



Electrochromic properties of organic-inorganic composite materials



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ABSTRACT

A new type of electroactive composite material based on tungsten trioxide (WO₃) and poly (tri(4-(2'-thienyl)) phenylamine) (P3TPA) was prepared. Using the WO₃/P3TPA as electroactive material, the electrochromic device was fabricated, and the electrochromic properties of WO₃/P3TPA were investigated. Compared with WO₃, the coloring switching time and the bleaching switching time of WO₃/P3TPA was decreased. The response speed became faster. The open-circuit memory of WO₃/P3TPA became better. The coloration efficiency of WO₃/P3TPA increased.

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1. Introduction

Electrochromic materials have attracted attention because of their potential applications in energy conservation [1]. Electrochromic devices (ECDs) are used in smart windows, automotive rear-view mirrors, displays, sunglasses, etc. [2]. The most important prerequisites for ideal EC materials include high initial transparency, large optical contrast between colored and bleach states, short switching time, and long-term cyclic stability [3]. EC materials can be divided into two large kinds on the basis of the component, organic and inorganic materials. Traditional metal oxide semiconductors, such as WO₃ [4], TiO₂ [5], MoO₃ [6], Nb₂O₅ [7,8], NiO [9] are some of the inorganic EC materials. At present, some other researchers also investigate organic EC materials due to their variety of visual coloration and versatile structural morphologies [10–12].

Among all the materials studied thus far, WO₃ has emerged as the most extensively studied material, not only for electrochromism, but also for a variety of other device applications. Deb in 1963 first reported on the electrochromic properties of WO₃ [13].

Subsequently, many other researchers expanded on the subject [14]. ECDs based on WO₃ show outstanding electrochromic properties and have been used on smart windows [15], automotive rear-view mirrors [16], and electrochromic displays [17]. Nevertheless, despite the plentiful amount of work that has been done on the coloration phenomena in thin films of various morphological WO₃, many contradictions still exist. Further commercialization is limited by the long switching times and low coloration efficiency.

Among all the electrochromic materials, the conjugated polymers have attracted much attention because of their low cost for easy processing, multicolor adjustability and mechanical flexibility [18,19]. Organic conjugated polymers such as polythiophene, polyaniline (PANI), polypyrrole (PPy), Poly(3,4-ethylene-dioxythiophene) (PEDOT) and their derivatives, have been extensively investigated [20] and many of them exhibit good electrochromic properties. As a typical organic electrochromic material, PANI and its derivatives are important organic conjugated polymers to ECDs because of short coloring and bleaching switching times, high optical contrast, and high coloration efficiency [21]. PEDOT and its derivatives are also a class of outstanding EC conjugated polymers due to their high coloration efficiency and ability of diverse colors [22,23]. However, the low electrochemical and thermal stability of conjugated polymers results in a relatively short lifetime. These disadvantages have hindered the further development of organic

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ECDs applications [24].

Usually, three methods are used to improve the performance of organic ECDs [25]. Firstly, designing and synthesizing new organic conjugated compounds; Secondly, developing new inorganic and organic composite materials, or inorganic heterojunction; Thirdly, electrolytes play an important role in determining the long-term stability of the devices, so finding appropriate electroplates is another way to improve the performance of ECDs [26]. The main characteristics of inorganic–organic composite electrochromic materials are the advantages of each component and the improved ECD performance. Recently, electrochromic inorganic–organic complexes such as TiO_2 /polythiophene, IrO_2 /polyaniline and WO_3 /polypyrrole have been studied [27–31].

In our previous work, we have investigated the electrochromic properties of the P3TPA denotes poly(tri(4-(2'-thienyl)) phenylamine) with triphenylamine and thiophene units (Scheme 1). It was found that the P3TPA exhibited reversible, clear color change from orange-yellow to blue on electrochemical doping and dedoping. However its electrochromic stability is poor. In this study, we prepared the inorganic–organic complex WO_3 /P3TPA. Using the WO_3 /P3TPA as electrochromic material, the electrochromic devices were prepared, and the electrochromic properties of WO_3 /P3TPA were investigated.

2. Experimental

2.1. Materials

Tungsten trioxide (WO_3) and $\text{PdCl}_2(\text{PPh}_3)_2$ were purchased from Beijing HWRK Chem Co., Ltd. 2-Thienylboronic acid, tetrabutylammonium perchlorate (TBAP) and tris(4-bromophenyl) amine were purchased from Alfa Aesar. Potassium carbonate (K_2CO_3), tetrahydrofuran (THF), acetonitrile (ACN), dichloromethane (DCM), and sodium chloride (NaCl) were purchased from Tianjin Hongyan Chem Co., Ltd. Lithium perchlorate (LiClO_4) was purchased from Aladdin Industrial Corporation. Sodium sulfate (Na_2SO_4) was purchased from Beijing Liudian Chem Co., Ltd. Petroleum ether was purchased from Guangdong JHD Co., Ltd.

2.2. Instruments

NMR spectra were obtained on a Bruker AVANCE-500 FT-NMR using tetramethylsilane as internal standard. IR spectra were recorded on a PerkinElmer Spectrum 100 FT-IR spectrometer. Mass spectra were recorded on a GCMS-QP 2010 mass spectrometer. The cyclic voltammogram (CV) and electropolymerization were carried out by the CHI750A electrochemical workstation. AFM images were obtained on a DI/MultiMode from Veeco.

The parameters of the ECD including optical contrast and open-circuit memory were measured on a Helios- γ UV–vis spectrophotometer. The coloration efficiency and switching time were

measured on the CHI750A electrochemical workstation.

2.3. Synthesis of tri (4-(2'-thienyl)) phenylamine (3TPA)

The synthesis of 3TPA is described in Scheme 1. 3TPA was prepared by Suzuki coupling reactions according to the procedure as followed: 1.45 g tris(4-bromophenyl)amine, 1.92 g 2-thienylboronic acid, 90 mg $\text{PdCl}_2(\text{PPh}_3)_2$, 150 ml THF and 100 ml K_2CO_3 (2 M) were added into a dry and clean 500 ml three-neck flask. The mixture was heated to reflux for 48 h, and then poured into a saturated solution of ammonium chloride and extracted with dichloromethane for three times. The organic phase was washed with brine and then dried over anhydrous sodium sulfate, filtered and removed the solvent. The solid residue was purified by silica-gel column chromatography to give a pale yellow crystalline solid. Yield: 59%.

2.4. Preparation of the WO_3 /P3TPA composite film

A 100–150 nm thick WO_3 film was deposited on a ITO-coated glass substrate by vacuum deposition (vacuum pressure 4×10^{-3} Pa).

At room temperature, the electropolymerization of 3TPA was carried out in an ACN solution of 5.0×10^{-2} M 3TPA and 0.1 M TBAP by repetitive cycling at scan rate of 100 mV s^{-1} . The reference electrode was the Hg/Hg^{2+} electrode; the counter electrode was the platinum wire, and the ITO-coated WO_3 was the working electrode. The P3TPA was directly coated onto WO_3 . Fig. 1 shows the cyclic voltammogram of the electropolymerization of 3TPA on the ITO-coated WO_3 electrode. The current density increased during the repeated potential scanning. The results suggest that the electropolymerization of 3TPA was achieved and P3TPA finally formed.

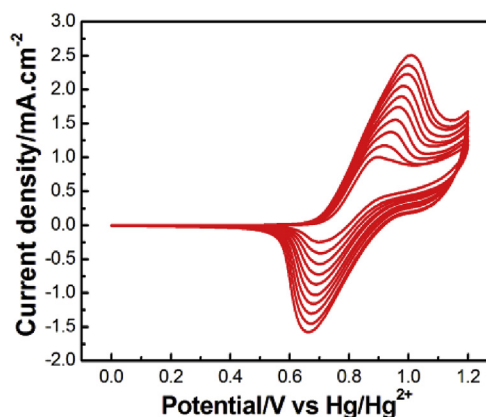
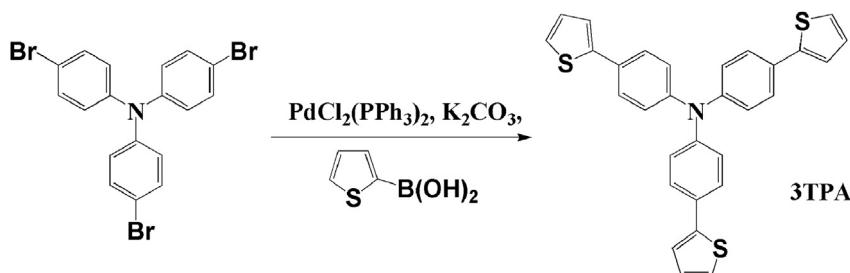


Fig. 1. The electropolymerization of 3TPA (5.0×10^{-2} M) at 100 mV/s for ten times in the ACN solution with 0.1 M TBAP.



Scheme 1. The Synthesis route of 3TPA.

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