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Cross-linkable fluorenyl-substituted aromatic amines for polymeric hole transporting networks

S. Lengvinaite^a, J.V. Grazulevicius^a, B. Zhang^b, Z. Xie^b, S. Grigalevicius^{a,*}

^a Department of Organic Technology, Kaunas University of Technology, Radvilenu Plentas 19, LT50254 Kaunas, Lithuania ^b State Key Laboratory of Polymer Physics and Chemistry, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun 130022, China

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ABSTRACT

Fluorenyl-substituted aromatic amines containing reactive oxetanyl groups have been synthesized. Full characterization of their structure by NMR-, IR- and mass spectrometry is presented. The synthesized materials were examined by various techniques including differential scanning calorimetry, thermogravimetry, electron photoemission spectrometry and xerographic time of flight technique. The electron photoemission spectra of the layers of the derivatives showed ionization potentials of 5.4–5.65 eV. These derivatives have been tested as hole transporting materials in bilayer OLEDs with Alq₃ as the emitter. The device with derivative containing benzidine core exhibited the best overall performance with turn-on voltage of ~ 2.5 V, a maximum luminance efficiency of 4 cd/A and maximum brightness of about 6000 cd/m². The derivative was also used for the preparation of cross-linked hole transporting layers by photoinduced polymerization. The layers obtained were also tested in light emitting diodes. The devices demonstrated maximum photometric efficiency of 1.65 cd/A and maximum brightness of about 3000 cd/m^2 .

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摺

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1. Introduction

Organic light- emitting diodes (OLEDs) based on organic small molecules and polymers have attracted much attention because of their potential use in flat panel displays and lighting [1]. Efficient OLEDs can be obtained only by building multilayer structures [2-4]. The main difficulty in the preparation of such devices by spincoating is the solubility of the material which forms the bottom layer onto which the top layer has to be cast, because most organic semiconductors are soluble in the same solvents. One approach that has been employed to circumvent this problem is the appliance of bi-functional electro-active derivatives, which could be converted into insoluble networks by cross-linking reactions. Several series of photo-cross-linkable polymers and monomers for the fabrication of OLEDs have been reported [5-9]. Among the derivatives earlier studied, triphenyldiamine (TPD) core having derivatives were mostly used. For the purpose of developing of organic layers with new optoelectronic properties, we have been studying the synthesis and applications of new photo-cross-linkable derivatives with fluorenyl fragments. Films of the compounds should demonstrate suitable ionization potentials and could be used as additional layers in multilayer devices. On the other hand, we have used photo-cross-linkable oxetanyl moieties which neither form additions after polymerization nor decrease the concentration of charge transporting chromophores. The advantages as well as cross-linking mechanism of oxetanyl functionalized derivatives containing electro-active fragments is well demonstrated in the literature [5–7,10].

2. Experimental

2.1. Instrumentation

¹H NMR spectra were recorded using Varian Unity Inova (300 MHz) apparatus. Mass spectra were obtained on a Waters ZQ 2000 spectrometer. FTIR spectra were recorded using Perkin Elmer FT-IR System. UV spectra were measured with a Spectronic Genesys[™] 8 spectrometer. Fluorescence (FL) spectra were recorded with a MPF-4 spectrometer. Differential scanning calorimetry (DSC) measurements were carried out using a Bruker Reflex II thermosystem. Thermogravimetric analysis (TGA) was performed on a Netzsch STA 409. The TGA and DSC curves were recorded in a nitrogen atmosphere at a heating rate of 10 °C/min.

The ionization potentials of the layers of the compounds synthesized were measured by the electron photoemission method in air, which was described earlier [11]. The measurement method was, in principle, similar to that described by Miyamoto et al. [12].The samples for the ionization potential measurement were prepared as we described earlier [13].



^{*} Corresponding author. Tel./fax: +370 37 456 525. E-mail address: saugrig@ktu.lt (S. Grigalevicius).

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Thin films of the materials were prepared by spin-coating from their chloroform solutions. Solutions for preparation of crosslinked films contained 7 wt% of $\{4-[(2-hydroxytetradecyl)-oxyl]$ phenyl}-phenyliodonium hexafluorantimonate as initiator for the cationic polymerization of oxetane units of **3** or **4**. The cross-linkable films were irradiated (standard handheld UV lamp) for 2 min at room temperature. Afterwards, to further advance the cross-linking process in the growing network, the films were annealed at 180 °C for 7 min. The resulting polymer networks were found to be insoluble in organic solvents.

The multilayer electroluminescent devices were fabricated on glass substrates and had the typical structure with the organic layers sandwiched between a bottom ITO anode and a top metal cathode. Before use in device fabrication, the ITO-coated glass substrates were carefully cleaned and treated with UV/ozone right before deposition of the organic layers. PEDOT layers were deposited by spincoating and heated at 120 °C for 30 min. The hole transporting layers (HTL) were made by spin-coating a 25-30 nm layer of the derivatives 3 or 4. Cross-linkable HTL contained the mentioned initiator and were converted to insoluble networks as described above. Tris(quinolin-8-olato)aluminum (Alq₃) or poly(9,9-dioctylfluorene-2,7-diylco-2,5-di(phenyl-4-yl)-2,1,3-benzothiadiazole) (PFBT)were used as green light emitters. The polymer was spin-coated from its toluene solution onto the cross-linked hole transporting network. Evaporation of Alq₃ as well as of Al cathode was done at a pressure of 3×10^{-6} mbar in vacuum evaporation equipment.

The current–voltage and luminance-voltage characteristics were recorded under forward bias using a computer controlled Keithley 2400 source meter and a PR650 Spectrometer [14]. All measurements were performed at ambient conditions in air.

2.2. Materials

2-Bromofluorene (1), benzyl triethyl ammonium chloride (BEAC), NaOH, aniline, N,N-diphenylbenzidine, sodium *tert*-butoxide (*t*-BuONa), tri(*tert*-butyl)phosphine {(*t*-Bu)₃P)} solution (1.0 M in toluene), tris(dibenzylideneacetone)dipalladium(0) {Pd₂(dba)₃} and Alq₃ were received from Sigma–Aldrich.

2.2.1. 3-Bromomethyl-3-methyloxetane was obtained from Chemada Fine Chemicals

Poly(9,9-dioctylfluorene-2,7-diyl-co-2,5-di(phenyl-4_-yl)-2,1,3benzothiadiazole) (**PFBT**) was prepared by the procedure as described earlier [15]. The polymer was used as an green light emitter in OLEDs.

2.2.2. 2-Bromo-9,9-di(3-methyloxetan-3-ylmethyl)fluorene (2)

A 5 g (20 mmol) of 2-bromofluorene (**1**) and 0.2 g (0.92 mmol) of BEAC were dissolved in 20 ml of DMSO and 20 ml of aqueous sodium hydroxide solution (50%) were added. After 5 min 8.42 g (50 mmol) of 3-bromomethyl-3-methyloxetane were added drop wise into the reaction mixture. The two-phase system was stirred at 80 °C for 6 h under argon atmosphere. After TLC control the reaction mixture was treated with 2 N HCl and extracted with chloroform. After extraction the organic layer was dried over MgSO₄. After filtration and evaporation of the solvent, the crude product was purified by silica gel column chromatography using ethyl acetate and hexane (vol. ratio 1:3) as eluent. Yield: 5.6 g (68%) of orange crystals. M.p.: 128–129 °C.

¹H NMR spectrum (300 MHz, CDCl₃, δ, ppm): 7.62–7.22 (m, 7H, Ar), 4.47 (d, 2H, CH₂ of oxetane ring, J = 5.7 Hz), 4.32 (d, 2H, CH₂ of oxetane ring, J = 5.7 Hz), 3.59 (d, 2H, CH₂ of oxetane ring, J = 5.6 Hz), 3.50 (d, 2H, CH₂ of oxetane ring, J = 5.6 Hz), 2.54 (d, 2H, CCH₂C, J = 14 Hz), 2.45 (d, 2H, CCH₂C, J = 14 Hz), 0.40 (s, 6H, 2 × CH₃). MS (APCl⁺, 20 V), m/z (%): 414.4 ([M + H]⁺, 100). IR (KBr,

v, cm⁻¹): 3048 (aromatic C–H), 2963, 2858 (aliphatic C–H), 976 (C–O–C in oxetane ring), 741; 694 (CH=CH).

2.2.3. N,N-Di[9,9-di(3-methyloxetan-3-ylmethyl)fluoren-2-yl]-N-phenylamine (3).

A 23.5 mg (0.025 mmol) of $Pd_2(dba)_3$, 10.5 mg, (0.05 mmol) of *t*-Bu₃P and toluene (7 ml) were charged under nitrogen in a two neck flask equipped with a reflux condenser. The mixture was stirred at room temperature for 10 min. Then 1.06 g (2.6 mmol) of 2-bromo-9,9-di(3-methyloxetan-3-ylmethyl)fluorene (**2**), 0.1 g (1.1 mmol) of aniline and 1.47 g (15.6 mmol) of *t*-BuONa were added into the reaction mixture, and it was stirred under nitrogen for 24 h at 80 °C. The mixture was cooled and quenched by an addition of ice water. The product was extracted with ethyl acetate. The combined extract was dried over anhydrous MgSO₄. The crude product was purified by silica gel column chromatography using ethyl acetate and hexane mixture (vol. ratio 1:3) as an eluent. Yield: 0.23 g (31%) of yellowish crystals. M.p.: 253–254 °C.

¹H NMR spectrum (300 MHz, CDCl₃, δ, ppm): 7.66–7.05 (m, 19H, Ar), 4.28 (d, 4H, CH₂ of oxetane ring, J = 6 Hz), 4.23 (d, 4H, CH₂ of oxetane ring, J = 6 Hz), 3.7 (d, 4H, CH₂ of oxetane ring, J = 6 Hz), 3.55 (d, 2H, CH₂ of oxetane ring, J = 6 Hz), 2.48 (d, 4H, 2 × CCH₂C, J = 14 Hz), 2.33 (d, 4H, 2 × CCH₂C, J = 14 Hz), 0.57 (s, 12H, 4 × CH₃). ¹³C NMR spectrum (75.4 MHz, CDCl₃, δ, ppm): 24.41, 39.97, 49.75, 53.01, 77.31, 83.92, 84.57, 120.04, 121.40, 123.31, 124.22, 124.53, 125.11, 126.22, 128.09, 129.62, 134.99, 136.45, 147.99, 149.97.

MS (APCI+, 20 V), m/z (%): 759.2 ([M + H]⁺, 100). IR (KBr, , cm⁻¹): 3035 (aromatic C–H), 2959; 2864 (aliphatic C–H), 1487; 1450 (C–N), 976 (C–O–C in oxetane ring), 741; 694 (CH=CH of mono substituted benzene). Elemental analysis for C₅₂H₅₅N₄O₄,% Calc.: C 82.40, H 7.31, N 1.85, O 8.44;% Found: C 82.31, H 7.43, N 1.85.

2.2.4. N,N'-Di[9,9-di(3-methyloxetan-3-ylmethyl)fluoren-9-yl]-N,N'diphenylbenzidine (**4**)

A 10 mg (0.01 mmol) of $Pd_2(dba)_3$, 4.4 mg (0.02 mmol) of t-Bu₃P and toluene (4 ml) were charged under nitrogen in a two neck flask equipped with a reflux condenser. The mixture was stirred at room temperature for 10 min. Then 0.49 g (1.1 mmol) of 2-bromo-9,9-di(3-methyloxetan-3-ylmethyl)fluorene (**2**), 0.1 g (0.3 mmol) of N,N'-diphenylbenzidine and 0.69 g (6.6 mmol) of *t*-BuONa were added into the reaction mixture, and it was stirred under nitrogen for 18 h at 80 °C. The mixture was cooled and quenched by an addition of ice water. The product was extracted into ethyl acetate. The combined extract was dried over anhydrous MgSO₄. The crude product was purified by silica gel column chromatography using ethyl acetate and hexane (vol. ratio 1:3) as an eluent. Yield: 0.25 g (21%) of yellow amorphous powder.

¹H NMR spectrum (300 MHz, CDCl₃, δ, ppm): 7.73–7.05 (m, 32H, Ar), 4.3–4.22 (m, 8H, CH₂ of oxetane ring), 3.71 (d, 2H, CH₂ of oxetane ring, *J* = 5.5 Hz), 3.56 (d, 6H, CH₂ of oxetane ring, *J* = 5.6 Hz), 2.55–2.36 (m, 8H, $4 \times$ CCH₂C), 0.56 (s, 12H, $4 \times$ CH₃). ¹³C NMR spectrum (75.4 MHz, CDCl₃, δ, ppm): 24.41, 39.97, 49.75, 53.01, 77.31, 83.92, 84.57, 120.04, 121.40, 123.31, 124.22, 124.53, 125.11, 126.22, 127.62, 128.09, 129.62, 134.99, 136,45, 140.95, 147.12, 147.99, 148.01, 149.97.

MS (APCI+, 20 V), m/z (%): 1002.5 ($[M + H]^+$, 100). IR (KBr, cm⁻¹): 3035 (aromatic C—H), 2956; 2864 (aliphatic C—H), 1489; 1451 (C—N), 969 (C—O—C in oxetane ring), 744; 696 (CH=CH of mono substituted benzene). Elemental analysis for C₇₀H₆₈N₂O₄% Calc.: C 83.97, H 6.85, N 2.80, O 6.39;% Found: C 83.91, H 6.80, N 2.84.

3. Results and discussion

The synthetic route of cross-linkable aromatic amines (**3–4**) containing fluorenyl fragments is shown in Scheme 1. 2-Bromo-

9,9-di(3-methyloxetan-3-ylmethyl)fluorene (**2**) as a key material was synthesized from commercially available 2-bromofluorene and 3-bromomethyl-3-methyloxetane in a two phase system by similar procedure as it is described in the literature [16,17]. An excess of the bromo compound **2** was reacted with aniline or N,N'-diphenylbenzidine by the method of Buchwald-Hartwig [18] to afford N,N'-di[9,9-di(3-methyloxetan-3-ylmethyl)fluorenyl]-N-phenylamine (**3**) or N,N'-di[9,9-di(3-methyloxetan-3-ylmethyl)fluoren-9-yl]-N,N'-diphenylbenzidine (**4**), respectively.

The newly synthesized compounds **3** and **4** were identified by mass spectrometry, IR and by ¹H NMR spectroscopy. The data were found to be in good agreement with the proposed structures. The materials were soluble in common organic solvents, such as chloroform and THF at room temperature. Transparent thin films of these materials could be prepared by spin coating from their solutions.

The behavior under heating of the compounds **3** and **4** was studied by DSC and TGA under a nitrogen atmosphere. It was established that both the materials demonstrate high thermal stability. The temperatures at which 5% loss of mass was observed are 384 °C for **3** and 400 °C for **4** as confirmed by TGA with a heating rate of 10 °C/min.

The compound **4** was obtained in nature as amorphous material. When the sample of **4** was heated T_g was observed at 115 °C and no peaks due to crystallization and melting appeared. Cooling down and repeated heating revealed only the glass transition again. The derivative **3** was obtained as crystalline material by re-crystallization from solution, however it readily formed glass when the melt sample was cooled on standing in air or with liquid nitrogen. The DSC thermograms of **3** are shown in Fig. 1. When the crystalline sample was heated, the endothermic peak due to melting was observed at 253 °C. When the melt sample was cooled down and heated again, the glass-transition phenomenon was observed at 122 °C and on further heating no peaks due to crystallization and melting appeared.

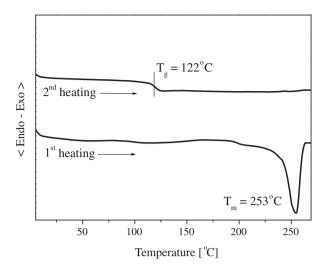
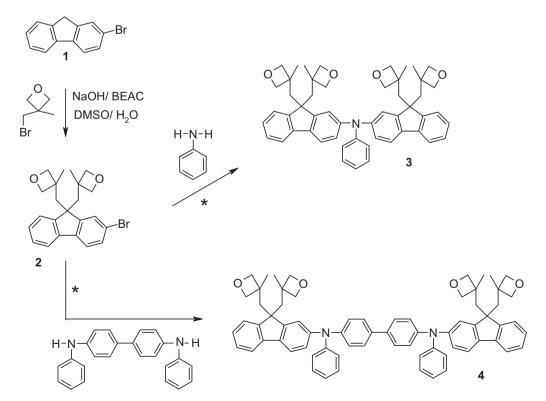


Fig. 1. DSC curves of the material 3. Heating rate 10 °C/min.

UV absorption and FL spectra of dilute solutions of the compounds synthesized were recorded. The pertinent data are presented in Fig. 2. The electronic absorption energy of the derivatives is similar, and the λ_{max} values are in the range of 220–380 m. Their FL maxima were registered at 398 nm for **3** and at 409 nm for **4**. Comparison of the FL spectra of these derivatives demonstrates that benzidine fragment has an evident effect on electronic structure of compound **4**. It could be expected that ionization potentials (Ip) of the materials **3** and **4** will be different. On the other hand comparison of the UV absorption and FL spectra of material **4** with those of similar triphenyldiamine (TPD)-based derivatives [19] revealed that conjugation along the backbones of the compound **4** is similar to that of TPD, and layers of **4** should demonstrate Ip similar as of TPD.



Scheme 1. *Pd₂(dba)₃, t-Bu₃P, and t-BuONa in toluene.

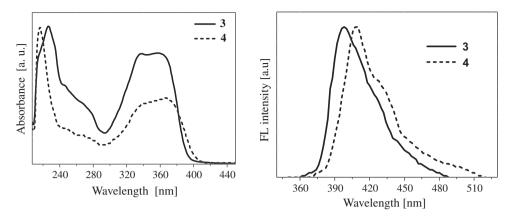


Fig. 2. UV absorption and FL spectra of dilute THF solutions of 3 and 4.

The electron photoemission spectra of layers of the compounds synthesized and values of their Ip are presented in Fig. 3. I_p of the layer of material **4** was lower than that of **3**. This observation indicates that conjugation in molecules of derivative **4** is slightly larger than that of **3**. As it was expected from UV absorption and FL spectra, Ip of layer of **4** was rather close to that of TPD derivatives [19]. It is evident that the benzidine fragment has considerable influence on Ip of the materials.

The electron affinity of the materials was determined from their optical band gaps (E_g) and ionization potentials ($E_g = Ip - E_{LUMO}$). The energy band gaps of **3** and **4**, which were taken as the absorption onsets of the UV–vis spectra of the materials, were estimated to be 3.15 and 3.05 eV, respectively. These values correspond to LUMO of -2.35 eV for **3** and -2.5 eV for derivative **4**.

The layers of compound **4** should demonstrate better hole injection and transport properties. This was studied by application of the synthesized materials as HTL in OLEDs. The devices were of the form: ITO/PEDOT/**3** or **4**/Alq₃/LiF/Al with Alq₃ as an electroluminescent/electron transporting layer. When a positive voltage was applied to the OLEDs the bright green electroluminescence of Alq₃ was observed with an emission maximum at around 520 nm. This implies that hole drift mobility in layers of the compounds **3** and **4** is sufficient for an effective charge carrier transport to Alq₃ layer.

Fig. 4 shows efficiency and luminance – voltage characteristics of the OLEDs containing HTL of the compounds **3** or **4**. The characteristics of a device without HTL are presented for comparison. The devices containing HTL exhibit turn-on voltages of 2.5–4 V,

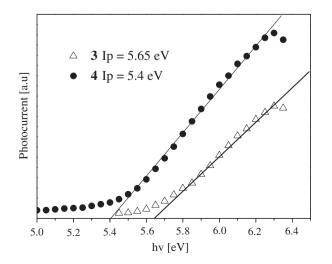


Fig. 3. Electron photoemission spectra of layers prepared using 3 and 4.

photometric efficiencies of 2–4 cd/A, and maximum brightness of 4450–5980 cd/m². As it was expected from Ip values of the layers, the device using derivative **4** as the HT material exhibited better overall performance due to more effective hole injection and transport. Efficiency of the device with **4** showed only a moderate drop in the observed current density window up to 50 mA/cm². For the technically important brightness of 100 cd/m², an efficiency above 3.5 cd/A was detected. It is also evident that HTL of the synthesized

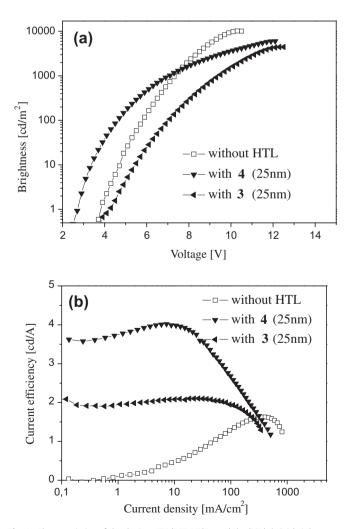


Fig. 4. Characteristics of the devices ITO/PEDOT/**3** or **4**/Alq₃/LiF/Al; (a) brightness – voltage, (b) current efficiency – current density.

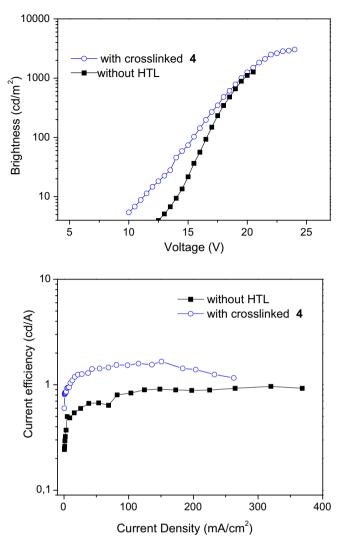


Fig. 5. LED characteristics of the devices ITO/HTL of cross-linked 4/PFBT/(Ca/AI) and ITO/PFBT/(Ca/AI).

materials improve considerably efficiencies of the devices as compared with that of device without HTL.

Cross-linked layers of the material **4**, which demonstrated better characteristics in Alq₃ based devices, were tested as hole transporting networks in two layer LEDs of the configuration ITO/cross-linked material **4**/green light emitting polymer **PFBT**/(Ca/Al). The hole transporting layers were made by spin-coating from solution a 30–35 nm layer of the derivative **4** containing initiator onto the substrates with ITO and by following photoinitiated cross-linking as described in experimental part. A similar device without HTL was prepared for comparison.

When a positive voltage was applied to the device bright green electroluminescence of PFBT was observed with an emission maximum at around 528 nm. This implies that the hole mobility in the hole transporting network of 4 was sufficient for charge carrier recombination occurring within the PFBT layer. Fig. 5 shows luminance- voltage characteristics as well as efficiencies of the OLEDs containing hole transporting networks of 4. The characteristics of a devise without the HTL are also presented in the figure. Turn-on voltage of the single layer device was ca 12 V, maximum brightness was 1270 cd/m² and the maximum EL efficiency of the device reached 0.96 cd/A. For the device having HT network

of 4, the turn-on voltage decreased to 10 V, showing the effect of the improved hole injection and transport. EL efficiency of the device reached 1.65 cd/A and the maximum brightness exceeded 3000 cd/m^2 .

It should be pointed out that these characteristics were obtained in non-optimized test devices under ordinary laboratory conditions. The device performance may be further improved by an optimization of the layer thicknesses and processing conditions.

In conclusion, cross-linkable monomers with fluorenyl-substituted arylamine core as hole transport agent, and oxetanyl groups as photocros-linking moieties have been synthesized and characterized. The materials are thermally stable and form amorphous films with glass transition temperatures in the range of 115– 122 °C. These derivatives have been studied as hole transporting materials in bilayer OLEDs with Alq₃ as an emitter. The devices with derivative containing benzidine core exhibited the best overall performance (turn-on voltage: ~ 2.5 V; maximum luminance efficiency: 4 cd/A; maximum brightness: approx. 6000 cd/m²). Cross-linked networks of the derivative have also been tested as hole transporting structures in multilayer OLEDs with green light emitting polymer. The device demonstrated maximum photometric efficiency of 1.65 cd/A and maximum brightness of about 3000 cd/m².

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