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Preparation and characterization, stable bismaleimide-triarylamine polymers with reversible electrochromic properties

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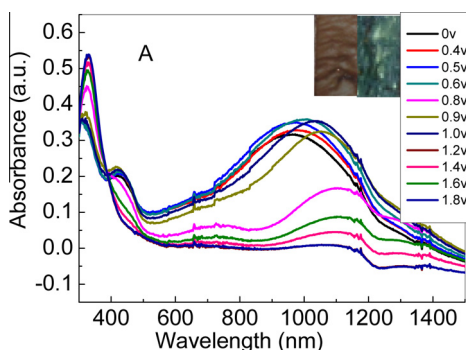
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HIGHLIGHTS

- It is a simple approach to obtain polyimides by Michael addition reaction.
- The prepolymers had excellent solubility in many common organic solvents.
- The polyimides film revealed good thermal stabilities and electrochromic characteristics.

GRAPHICAL ABSTRACT



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ABSTRACT

A series of novel polyimides were synthesized from bismaleimide containing different diaminetriaryl- amines by Michael addition reaction. The prepolymer is readily soluble in many common organic solvents, such as CHCl_3 , Tetrahydrofuran (THF) and N, N-dimethyl formamide (DMF). Prepolymers can be solution-cast into transparent, tough, and flexible films. These aromatic polyimides display good thermal stabilities, i.e. 5% weight-loss temperatures in excess of 200 °C under nitrogen. All obtained polyimides revealed excellent stability of electrochromic characteristics, changing color from original yellowish to green. The energies of the highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) levels of the investigated the polymers were estimated by experimental method are in the range of -4.78 eV to -4.98 eV and -1.64 eV to -2.09 eV vs the vacuum level, respectively. All the polymer films reveal good electrochemical and electrochromic stability under repeatedly switching electrode voltages, with coloration change from the yellow neutral state to green oxidized state.

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Introduction

Electrochromic materials exhibit a reversible optical change in absorption or transmittance upon electrochemically oxidized or

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reduced process [1]. This interesting property led to the development of many technological applications such as display panels, camouflage materials, variable reflectance mirrors, and variable transmittance windows [2]. Hitherto, a wide variety of electrochromic materials showing high coloration efficiencies, low operating voltage, and fast switching capabilities have been developed, which can be classified into several distinct categories such as

metal oxides (tungsten trioxide (WO₃) or iridium dioxide (IrO₂)), mixed-valence metal complexes (Prussian blue), small organic molecules (viologens, bipyridinium, and phthalocyanines), and conjugated polymers [3]. The most frequently studied electrochromic candidates are organic conducting polymers and metal oxides [4]. In the available electrochromic π -conjugated organic polymers, poly(3,4-ethylenedioxythiophene) (PEDOT) in particular, has stimulated much interest as a cathodic electrochrome due to its fast switching between deep-blue and pale-blue hues, high coloration efficiency, optical contrast ratio, long-term redox stability, and wide range of colors [5,6].

Triarylamine (TPA) derivatives are well-known for photo and electroactive properties that find optoelectronic applications as photoconductors, hole transporters, and light emitters [7]. The characteristic structure feature of triarylamine is the nitrogen center, the electroactive site of TPA, which is linked to three electron-rich phenyl groups in a propeller-like geometry [8]. In recent years, TPA-based high-performance polymers such as polyamides and polyimides with attractive electrochromic properties have been reported by Liou [9,10]. The electrochromic function of these polymers originates from the electroactive TPA moieties, which can reversibly be oxidized as long as the para-position of the phenyl rings is protected. We also demonstrated that polyazomethines containing bulky, propeller-shaped TPA unit were amorphous which had good solubility in organic solvents, and thermal stability.

Aromatic polyimides are widely used in the semiconductor and electronic packaging industry because of their outstanding thermal stability, good insulation properties with low dielectric constant, good adhesion to common substrates, and superior chemical stability [11,12]. However, their processability is difficult owing to the poor solubility and high softening and/or melting temperature [12–14]. In order to overcome this drawback, either bulky lateral substituents, flexible alkyl side chains, unsymmetric, alicyclic or kinked structure has been attached along the backbone [15]. One of the common approaches for increasing solubility and processability of polyimides without sacrificing high thermal stability is the introduction of bulky, packing disruptive TPA groups into the polymer backbone [16].

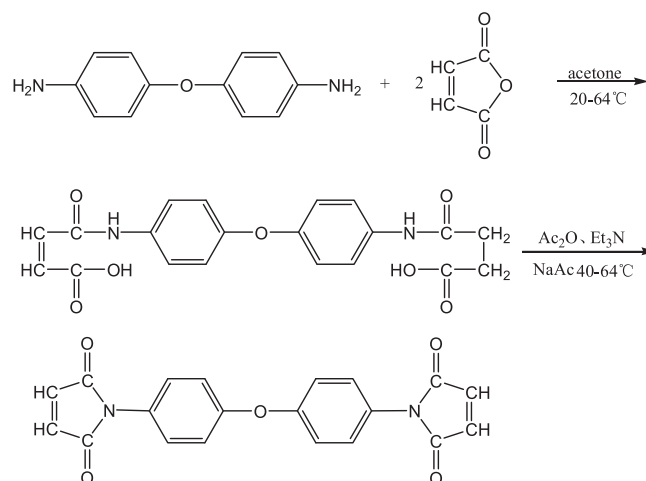
Among polyimides, bismaleimide resins have attracted much attention in the fields of advanced composites because of their excellent processing characteristics, outstanding thermomechanical without the formation of volatile byproducts and flammability behaviors in the finally cured state [17–22]. Although a variety of bismaleimide derivatives containing rigid or condensed aromatic rings were synthesized for the preparation of bismaleimide resins, studies on their electrochemical behavior were scarce.

In the present article, a novel series of polyimides were synthesized from bismaleimides with six triarylamine derivatives by Michael addition, as well as the characterizations were carried out by means of Fourier transform infrared spectroscopy (FT-IR), X Ray diffraction (XRD), thermo gravimetric analyzer (TGA) and scanning electron microscope (SEM). The general properties such as solubility and thermal properties are reported. The six materials have a good film forming ability, and are insoluble in acetonitrile. All the polymer films prepared by casting solution onto an ITO reveal good electrochemical and electrochromic stability under repeatedly switching electrode voltages, with coloration changing from the yellow neutral state to green oxidized state.

Experimental

Materials

Maleic anhydride, 4-(4-aminophenoxy) aniline, triethylamine, acetic anhydride were purchased from Shanghai Sinopharm Co. and Acros Co. Acetic anhydride and DMF, THF were vacuum



Scheme 1. Synthesis routes of BMI monomer.

distilled and triethylamine was distilled before use. Lithium perchlorate (LiClO₄) was dried under vacuum at 120 °C for 36 h. N, N'-diphenyl-N, N'-bis (4-aniline)-p-phenylenediamine, N, N'-diphenyl-N, N'-bis (4-aniline) biphenyl diamine, N, N'-bis (α -naphthene)-N, N'-bis (4-aniline) biphenyl diamine, N, N'-bis(β -naphthene)-N, N'-bis (4-aniline)-p-phenylenediamine, N'-4-(9H-carbazol-9-yl) phenyl -N'-(4-aminophenyl)benzene-1,4-diamine, N'-(4-aminophenyl)-N'-(4-(diphenylamino) phenyl) benzene-1,4-diamine were prepared according to the Ref. [23].

Monomer synthesis

N, N'-bismaleimide-4,4'-diphenyl ether (BMI) was prepared by the condensation of 2.0476 g (0.021 mol) of maleic anhydride with the required equivalent ratio of 1.8235 g (0.009 mol) of corresponding aromatic diamines by a widely used method [17]. The resulting BMIs were purified by recrystallization from chloroform/methanol (shown in Scheme 1).

FT-IR/cm⁻¹ (KBr): 3080 cm⁻¹(C=C–H stretching absorption), 1641 cm⁻¹(C=C absorption), 1780, 1726 cm⁻¹ (imide ring absorption), 1461, 1488, 1503, 1586, 1622 cm⁻¹ (aromatic ring skeleton vibration absorption), 1226, 1281 cm⁻¹ (aryloxy stretching absorption).mp:160–168 °C.

Pre-polymer synthesis

The synthesis of pre-polymer (PPa) was used as an example to illustrate the general synthetic procedure. Diaminetriarylamine 1 was charged into a 50 mL three-necked flask fitted with a paddle stirrer and nitrogen inlet; and the flask was maintained in an oil bath. After diaminetriarylamine dissolved evenly in DMF, BMI was added under nitrogen. Then, the mixture solution of BMI and diamine (1:1 mol/mol) in DMF was stirred at 80–90 °C for 20–30 h until a homogeneous solution was formed. DMF was removed in vacuum at 45–50 °C to give powder of prepolymer. Following the same procedure, starting from BMI and diamines b–f, the prepolymers PPa–f were prepared (as shown in Scheme 2).

Film-forming curing

A solution of the prepolymer (PPa–f) in THF was cast on an ITO conductive glass. The solvent was evaporated in a vacuum oven at 60 °C overnight. Then the thermally cured films Pla–f were obtained from the prepolymers PPa–f by programmed temperature

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