

PREPARATION AND CHARACTERIZATION OF MONOMERIC AND POLYMERIC Ni-CYCLAM ETHYLENEDIOXYTHIOPHENE COMPLEX

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Introduction

The chemistry of the monomer 3,4-ethylenedioxythiophene (EDOT), has been investigated thoroughly in recent years with the aim of developing synthetic routes to prepare tailor-made functionalised EDOT derivatives.

Poly(3,4-ethylenedioxythiophene) (PEDOT) is a particularly stable polythiophene derivative. The electron rich oxygen-bridge in EDOT unit greatly lowers the redox potentials of both the monomer and the polymer compared to bare thiophene. In the present study, we have chosen cyclam, a square planar macrocyclic system, because it forms non-labile complexes of Ni(II) oxidation state while leaving coordination sites free for the formation of octahedral intermediates during reactions with electrophiles.

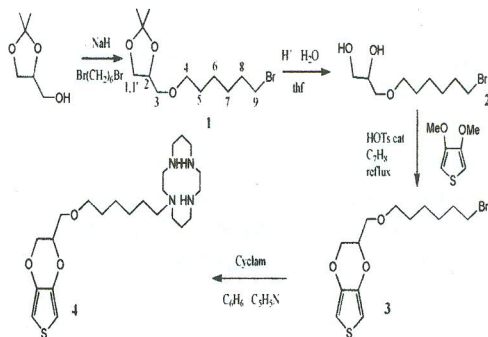
Materials and Methods

For the electrochemistry experiment, tetrabutylammonium tetrafluoroborate (Bu₄NBF₄) and anhydrous CH₃CN (99.97 %, electrochemistry grade) were used. All the solvents and electrolytes were handled under Schlenk conditions. The three-electrode electrochemical cell was used with separate compartments of the working (glassy carbon, 0.1 mm diameter), the auxiliary (Pt gauze), and Quasi reference (Polypyrrole coated Pt wire) electrodes. The potentiostat

was an Autolab PG-30 (EcoChimie, Netherlands). X-Ray Photoelectron Spectroscopy (XPS) was recorded in a Scienta ESCA 300 spectro meter (Daresbury laboratory, U.K). Thin film of copolymers were grown on the Au coated glass plate (11 mm x 11 mm) by repetitive scan cyclic voltammetry for XPS analysis

Results and Discussion

3,4 Dimethoxythiophene and complex **3** were prepared as described previously (Velauthamurty and Higgins, 2009). Treatment of **3** with 1,4,8,11-tetraazacyclotetradecane (cyclam) in benzene/ pyridine in the presence of catalytic amount of 4-(dimethyl- amino)pyridine gave a brown coloured semisolid which has been identified to be functionalized EDOT-cyclam **4**.



Scheme 1. Syntheses of ligand **4**

