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REVIEW

Electropolymerization of diaminofluorene and its electrochemical properties

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Abstract Poly 2,7-diaminofluorene (PDAF)/Au modified electrode was prepared using 2,7-diamino fluorene (DAF) dissolved in acetonitrile (ACN) containing 0.1 M $LiClO_4$ using consecutive multisweep cyclic voltammetry (CV) and controlled potential electrolysis (CPE) techniques. Factors affecting the film formation, such as limits of potential cycling, sweep rate, number of sweeping cycles, monomer concentration, and also polymerization techniques were examined in detail. It was found that the optimum conditions, using a potentiodynamic technique on Au electrode as the working electrode, are by sweeping the potential between –200 mV and 800 mV at a sweep rate of 50 mV/s for 10 cycles using 5 mM DAF monomer solution. The obtained modified electrode was active only in acidic aqueous solutions (pH range from 0 to 2) and its activity was found to be pH dependent. PDAF was isolated and characterized using UV–vis, ^{1}HNMR and SEM analyses. The electrical conductivity was found to be 1.5×10^{-5} S cm^{-1}. An electropolymerization mechanism was proposed and discussed based on the obtained experimental data and molecular orbital calculations. The obtained modified electrode has been found to improve the electrochemical reversibility and decrease the overpotential of hydroquinone. PDAF/Au was stable chemically and electrochemically both in aqueous and organic solutions, making it an excellent candidate for sensing and/ or electrocatalytic applications.

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Contents

1. Introduction

The study and the synthesis of conducting organic polymers have been of great interest since the last few years. Thus, the first chemical synthesis of such compounds dates from 1973 $(SN)_x$ by walatka. Since this discovery several other polymers have been synthesized: the $(CH)_x$ by McDiarmid and poly.p.phenylene by Miller in 1979. At that time, Diaz and coworkers reported the first electrochemical synthesis of a conducting polymer in organic medium: the polypyrrole (Rault-Berthelot and Simonet, 1986; Rault-Berthelot et al., 1988, 1995a,b).

The polymers obtained by anodic oxidation of fluorene and their monosubstituted or 9,9-disubstituted parents have been already described as materials whose reversible electroactivity lies in a high potential range when compared with other conducting polymers such as polypyrrole or polythiophene (Bidan, 1985; Rault-Berthelot and Raoult, 2001; Rault-Berthelot et al., 1997). The reversible oxidation of the polyfluorenic matrices occurs between 0.8 and 1.2 V (reference Ag/Ag^+NO_3) 0.1 M in CH_3CN. Because of this rather positive potential, the polymers cannot be synthesized, studied and, therefore, used as electrode modifiers in aqueous media.

Anodic oxidation of aromatic amines has been extensively studied in both aqueous and nonaqueous solutions, and some general rules for reaction pathways have been obtained for mononuclear aromatic amines. For polynuclear aromatic amines (Rault-Berthelot and Roze, 1998; Ferraris et al., 1995; Leclerc, 2001; Cimrova et al., 1996; Groenendaal et al., 2003), however, little is known about the oxidation pathway. This is partially because in many cases the oxidation products film the electrode surface and no product could be isolated.

Since 1985, interest was shown in the anodic polymerization of fluorene derivatives. At the beginning, we demonstrated that polymers derived from fluorene, 9-substituted or 9,9-disubstituted fluorenes are electroactive materials possessing p- and n-doping processes. Their uses as electrochromic materials, electrode modifiers, in bi- or trilayer devices or as complexing materials were developed (Berkenkotter and Nelson, 1973; Nelson, 1974; Rault-Berthelot and Granger, 1999; Malitesta et al., 1990; Dong and Li, 1990). When the substitution was performed by cyano group, these matrices were demonstrated to present affinity towards metallic cations and metal-polymer composites were used as efficient electrode modifiers for electrocatalytic reactions (Imamoglu and Onal, 2004; Johansson et al., 2001; Millard, 2000; Yousef et al., 2001).

In this paper, electropolymerization of 2,7-DAF and characterization of its polymers were studied. PDAF films were easily prepared by the anodic oxidation of DAF monomer in acetonitrile. The electrochemical properties, conductivity and electroactivity of the resulting PDAF films were studied. PDAF was isolated and characterized using UV–vis, FT-IR and SEM analyses. The electrical conductivity was found to be 1.5×10^{-5} S cm^{-1}.

2. Experimental

2,7-Diaminofluorene (DAF), from Sigma and Fluka (97% pure), acetonitrile (99%), Lithium perchlorate (97%) and hydroquinone (90%) were obtained from Aldrich Co., and

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