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Multifunctional emissive material based on 1-phenyl-1,2,3,4-tetrahydroquinoline

T. Malinauskas [a], J. Stumbraite [a], V. Getautis [a,*], V. Gaidelis [b], V. Jankauskas [b], G. Juška [b], K. Arlauskas [b], K. Kazlauskas [c]

[a] *Faculty of Chemical Technology, Kaunas University of Technology, Radvilėnu pl. 19, LT-50270 Kaunas, Lithuania*
[b] *Department of Solid State Electronics, Vilnius University, Saulėtekio 9, LT-10222 Vilnius, Lithuania*
[c] *Institute of Materials Science and Applied Research, Vilnius University, Saulėtekio 9-III, LT-10222 Vilnius, Lithuania*

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ABSTRACT

A novel compound based on 3-hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline and electron affinitive cyano moieties incorporated into the bis(styryl) molecular structure was investigated as a potential multifunctional emissive material. The ionization potential and the LUMO level of the compound were estimated to be 5.66 eV and 3.2 eV, respectively. The hole drift mobility (5×10^{-5} cm^2/V s) of a vacuum-deposited thin film evaluated using time-of-flight was independent of the electric field. Moreover, the fluorescence and electroluminescence spectra of vacuum-deposited film demonstrated good coincidence and both displayed almost pure yellow-green emission color.

1. Introduction

The tremendous research effort following the discovery of efficient electroluminescence in organic and conjugated polymer thin films [1,2] has resulted in a vast number of publications on organic light-emitting diodes (OLEDs) [1–3]. Different aspects of OLEDs, including a variety of electroluminescent materials [4], device physics and engineering [5], have been reviewed. Applications of OLED-based flat-panel displays in commercially available digital cameras and cell phones with incredible market penetration demonstrate a considerable progress in the field. However, major challenges of improving the performance and durability of OLEDs for displays and lighting applications still remain. One of the key issues for developing the next generation high-performance OLEDs is the design and synthesis of readily processable and thermally robust emissive and charge transport materials with improved multifunctional properties.

OLED device typically contains three or more layers of amorphous organic/organometallic materials with each layer designed to perform only a certain function, e.g. to transport either holes or electrons, or emit light. The simultaneous exploitation of several materials to perform the required functions of light emission, charge injection and transport in an OLED device has resulted in the improvement of device performance, however, at the cost of much more complex OLED architecture [1–5]. Thus, materials featuring multifunctional properties of light emission and ambipolar charge transport are of great interest for the development of highly efficient OLEDs. Such multifunctional materials allow simplifying the device structure and fabrication [6].

Recently, it was demonstrated that compounds featuring bis(styryl) molecular structure and triphenylamine or carbazole chromophores are highly fluorescent [7–9]. Vinylene linkage was found to serve not only as a “conjugated bridge”, but also it improved electroluminescent (EL) characteristics by reducing steric interactions between aromatic rings, and thus, decreasing the free rotation between units [10]. As π-conjugated emissive compounds often exhibit better holes injection and transport than electrons, incorporation of electron affinitive moieties (A) such as cyano, oxadiazole, triazole, or triazine functionalities into the emissive π-conjugated core, possessing electron-donating groups (D), is a reasonable approach to develop multifunctional emissive materials [11].

A common problem with D-π-A-π-D structure materials is their poor solubility and tendency to crystallize, therefore such compounds are deposited as thin films by sublimation. Crystallinity also affects the homogeneity of the layered material and results in

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* Corresponding author. Tel.: +370 37 300196; fax: +370 37 300152.
E-mail address: vgetaut@ktu.lt (V. Getautis).

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grain boundaries that are detrimental to OLED performance. These problems can be addressed by utilizing polymeric multifunctional emissive materials. Unfortunately, polymeric materials often suffer from purity related problems. A possible solution: high purity and well-defined structure, low molecular weight compounds. These features combined with the excellent solubility of low molecular weight compounds are highly attractive for the synthesis of multifunctional emissive materials.

Recently, we have reported the synthesis and hole transporting properties of the derivatives based on 3-hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline [12,13]. Excellent solubility of these derivatives makes them readily processable into large-area thin films by spin-coating from dilute solutions as well as deposited as thin films by sublimation. Additionally, 3-hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline compounds possess flexible aliphatic chain and reactive hydroxyl group, which can be exploited in further modification of the molecules in order to further enhance their solubility and decrease tendency of crystallization.

All these findings prompted us to synthesize a novel multifunctional emissive compound, which was obtained by incorporation of 1-phenyl-1,2,3,4-tetrahydroquinoline moiety into the bis(styryl) molecular structure.

2. Experimental details

2.1. Measurements

The ^{1}H NMR spectra were measured on Varian Unity Inova (300 MHz) spectrometer in $CDCl_3$. The IR spectra of the samples in KBr pellets were recorded on a Perkin Elmer Spectrum BX II FT-IR System spectrometer. The UV spectra of the sample THF solutions (10^{-4} M) placed in a 1 mm path length microcell were recorded on a Spectronic Genesys 8 spectrometer. Fluorescense (FL) emission and excitation spectra of THF solutions (10^{-4} M) were measured using Perkin Elmer LS-5 luminescence spectrometer. Electroluminescence (EL) spectra were measured using fiber-optic photonic multi-channel spectral analyzer (Hamamatsu PMA-11). The course of the reactions was monitored by TLC on Alugram® Sil G/UV_{254} plates and developed with I_2 or UV light. Silica gel (grade 62, 60–200 mesh, 150 Å, Aldrich) was used for column chromatography. Elemental analyses were performed with an Exeter Analytical CE-44 Elemental Analyzer; their results agreed satisfactorily with the calculated values. Melting points were determined in capillary tubes on capillary melting point apparatus Electrothermal MEL-TEMP®. The ionization potential (I_p) was measured by the electron photoemission in air method, similar to that described in Refs. [14,15]. The samples for mobility measurements were prepared from THF solutions using 1:1 mass proportion compositions of transporting material with Polycarbonate-Z (PC-Z) (Iupilon Z-200 from Mitsubishi Gas Chemical Co.). The sample substrate was polyester film with conductive Al layer. The charge carrier mobilities were measured by the xerographic time-of-flight technique [16,19]. Thermal properties for compound **4** were examined using a Netzsch STA 409 PC Luxx apparatus at a heating rate of 10 K/min under nitrogen atmosphere. The glass transition temperatures (T_g) were determined from the second heating. Compound **1** was synthesized according to Ref. [12].

2.2. Fabrication of EL devices

Electroluminescent (EL) devices with the key-component compound **4** have been fabricated using two techniques, namely, vacuum-deposition and spin-coating.

In the first approach, the thin film of compound **4** was vacuum-deposited (at pressure 2.66 mPa) onto indium-tin-oxide (ITO) coated glass substrate at substrate temperature of 293 K. The fabrication of EL device was completed by evaporating of semi-transparent Al electrode on top of the structure. The thickness of the film (81 nm) was estimated using charge extraction by linearly increasing voltage (CELIV) method [17,18].

Compound **4** was also used as an emitting and electron transporting material in another EL device obtained by spin-coating technique. This EL device consisted of the following structure: ITO/PEDOT:PSS/[DEDPB:DM, 3:2]/[PVK:**4**, 6:4]/Ca/Al. Here, glass substrate with conductive ITO layer was additionally coated with a layer of poly(3,4-ethylenedioxythiophene): poly(styrenesulfonate) (PEDOT:PSS) and a cross-linked layer consisting of *N*,*N*-dimethyldipropylenetriamine (DM) and *N*,*N*′-di(2,3-epoxypropyl)- *N*,*N*′-diphenylbenzidine (DEDPB). Both layers, PEDOT:PSS and DEDPB:DM, were used for hole-injection. Composition of poly(9-vinylcarbazole) (PVK) (Luvican M170 from BASF SE) and compound **4** was used as the emitting and electron transporting layer. Finally, the fabrication of the device was completed by depositing Ca/Al cathode using vacuum thermal evaporation.

2.3. Materials

2.3.1. 1-Phenyl-3-propoxy-1,2,3,4-tetrahydroquinoline (**2**, $C_{18}H_{21}NO$)

Compound **1** (50 g, 0.22 mol), 14 g (0.11 mol) Na_2SO_4, 44 g (0.67 mol) KOH and 150 ml (1.7 mol) 1-bromopropane were added

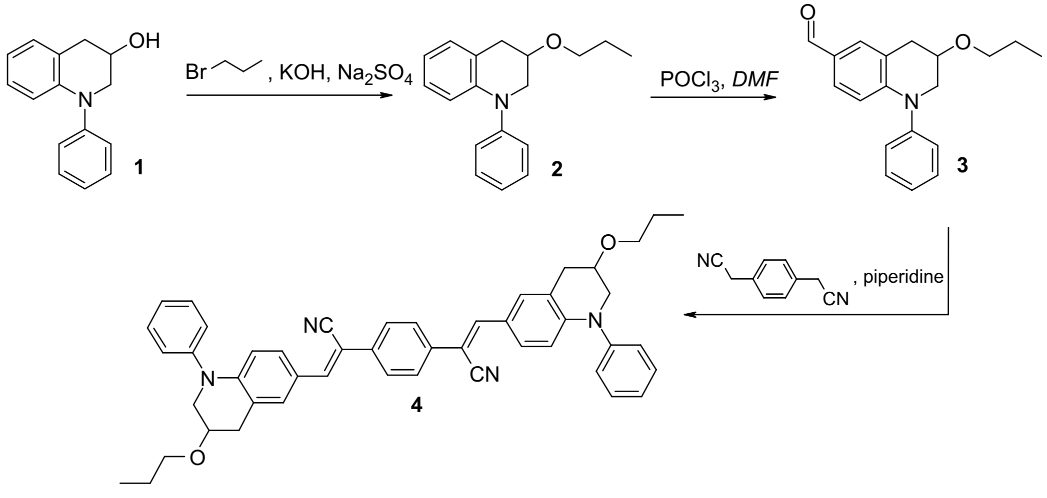

Fig. 1. Synthesis route to 1-phenyl-1,2,3,4-tetrahydroquinoline based compound **4**.

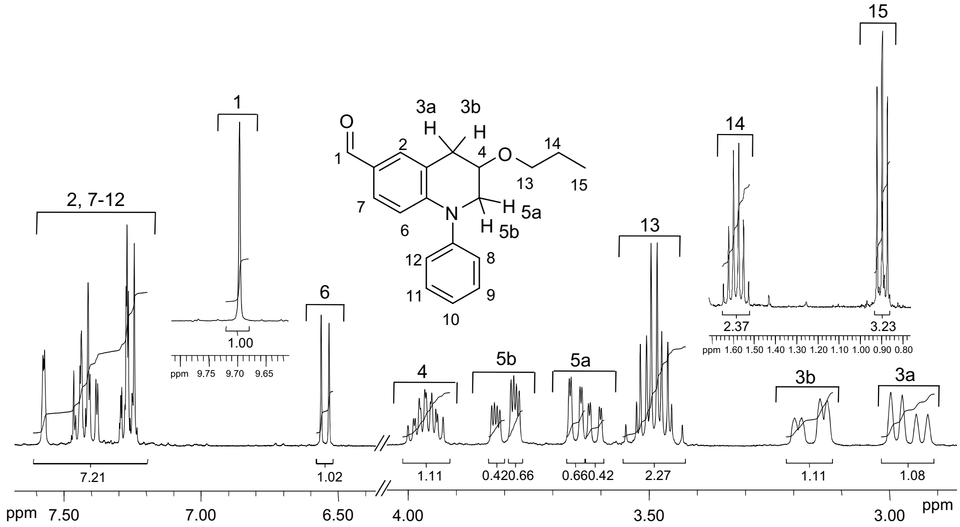

Fig. 2. ^{1}H NMR spectra of **3** (300 MHz, $CDCl_3$).

to the reaction mixture and refluxed. Further 14.6 g (0.22 mol) KOH and 50 ml (0.6 mol) 1-bromopropane were added after 1 and 2 h. Afterward the mixture was refluxed for another 22 h (TLC control, eluent: acetone:*n*-hexane = 1:3). The reaction mixture was cooled to the room temperature and extracted with ethyl acetate and washed with H_2O until wash water was neutral. The organic layer was dried ($MgSO_4$), filtered off and ethyl acetate was removed. The residue was purified by column chromatography (eluent: acetone:*n*-hexane = 1:49). Yield of **2** was 83% (49 g); IR (KBr; $\bar{\nu}$, cm^{-1}): 3062, 3035 (CH_{arom}), 2961, 2933, 2874 (CH_{aliph}), 1605, 1593, 1576, 1461 (C=C, C–N), 1101 (C–O–C), 749, 728, 699 (CH=CH of mono- and disubstituted benzenes); ^{1}H NMR (300 MHz, $CDCl_3$; δ, ppm): 7.37–7.29 (m, 2H, Ar), 7.25–7.18 (m, 2H, Ar), 7.11–7.03 (m, 2H, Ar), 6.93 (m, 1H, Ar), 6.80–6.69 (m, 2H, Ar), 3.95–3.84 (m, 1H, CH), 3.81 (dd, $J_{AB} = 3.9$ Hz, $J_{Ax} = 1.75$ Hz, 0.4H, H_A of one conformer of NCH_2), 3.76 (dd, $J_{A'B} = 3.9$ Hz, $J_{Ax} = 1.75$ Hz, 0.6H, $H_{\acute{A}}$ of other conformer of NCH_2), 3.54–3.39 (m, 3H, other proton of NCH_2, OCH_2), 3.16 (dd, $J_{AB} = 15.9$ Hz, $J_{Ax} = 4.7$ Hz, 1H, H_A of $C\underline{H_2}CH$), 2.86 (dd, $J_{AB} = 15.9$ Hz, $J_{Bx} = 8.3$ Hz, 1H, H_B of $C\underline{H_2}CH$), 1.66–1.49 (m, 2H, $C\underline{H_2}CH_3$), 0.89 (t, $J = 7.4$ Hz, 3H, CH_3); APCI-MS: *m*/*z* (%) = 268 $[M+H]^+$ (100); Anal. calcd for $C_{18}H_{21}NO$: C, 80.86; H, 7.92; N, 5.24. Found, %: C, 81.02; H, 7.84; N, 5.37.

2.3.2. 1-Phenyl-3-propoxy-1,2,3,4-tetrahydroquinoline-6-carbaldehyde (3, $C_{19}H_{21}NO_2$)

DMF (57 ml, 0.73 mol) was cooled in a salt/ice bath, and 43 ml (0.46 mol) of $POCl_3$ was slowly added to the mixture. During the addition of $POCl_3$, the temperature of mixture was not allowed to rise above 5 °C. After addition of $POCl_3$ the reaction mixture was allowed to warm to room temperature. Compound **2** (49 g, 0.1 mol) was dissolved in 90 ml of DMF and added to the mixture. Afterwards it was heated to 90 °C for 1 h (TLC control, eluent: ethyl acetate:*n*-hexane = 1:3). The hot reaction mixture was poured into a 1000 ml beaker containing 400 g of ice. The mixture was neutralized by adding 58 g (1.45 mol) of sodium hydroxyde dissolved in water, and allowed to stand at 5 °C for 24 h. The precipitate was decanted, dissolved in ethyl acetate and washed with water until the wash water was neutral. The organic layer was dried

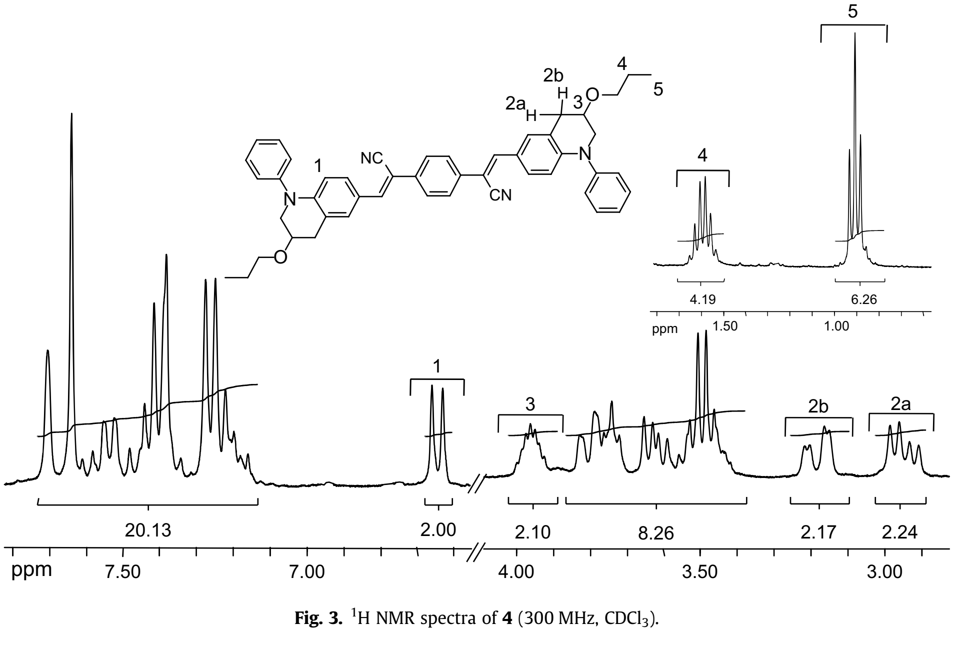

Fig. 3. ^{1}H NMR spectra of **4** (300 MHz, $CDCl_3$).

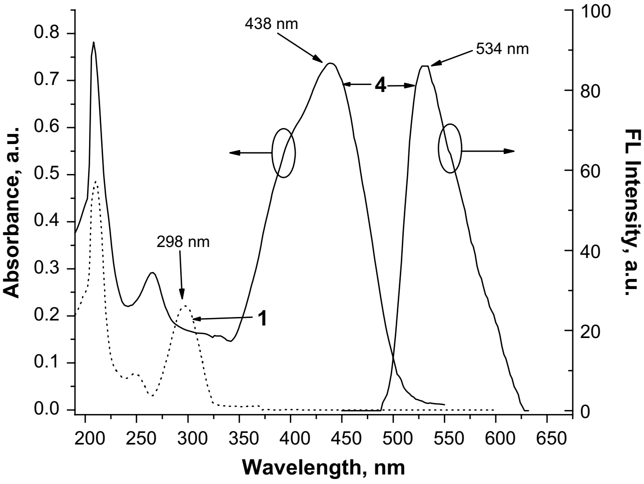

Fig. 4. Absorption and fluorescence spectra of **4** (THF, $c = 10^{-4}$ M, $\lambda_{ex} = 450$ nm).

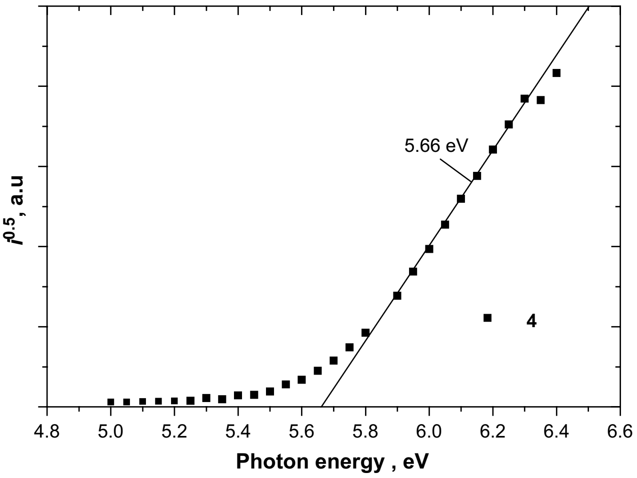

Fig. 6. Electron photoemission (in air) spectrum of **4**.

(MgSO$_4$), filtered off and ethyl acetate was removed. The residue was purified by column chromatography (eluent: ethyl acetate:*n*-hexane = 1:4) to give aldehyde **3**. Yield 60% (32 g); IR (KBr; $\bar{\nu}$, cm^{-1}): 3062, 3038 (CH$_{arom}$), 2962, 2935, 2875 (CH$_{aliph}$), 1680 (C=O), 1607, 1589, 1561, 1507, 1493 (C=C, C–N), 1103 (C–O–C), 946, 914, 870, 769, 730, 700 (CH=CH of mono- and trisubstituted benzenes); ^{1}H NMR (300 MHz, CDCl$_3$; δ, ppm): 9.70 (s, 1H, CHO), 7.59–7.23 (m, 7H, Ar), 6.55 (d, $J = 8.6$ Hz, 1H, 8-H of Ht), 4.01–3.92 (m, 1H, CH), 3.82 (dd, $J_{AB} = 3.4$ Hz, $J_{Ax} = 1.7$ Hz, 0.4H, H$_A$ of one conformer of NCH$_2$), 3.78 (dd, $J_{A'B} = 3.4$ Hz, $J_{Ax} = 1.7$ Hz, 0.6H, H$_{A'}$ of other conformer of NCH$_2$), 3.65 (dd, $J_{AB} = 6.9$ Hz, $J_{Bx} = 1.2$ Hz, 0.6H, H$_B$ of one conformer of NCH$_2$), 3.61 (dd, $J_{A'B} = 6.9$ Hz, $J_{A'x} = 1.2$ Hz, 0.4H, H$_{B'}$ of other conformer of NCH$_2$), 3.55–3.43 (m, 2H, OCH$_2$), 3.16 (dd, $J_{AB} = 16.1$ Hz, $J_{Ax} = 4.5$ Hz, 1H, H$_A$ of $C\underline{H_2}CH$), 2.96 (dd, $J_{AB} = 16.1$ Hz, $J_{Bx} = 7.2$ Hz, 1H, H$_B$ of $C\underline{H_2}CH$), 1.66–1.52 (m, 2H, $C\underline{H_2}CH_3$), 0.90 (t, $J = 7.4$ Hz, 3H, CH$_3$); APCI-MS: *m*/*z* (%) = 296 $[M + H]^+$ (100); Anal. calcd for $C_{19}H_{21}NO_2$: C, 77.26; H, 7.17; N, 4.74. Found, %: C, 77.42; H, 7.23; N, 4.65.

*2.3.3. 1,4-Bis-{1-nitrile-2-[(1-phenyl-3-propoxy)-1,2,3,4-tetrahydroquinolin-6-yl]ethenyl}-benzene (**4**, $C_{48}H_{46}N_4O_2$)*

Compound **3** (3 g, 0.01 mol), 0.6 g (0.004 mol) of 1,4-phenylenediacetonitrile were dissolved in 10 ml (0.1 mol) of piperidine and refluxed for 67 h (TLC control, eluent: ethyl acetate:*n*-hexane = 1:3). Piperidine was removed and the residue was purified by column chromatography (eluent: ethyl acetate:*n*-hexane = 1:7). The obtained product was crystallized from ethyl acetate:2-propanol = 2:1. Yield of **4** was 42% (1.5 g); mp 146–148 °C. IR (KBr; $\bar{\nu}$, cm^{-1}): 3056, 3033 (CH$_{ar}$), 2959, 2933, 2874, 2853 (CH$_{aliph}$), 2201 (C≡N), 1611, 1577, 1513, 1492 (C=C, C–N), 1103 (C–O–C), 934, 918, 839, 831, 760, 733, 701 (CH=CH of mono-, 1,4-di- and 1,2,4-trisubstituted benzenes); ^{1}H NMR (300 MHz, CDCl$_3$; δ, ppm): 7.73–7.16 (m, 20H, Ar, Ht, CH=C), 6.64 (d, $J = 8.8$ Hz, 2H, 8-H of Ht), 4.02–3.91 (m, 2H, CH), 3.85–3.41 (m, 8H, NCH$_2$, OCH$_2$), 3.19 (dd, $J_{AB} = 16.1$ Hz, $J_{Ax} = 4.3$ Hz, 2H, H$_A$ of $C\underline{H_2}CH$), 2.95 (dd, $J_{AB} = 16.1$ Hz, $J_{Bx} = 7.0$ Hz, 2H, H$_B$ of $C\underline{H_2}CH$), 1.66–1.50 (m, 4H, $C\underline{H_2}CH_3$), 0.91 (t, $J = 7.4$ Hz, 6H, CH$_3$); APCI-MS: *m*/*z* (%) = 711 $[M + H]^+$ (100). Anal. calcd for $C_{48}H_{46}N_4O_2$: C, 81.10; H, 6.52; N, 7.88. Found, %: C, 79.92; H, 6.67; N, 7.73.

3. Results and discussion

The novel multifunctional material with D-π-A-π-D structure, possessing 1-phenyl-1,2,3,4-tetrahydroquinoline moieties, was synthesized according to the synthesis route shown in Fig. 1.

Alkylation of 3-hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline (**1**) with 1-bromopropane in the presence of KOH and Na$_2$SO$_4$ yielded phenyltetrahydroquinoline **2** with substituted hydroxyl group.

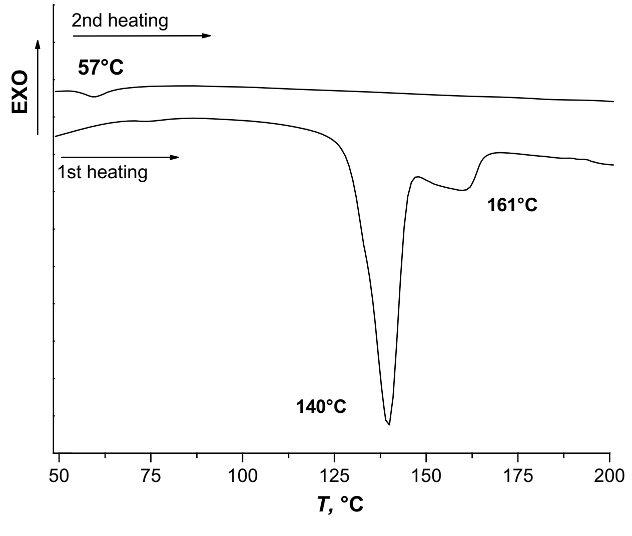

Fig. 5. DSC first and second heating curves for **4** (heating rate 10 K/min).

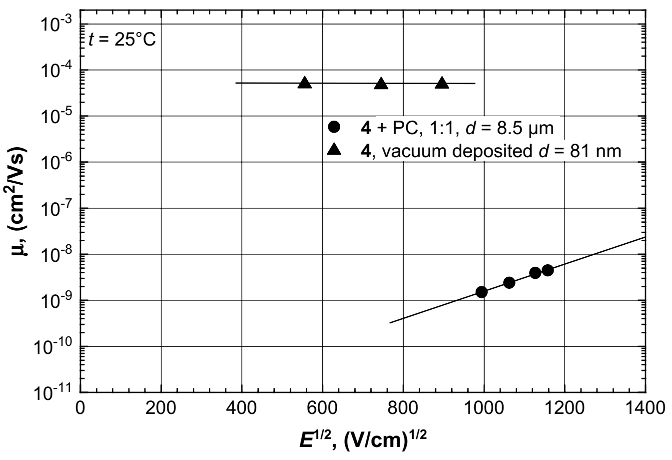

Fig. 7. Dependencies of hole drift mobility in vacuum-evaporated and spin-coated films 4.

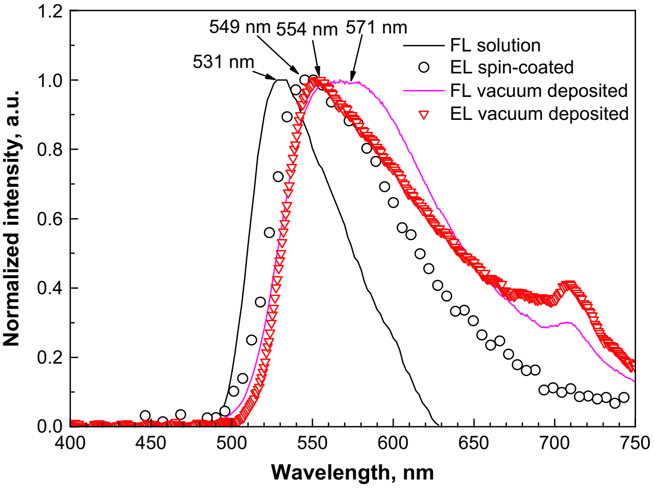

Fig. 8. Normalized FL and EL spectra of vacuum-deposited and spin-coated samples.

Carbaldehyde **3**, obtained after Vilsmeier formylation reaction, was used in Knoevenagel condensation with 1,4-phenylenediacetonitrile (in the presence of piperidine) and 1,4-bis-{1-nitrile-2-[(1-phenyl-3-propoxy)-1,2,3,4-tetrahydroquinolin-6-yl]ethenyl}benzene (**4**) was isolated.

The chemical structure of **4** was analyzed and identified through IR, UV, ^{1}H NMR and APCI-MS spectroscopy. The ^{1}H NMR spectra (Figs. 2 and 3) showing characteristic signals associated with the resonance of the hydrogen atoms of the intermediate **3** and goal product **4** confirm the molecular structure.

The mass spectrum and elemental analysis of **4** were consistent with the designed structure. Absorption band at 2201 cm^{-1} in IR spectrum of the twin molecule **4** confirms the presence of the nitrile groups in the isolated compound. Compared with 3-hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline (**1**), dimer **4** possesses significantly larger conjugated π-electron system. Significant bathochromic ($\approx$140 nm) and hyperchromic shifts were detected in absorption spectra (Fig. 4). Conjugated dimer **4** exhibited green fluorescence peaking at 530 nm.

The formation of the glassy state in **4** was confirmed by differential scanning calorimetry (DSC). These investigations revealed, that the twin molecule **4** exists both in crystalline and amorphous state. During the first heating cycle the sample produced endothermic polymorphic melting transitions at 140 °C and 161 °C with the predominant first crystalline phase (at 140 °C) (Fig. 5). No crystallization took place during second heating, only the glass transition was revealed at 57 °C.

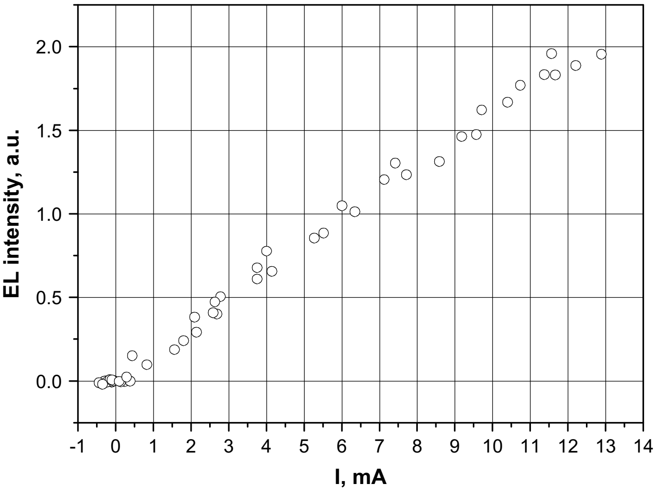

Fig. 9. EL intensity dependence on electric current in spin-coated sample.

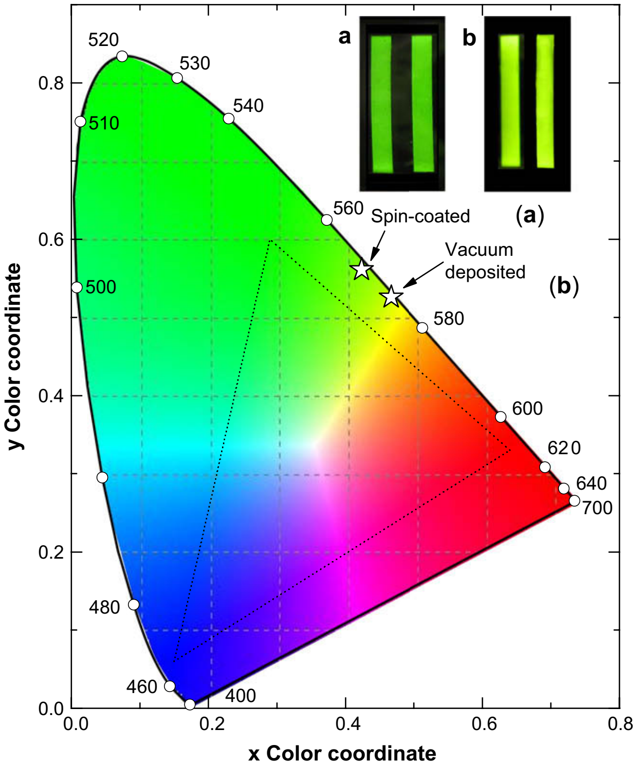

Fig. 10. The CIE chromaticity diagram. Stars indicate chromaticity coordinates of investigated samples.

The 1-phenyl-1,2,3,4-tetrahydroquinoline based glass-forming dimer **4** is soluble in common organic solvents such as acetone, ethyl acetate, chloroform, THF, etc. This superior solubility is mainly due to the flexible aliphatic bridging segment in tetrahydroquinoline fragment and additional propoxy groups.

Transparent films on substrates were prepared by the casting technique. The photoelectron emission in air spectrum for the investigated molecular glass **4** is shown in Fig. 6. The ionization potential (I_p) value for compound **4** was estimated to be 5.66 eV. Taking into account the energy gap between LUMO and HOMO levels of 2.48 eV as determined from optical spectra (Fig. 4), LUMO level position was found to be 3.2 eV.

Fig. 7 shows the room temperature dependencies of the hole drift mobility on the electric field for **4** in the composition with PC-Z and in the vacuum-deposited layer, when measured by xerographic time-of-flight (TOF) technique. The hole drift mobility in the cast layer of **4** was found to be 1.61×10^{-9} cm^2 V^{-1} s^{-1}at 10^6 V cm^{-1} electric field, whereas the hole drift mobility of the vacuum-deposited thin film of **4** was found to be independent of the electric field and attained the value of 5×10^{-5} cm^2/V s. Although the electron mobility was not estimated, the presence of electroluminescence suggested that electrons were injected into the thin emissive layer close to Al electrode [20].

The fluorescence spectrum of the vacuum-deposited film of **4** peaking at 571 nm demonstrates fairly good coincidence with electroluminescence spectrum centered at 554 nm (Fig. 8). However, the EL band is less broad and is more blueshifted with respect to FL band. Both spectra differ considerably from the spectrum of the material **4** solution, which peaks at 531 nm ($\lambda_{ex} = 450$ nm) (Fig. 8). The EL spectrum of the spin-coated sample has a maximum at 549 nm, which is in close proximity to that of the vacuum-deposited film. The EL intensity of the spin-coated sample exhibits linear dependence on electric current (Fig. 9) indicating well-balanced flows of electrons and holes.

Commission Internationale d'Eclairage (CIE) chromaticity coordinates (x, y) of spin-coated (0.42, 0.56) and vacuum-deposited (0.47, 0.53) samples, respectively, refer to the yellow-green part of the chromaticity diagram shown in Fig. 10.

In conclusion, the presented results demonstrate the potential of the novel material **4**, to be introduced into the OLED structures either by vacuum-evaporation or spin-coating techniques, and to be used as the multifunctional material capable of performing both charge transporting and light-emitting functions. The vacuum-deposited and spin-coated EL devices consisting of the key-compound **4** demonstrate similar emission spectra and almost pure yellow-green emission color. The energetic value of the LUMO level (3.2 eV) of the material **4** is found to be favorable for the electron injection from Ca sublayer (work function 2.8 eV), and more complicated from Al cathode (4.2 eV).

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