



# Synthesis and study of morphological, optical and electrical properties of new organic semi conducting polymers containing isosorbide pendant group



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## ARTICLE INFO

### Article history:

Received 21 July 2016

Received in revised form 8 September 2016

Accepted 14 September 2016

Available online 27 September 2016

### Keywords:

Semi-conducting polymer

Isosorbide

Optical properties

Thin films

Space-charge limited current (SCLC)

## ABSTRACT

Two isosorbide containing poly(phenylene vinylene-arylene vinylene)s have been synthesized via Wittig polycondensation: **PPVIs-Carb** (arylene: carbazole) and **PPVIs-Naph** (arylene: naphthalene). The macromolecular structures were characterized by NMR and FTIR spectroscopies. The polymers are soluble in common organic solvents and show good film-forming abilities thanks to the presence of the polar isosorbide side groups. These organic materials showed an amorphous behavior. A higher polarity of the surface was obtained for **PPVIs-Naph**. The both polymer films are photoluminescent and exhibit an optical gap of 2.3 eV. The carbazole-based polymer emits in yellow-green while a green-yellow emission was obtained in the naphthalene containing polymer with higher fluorescence quantum efficiency. The HOMO/LUMO energy levels were determined by cyclic voltammetry and showed higher ionization potential and electronic affinity for **PPVIs-Carb**. The study of the ITO/polymer thin film/Al devices showed a space-charge limited current transport mechanism with charge carrier mobility in order of  $10^{-8} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ .

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## 1. Introduction

Organic semiconductors have gained a lot of interest due to their potential electronic applications [1–3]. Poly (*p*-arylene-vinylene)s are interesting class of  $\pi$ -conjugated polymers thanks to their luminescent properties [4]. Since, the discovery of the first light emitting diodes based on PPV, different polymers archetype have been developed for different purposes [5–8]. Indeed, several studies were investigated for elaboration of new conjugated structures with improved properties [9]. The generic molecular architecture of poly (*p*-arylene-vinylene)s is comprised of a semi-rigid conjugated backbone, responsible for charge conductance, and flexible side groups that simplify their value in the electronic devices. In fact, abundant derivatives of PPV have been reported with various substituents such as alkoxyl [10], silyl [11], phenyl [12], fluorenyl [13], halide [14], or cyano groups [15].

In our previously work, we have reported the synthesis and optoelectronic properties of a poly(phenylene vinylene anthrylene vinylene) semi-conducting polymer containing isosorbide in the side chain (**PPVIs-An**, Scheme 2) [16]. The introduction of isosorbide group was showed to significantly improve the optical behavior of such organic semi-conductors. Hence, we aim to continue the study of the isosorbide-containing semi-conducting polymers. In particular, the effect of the chromophore structure on the optical and electrical properties will be evaluated. Herein, we report new analogues of the **PPVIs-An** in which we replace the anthryl central group by naphthyl moiety (**PPVIs-Naph**, Scheme 2) or by carbazolyl unit (**PPVIs-carb**, Scheme 2). In fact, naphthalene based-materials have remarkable inherent properties such as intense fluorescence, strong  $\pi$ -stacking, and good chemical stabilities. Therefore, the naphthalene moiety has been explored and used as fluorophore in various photoluminescent materials [17] and organic semiconductors [18]. Besides, carbazole based-materials have been extensively studied for their original optoelectronic properties and air-stability [19]. They have been used as hole-transporting materials in diversity areas, such as organic

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photoconductors [20], organic light-emitting diodes [21], nonlinear optics [22] and organic field-effect transistors [23].

## 2. Experimental

### 2.1. Materials and measurements

Isosorbide (98%, Acros Organics, France), 4-toluenesulfonyl chloride (98%, Sigma-Aldrich, France), 4-methoxyphenol (99%, Acros Organics, France), ethyl bromide (98%, Acros organics, France), hexyl bromide (98%, Acros organics, France), anthracene (97%, Fluka, Taufkirchen, Germany), carbazole (>95%, Sigma-Aldrich, France), 2,6-dimethylnaphthalene (99%, Sigma-Aldrich, France), *N*-bromosuccinimide (99%, Sigma-Aldrich, France), benzophenone (>99%, Sigma-Aldrich, France), potassium carbonate (99%, Acros Organics, France), triphenylphosphine (99%, Acros Organics, France), sodium hydride (60% dispersion in mineral oil, Sigma-Aldrich, France), paraformaldehyde (96%, Acros Organics, France), potassium carbonate (99%, Acros Organics, France), sodium acetate anhydrous (99%, Sigma-Aldrich), triphenylphosphine (99%, Acros Organics, France), sodium hydride (60% dispersion in mineral oil, Sigma-Aldrich, France), sodium (Sigma Aldrich, France), pyridiniumchlorochromate (PCC) (98%, Acros Organics, France), phosphorus (V) oxychloride (99.99%, Sigma-Aldrich, France), acetic acid glacial (99, 85%, Sigma-Aldrich, France) and hydrochloric acid (37%, Sigma-Aldrich, France) were used as received. Tetrahydrofuran (THF) was dried over Na/benzophenone and freshly distilled before use. Toluene was dried over Na under argon; dichloromethane was dried over CaH<sub>2</sub> under argon. All the solvents used in this study were purchased from Sigma-Aldrich (France). The conditions and measurements used for physico-chemical characterization (structural, thermal, optical and electrochemical) of polymers are the same as those used in our previously reported works [16,24].

### 2.2. Synthesis of monomers

The synthesis and structural characterization of the products (a), (b), (c), (d1, d2), (e1, e2), (f) (g) and the 1,4:3,6-dianhydro-2-O-hexyl-5-[3,6-diformyl-4-methoxyphenoxy]-5-deoxy-L-itol (M3) have been carried in our previously work [16]. The chemical structures are presented in Scheme 1.

#### 2.2.1. Synthesis of the 1,4:3,6-dianhydro-2-O-alkyl-5-[3,6-triphenylphosphoniomethyl-4-methoxyphenoxy]-5-deoxy-L-itol dichloride (M1)

A solution dichloromethyl compound (e1) (10 mmol) and triphenylphosphine (5.82 g, 22 mmol) in anhydrous toluene (50 mL) was stirred and heated at reflux for 24 h in argon atmosphere. After cooling, the resulting white precipitate was filtered, washed with diethyl ether several times and dried under vacuum.

**M1:** R = ethyl; yield: 90%; m.p: 157 °C; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>, δ): 7.76–7.59 (m, 30H, PPh<sub>3</sub>), 6.90 (s, 1H, Ar-H), 6.79 (s, 1H, Ar-H), 5.43–5.31 (m, 2H, CHP), 5.07–5.02 (m, 2H, CHP), 4.24–4.23 (m, 1H, H<sub>5</sub>), 3.99 (m, 1H, H<sub>2</sub>), 3.80–3.44 (m, 7H, H<sub>1</sub><sup>a</sup>, H<sub>3</sub>, H<sub>4</sub>, H<sub>6</sub><sup>a,b</sup>, OCH<sub>2</sub>), 2.98–2.82 (m, 4H, H<sub>1</sub><sup>b</sup>, OCH<sub>3</sub>), 1.36 (t, <sup>3</sup>J = 6.9 Hz, 3H, OCH<sub>2</sub>CH<sub>3</sub>); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>, δ): 151.50, 148.59, 135.04, 135.00, 134.97, 134.29, 134.19, 134.06, 130.27, 130.18, 130.10, 130.01, 118.36, 117.29, 117.16, 116.79, 115.72, 85.44, 84.36, 83.06, 81.05, 72.11, 71.50, 65.07, 55.15, 25.20, 25.17, 24.94, 24.54, 15.35; FTIR (cm<sup>-1</sup>): 1112 (s, P–C stretching), 501 (s, P–Cl stretching).

#### 2.2.2. Synthesis of the 2,6-bis(bromomethyl)naphthalene (h)

A mixture of 1.47 g (8.0 mmol) of *N*-bromosuccinimide (NBS) and 0.62 g (4.0 mmol) of 2,6-dimethylnaphthalene were dissolved

in 50 mL of CCl<sub>4</sub> under nitrogen atmosphere. Then a catalytic quantity of benzoyl peroxide (BPO) was added which was then heated at 90 °C for 3 h under argon atmosphere. After that, the crude reaction mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> and washed by NaOH solution. The organic layer was washed with water, dried over anhydrous MgSO<sub>4</sub> and the solvent was evaporated; yield 63%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>, δ): 7.96–7.29 (m, 6H, aromatic H), 4.62 (s, 4H, –CH<sub>2</sub>Br).

#### 2.2.3. Synthesis of the 2,6-bis(triphenylphosphoniomethyl)naphthalene dibromide (M2)

A solution of 3 mmol of compound (h) and 7.5 mmol of triphenylphosphine in 20 mL of DMF was dissolved and reacted at room temperature for 12 h. The resulting reaction was filtered and washed into diethyl ether. After filtration and vacuum drying, diphosphonium salt monomer, compound M<sub>2</sub>, was obtained as a white solid; yield 70%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>, δ): 7.96–7.39 (m, 36H, aromatic H), 5.26 (d, 4H, –PCH<sub>2</sub>–).

#### 2.2.4. Synthesis of the 9-hexylcarbazole (i)

A solution of carbazole (10 mmol) and sodium hydride NaH (15 mmol) in dimethylformamide (DMF) was dissolved at room temperature. Then the hexyl-bromide (15 mmol) was added dropwise into the reaction mixture and heated at 60 °C for 24 h. After cooling, resulting mixture was poured into distilled water and extracted with chloroform. The organic layer was washed several times with distilled water and dried over anhydrous magnesium sulfate. The obtained solution was then evaporated and purified by recrystallization from ethanol. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>, δ): 8.09 (2H, d, H<sub>cz</sub>), 7.35–7.47 (4H, m, H<sub>cz</sub>), 7.18–7.24 (2H, m, H<sub>cz</sub>), 4.25 (2H, t, NCH<sub>2</sub>), 1.84 (2H, m, NCH<sub>2</sub>CH<sub>2</sub>), 1.29 (6H, m, 4 × CH<sub>2</sub>), 0.84 (3H, t, CH<sub>3</sub>).

#### 2.2.5. Synthesis of the 3,6-diformyl-N-hexylcarbazole (M4)

To (1 mol) of DMF at 0 °C, (1 mol) of phosphorus oxychloride was added dropwise. The mixture was allowed to warm up to room temperature, and (0.16 mol) of hexylcarbazole (i) was added. After 24 h at 90 °C, the mixture was then poured into 500 mL of water and extracted with chloroform. The organic layer was washed several times with distilled water and dried over MgSO<sub>4</sub>. The obtained solution was then evaporated and purified by column chromatography with hexane/ethyl acetate (70: 30). M.p: 142–143 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>, δ): 10.14 (2H, s, –CHO), 8.67 (2H, t, H<sub>4,5</sub>), 8.08 (2H, dd, J<sub>1,2</sub> = 8.5 Hz, H<sub>2,7</sub>), 7.55 (2H, d, H<sub>1,8</sub>), 4.39 (2H, t, NCH<sub>2</sub>), 1.91 (2H, quintet, NC–CH<sub>2</sub>–), 1.28–1.43 (6H, m, (CH<sub>2</sub>)<sub>3</sub>) and 0.86 (3H, t, CH<sub>3</sub>).

### 2.3. Polymer synthesis

A stirred mixture of an equimolecular amount of diphosphonium salt (M<sub>1</sub>, M<sub>3</sub>) (1 mmol) and dialdehyde monomers (M<sub>2</sub>, M<sub>4</sub>) in 10 mL of anhydrous THF, 10 mL of a 0.5 M *t*-BuOK solution in THF (5 mmol) was added dropwise at room temperature under an argon atmosphere. After 24 h at room temperature, the reaction mixture was heated at reflux during 4 h. Then, after cooling, an aqueous hydrochloric acid (3 M) was added, poured into water and extracted with chloroform. The organic phase was washed several times with water, concentrated and then precipitated into methanol.

**PPVis-Naph:** yield: 65%; aspect: orange powder; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>, δ): 7.90–6.50 (br m, aromatic and vinylic H), 5.00–3.10 (m, H<sub>1</sub>, H<sub>2</sub>, H<sub>3</sub>, H<sub>4</sub>, H<sub>5</sub>, H<sub>6</sub>, OCH<sub>2</sub>, OCH<sub>3</sub>), 2.40 (s, CH<sub>3</sub> terminal group), 1.6–1.00 (m, OCH<sub>2</sub>(CH<sub>2</sub>)<sub>4</sub>), 1.50–0.50 (m, CH<sub>3</sub>); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>, δ): 156.09, 143.0, 140.57, 129.70, 129.40, 127.46, 126.40, 124.15, 114.82, 85.77, 83.36, 82.88, 76.45, 71.70, 69.84, 55.68, 31.63, 30.20, 17.60, 15.10, 14.06; FTIR (cm<sup>-1</sup>): 3054 (w,

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