, Mlynská Dolina, SK–842 15 Bratislava 4, Slovak Republic

*Tel: +421 55 234 2328, Fax: +421 55 622 2124,
E–mail: maria.filkusova@gmail.com

Renáta Oriňáková

Department of Physical Chemistry, Faculty of Science, P.J. Šafárik University, Moyzesova 11, SK–04154 Košice, Slovak Republic

The electrochemical measurements were performed in a classical three–electrode electrochemical cell. The working electrode was a paraffin impregnated graphite electrode (PIGE) composite rod with a 6 mm diameter. The reference electrode was Ag/AgCl/3 mol/l KCl. A large-area platinum electrode served as the counter electrode. To obtain reproducible results, the surface of PIGE was mechanically renewed with emery paper, polished on glossy paper, then washed with distilled water and degreased with acetone. All electrochemical polymerization procedures and electrochemical experiments were performed using an EcaStat potentiostat/galvanostat, model 110 V (Istran, Slovak Republic).
The polypyrrole films were obtained in previously degassed aqueous solution containing 0.6mol/l, 0.06mol/l or 0.006mol/l pyrrole (Py) and 0.1mol/l NaCl at different pH (2.2, 5.6 and 11.2) by cyclic voltammetry (CV) between –800 mV and +1100 mV (vs. Ag/AgCl/3 mol/l KCl), beginning at -800 mV.

Scanning electron microscope (SEM) images were obtained with a TESLA BS 340. The operating voltage for the SEM was maintained at 20 kV throughout the analysis.

Results and discussion

CV Study

The PPy films were prepared by oxidation of the Py monomer on a PIGE. A typical cyclic voltammograms for different Py concentration at pH = 2.2; 5.6; 11.2 are shown in Fig. 1a, b, c. The highest cathodic and anodic currents were observed for highest Py concentration (0.6 mol/l) and lowest pH (2.2) (Fig 1a). The anodic current appearing in the first scan is associated with irreversible oxidation of the pyrrole monomer to produce the Py radical cation. From the second scan a new anodic current appears at gradually less positive potentials. On reverse scans a cathodic wave corresponding to reduction of the oxidized polymer was registered as a counterpart to the reversible anodic current. The overlapping of both oxidation processes in one broad anodic wave was observed for the highest Py concentration. The magnitude of the PPy redox currents increased and shifted to more positive potentials with the voltammetric cycle number indicating the build–up of electroactive PPy film.

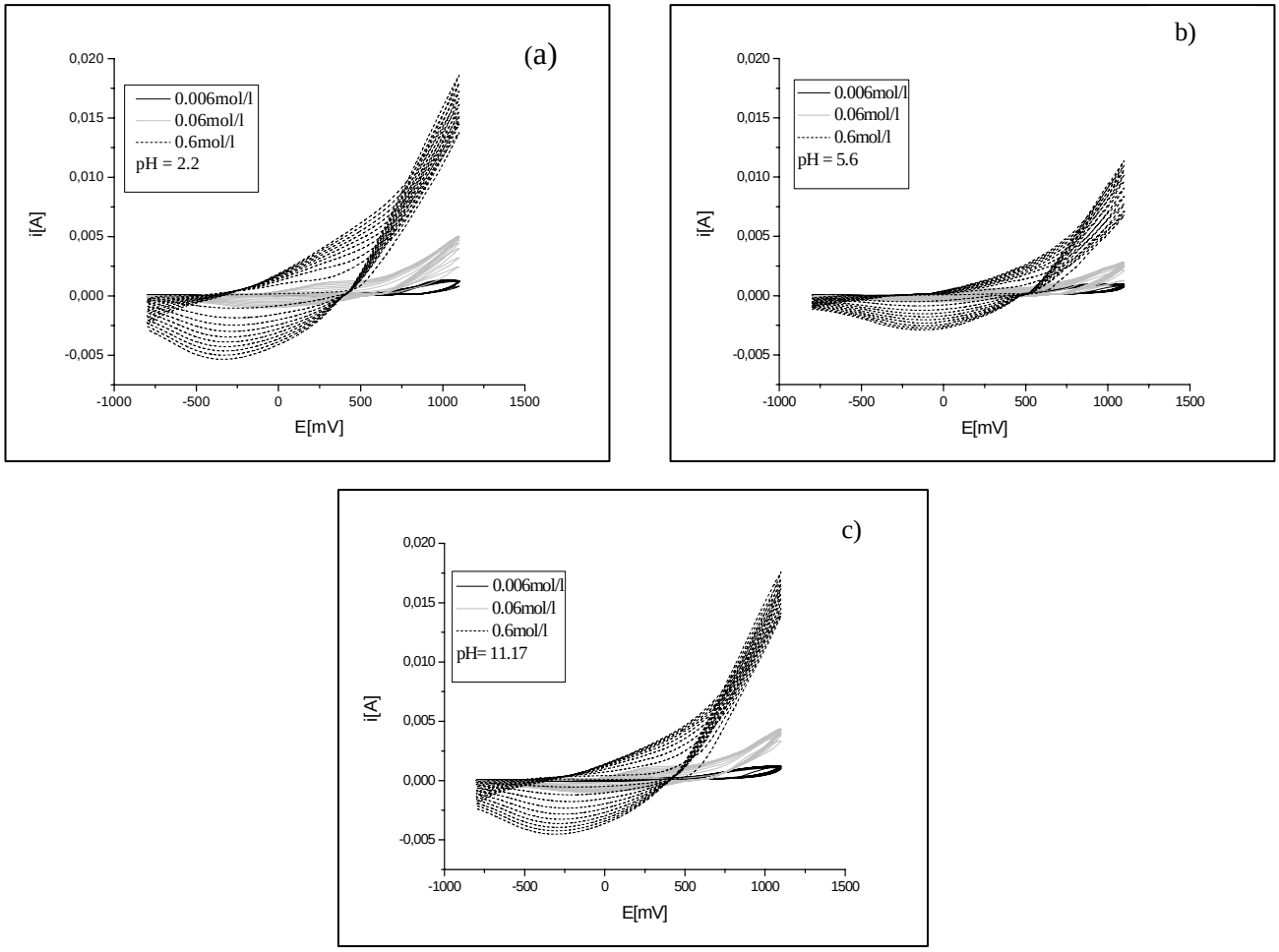

Figure 1: CV curves on PIGE electrode in 0.1 mol/l NaCl electrolyte for different concentration of Py at pH = 2.2 (a); pH=5.6 (b) and pH=11.2 (c)

From the CV studies it can be concluded that the low pH and high Py concentration favored the electropolymerization of Py. High concentration of hydrogen protons is assumed to facilitate the polymerization reaction

SEM Study

The properties of conducting polymers are strongly dependent on their morphology and structure. Scanning electron microscopy was used to investigate the morphology of the PPy films electrodeposited. Microscopic observation of PIGE surface after PPy deposition from alkaline media at lower Py concentration showed that the PIGE surface was not completely covered by the polymer. In acidic (Fig 2) and weakly acidic electrolyte, the PPy films present a cauliflower–like structure constituted by micro–spherical grains. In these cases, the electrode becomes rapidly covered by a black, homogeneous, thick and very adherent film. The cauliflower structure increased with an increased in Py concentration.

Conclusion

The use of an acidic electrolyte solution for the polypyrrole synthesis results in an enhanced electropolymerization and provides a high quality PPy film with potential application in biosensors.

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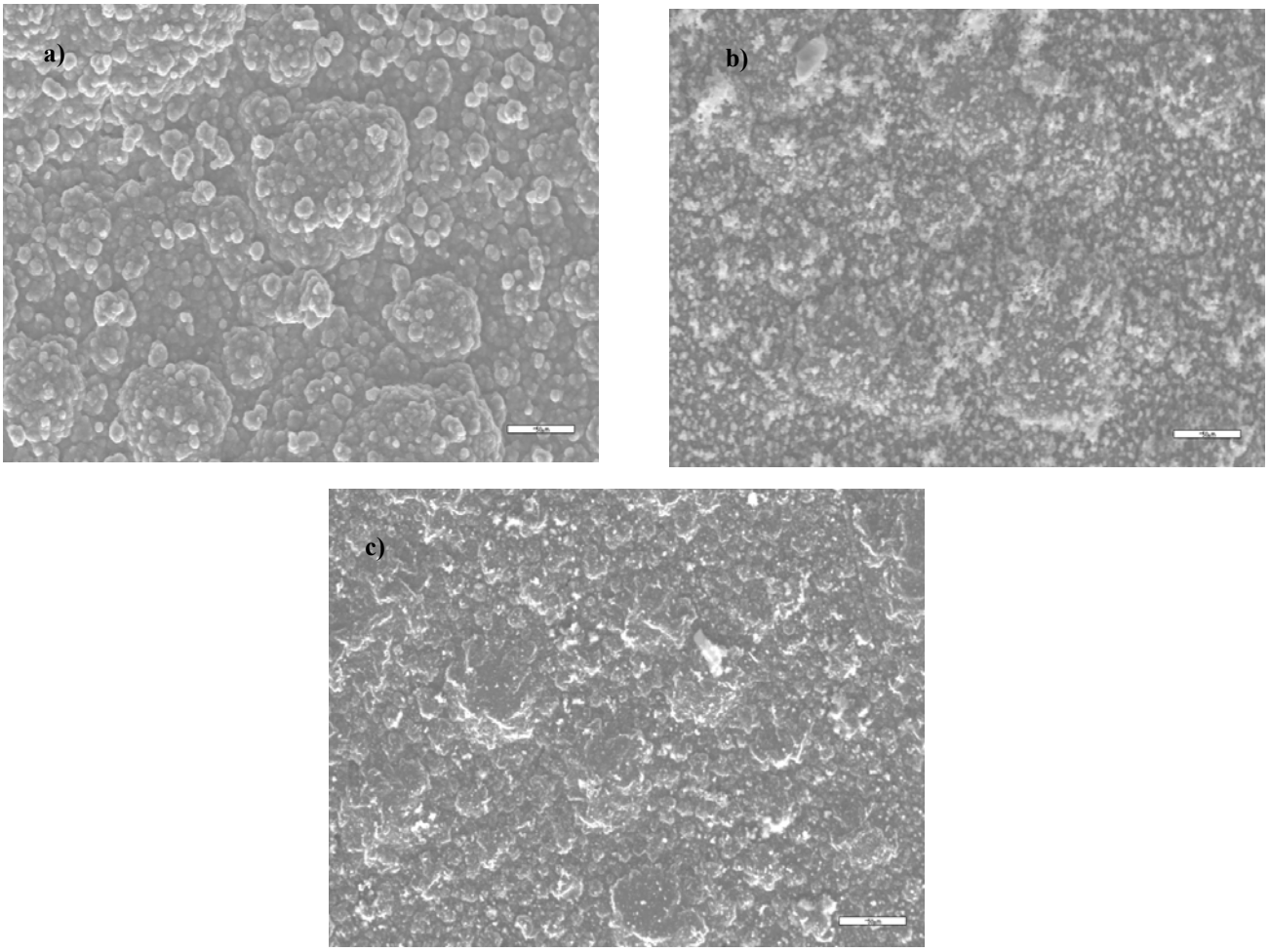

Figure 2: SEM image of PPy film deposited from the electrolyte with concentration 0.6 mol/l (a) 0.06 mol/l (b) 0.006 mol/l (c) at pH = 2.2

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