



New triphenylamine-based poly(amine-imide)s with carbazole-substituents for electrochromic applications

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ABSTRACT

A new carbazole-derived triphenylamine-containing diimide-diacid monomer (5), 4,4'-bis(trimellitimidido)-4''-N-carbazolytriphenylamine, is prepared by the condensation of 4,4'-diamino-4''-N-carbazolytriphenylamine (4) and two molar equivalents of trimellitic anhydride (TMA). A series of new poly(amide-imide)s (PAIs) 7a–7d with carbazole-substituted triphenylamine units are prepared by direct polymerization from the new diimide-diacid (5) and various aromatic diamines (6a–6d). The PAIs shows high glass transition temperature between 269 and 297 °C, and high 5% weight loss temperature between 526 and 561 °C under nitrogen environment. Cyclic voltammograms of the PAIs films, which are casted onto the indium–tin oxide (ITO)-coated glass substrate, exhibit two reversible oxidation redox couples at 1.05–1.08 V and 1.38–1.46 V under an anodic sweep. The PAI-7a film reveals excellent stability of electrochromic characteristics for the radical cations generated, changing color from original pale yellowish neutral form to the green and then to dark blue oxidized forms. Furthermore, the anodically coloring of PAI-7a shows high coloration efficiency (CE = 205 cm²/C), high contrast of optical transmittance change ($\Delta T = 80\%$ at 776 nm) and long-term redox reversibility.

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1. Introduction

Electrochromic materials, comprised redox-active species, exhibit significant, lasting, and reversible changes in color upon reduction or oxidation [1]. Color changes are commonly between a transparent state, where the chromophore only absorbs in the UV region, and a colored state, or between two colored states in a given electrolyte solution. The potential applications of electrochromic materials include optical switching devices, automatic anti-glazing mirrors, smart windows, large-area information panels, electronic papers and chameleon materials

[2–9]. The properties of electrochromism are found not only in conducting polymers, but also in variety of organic and inorganic materials. The uses of conjugated polymers as active layers in electrochromic devices become popular because of the outstanding electrochromic properties such as fast switching time, ease of synthesis, and wide range of colors, which can be achieved through simple chemical synthesis [10]. The difficulties in achieving satisfactory values for all these parameters at the same time stimulate the development of new methods of electrochromic films preparation, new materials and new components for the devices [11].

Aromatic polyimides (PIs) are well known as useful high performance materials because of their excellent thermal stability, mechanical, electric and optical properties [12–14]. However, the widespread applications of PIs are often limited by processing difficulties because of their

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poor solubility and high processing temperature, which are caused by rigid polymer backbones and the strong inter-chain interaction [15,16]. To overcome these problems, typical approaches include the introduction of flexible linkages, kinked or unsymmetrical structures, bulky packing-disruptive units, and bulky lateral groups into the polymer backbone. In our previous work several soluble fluorinated poly(ether imide)s with different pendant groups have been explored, and the introduction of bulky trifluoromethyl group into polymer backbones result in an enhanced solubility and optical transparency [17]. Another common approach for increasing solubility and processability of PIs without sacrificing high thermal stability is the development of various copolyimides. Replacement of polyimides by copolyimides such as poly(amide-imide)s (PAIs) may be useful in modifying the intractable character of polyimides [18,19]. Because of the PAIs possess both amide and imide groups in their mainchain. This class of polymers combines the advantages of polyamides and polyimides and offers good comprehensive properties such as high thermal property and processability.

In recent years, triarylamine-based PAIs with attractive electrochromic properties have been reported in G.S. Liou's laboratory [20]. Triarylamine derivatives are well-known for their electroactive and photoactive properties, which make them be used as photoconductors, hole-transporters, and light-emitters. Polymers bearing triarylamine units are being received considerable interest as ideal hole-transporters, and light-emitting diodes (OLEDs) because of the strong electron-donating and hole-transporting/injecting properties of triarylamine units [21]. What's more, triarylamine can be easily oxidized to form stable radical cations, and the oxidation process is always associated with a strong change of coloration [22]. Except triarylamine, carbazole is another well-known hole-transporting and light-emitting unit. From structural point of view, carbazole differs from diphenylamine in its planar structure because it can be further imagine as the bonded diphenylamine; the thermal stability of materials with the incorporation of carbazolyl units therefore is improved. In addition, carbazole could be easily functionalized at its 3,6-, 2,7-, or *N*-positions and then covalently linked to polymeric systems, both in the main chain as building blocks and in a side chain as pendant groups [23]. Thus, incorporation of three-dimensional, packing-disruptive triarylamine and carbazole units into the poly(amide-imide) backbone not only enhance the solubility but also form some new electronic factions of poly(amide-imide) such as electrochromic characteristics.

In some reports, the triphenylamine cationic radical of the first electron oxidation is not stable. However, the coupling reactions that afford stable cationic radical are greatly prevented when the phenyl groups are incorporated by electron-donating substituents at the para position of triarylamines [24,25]. In this contribution, we reported the synthesis of novel carbazole-derived triphenylamine-containing aromatic dicarboxylic acid monomer, 4,4'-bis(trimellitimido)-4''-*N*-carbazolytriphenylamine (5), and its derived PAIs bearing electron-rich pendent triphenylamine and carbazole groups. The PAIs exhibited good sol-

ubility and excellent thermal properties. The electrochemical and electrochromic properties of these films prepared by casting the PAIs onto an indium-tin oxide (ITO)-coated glass substrate are also described herein.

1.1. Measurements

IR spectra (KBr) were measured on a Nicolet Impact 410 Fourier transform infrared spectrometer. ¹H NMR and ¹³C NMR were recorded on a Bruker 510 NMR spectrometer (500 MHz) with tetramethyl silane as a reference. Elemental analyses were performed on an Elemental Analyses MOD-1106. Gel permeation chromatograms (GPC) using polystyrene as a standard were obtained on a Waters 410 instrument with a DMF as an eluent at a flow rate of 1 mL/min. Inherent viscosity was determined on an Ubblohe viscometer in thermostatic container with the polymer concentration of 0.5 g/dL in NMP at 30 °C. Differential scanning calorimetry (DSC) measurements were performed on a Mettler Toledo DSC 821^e instrument at a heating rate of 20 °C/min under nitrogen. Thermal gravimetric analyses (TGA) were determined in nitrogen atmosphere using a heating rate of 10 °C/min and polymers were contained within open aluminum pans on a PERKIN ELMER TGA-7. The mechanical tests in tension were carried out using a SHIMADZU AG-I at a constant crosshead speed of 10 mm/min. Wide-angle X-ray diffraction (WAXD) measurements were carried out at room temperature using a Rigaku/max-rA diffractometer equipped with a Cu K α radiation source. Voltammograms were presented with the potential pointing to the left and with increasing anodic currents pointing downwards. Cyclic voltammetry was performed with the use of a three-electrode cell in which ITO (polymer films area about 0.4 cm \times 2.5 cm) was used as a working electrode. A platinum wire was used as an auxiliary electrode. All cell potentials were taken with the use of a homemade Ag/AgCl, KCl (sat.) reference electrode. The spectroelectrochemical cell was composed of a 1 cm cuvette, ITO as a working electrode, a platinum wire as an auxiliary electrode and Ag/AgCl reference electrode. Absorption spectra were measured by a Shimadzu UV 3101-PC spectrophotometer.

2. Experimental

2.1. Materials

All the reagents were purchased from commercial sources and used as received. Carbazole (Acros), 4-fluoronitrobenzene (Acros), trimellitic anhydride (TMA) (TCI), triphenyl phosphite (TCI), 10% palladium on charcoal (Pd/C, TCI), and hydrazine monohydrate (TCI) were used as received. *N,N*-dimethylformamide (DMF), *N*-Methyl-2-pyrrolidone (NMP) and pyridine were purified by distillation under reduced pressure over calcium hydride and stored over 4 Å molecular sieve. 1,4-Bis(4-amino-2-trifluoromethylphenoxy) benzene (6FAPB) was synthesized in our laboratory according to the literature. 4,4'-Oxydianiline (ODA) (TCI) and 1,4-bis(4-aminophenoxy) benzene (APB)

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