

New Conjugated Electroluminescent Triphenylamine-Containing Polymers with Side-Chain Pyridin-2-ylimidazo[1,5-*a*]pyridine Groups for Polymer Light-Emitting Diodes

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In recent years, light emitting diodes (LEDs) based on conjugated polymers have attracted considerable attention of researchers owing to their promising potential as new-generation full-color flat-panel displays [1–6]. An important problem that still remains unsolved is improving of the external quantum efficiency (EQE) of polymeric light-emitting diodes. The principle of operation of thin-layer LED structures is based on electroluminescence (EL) caused by radiative decay of excitons arising upon recombination of the electrons and holes injected from opposite electrodes into the polymer layer. Perfect conditions for producing intense EL and reaching high EQE values for a polymer are high electron–hole conductivity of the medium (that is, high mobility of charge carriers) and maintenance of the balance of injected charge carriers of different signs from the opposite electrodes into the material bulk. The mobilities of electrons and holes should not differ considerably. The main approach to the improvement of EQE for polymeric light-emitting diodes is development of luminescent bipolar (donor–acceptor) polymers [7, 8] where charge transport and light emission functions are concentrated within the same macromolecule. However, in donor–acceptor polymers, rather strong intramolecular charge transfer (ICT) occurs along the backbone, resulting in a decrease in the luminescence intensity despite better charge transport [7, 8]. An alternative strategy to overcome this issue is development of p-type polymers with n-type electronegative side groups [9]. According to this approach, a number of conjugated polymers with oxadiazole and quinoxaline side groups exhibiting promising electrolumines-

cence properties were successfully developed [10–12]. However, the application of these materials was held up due to complexity of the synthesis.

Development of simple methods for the synthesis of p-type polymers with electron-withdrawing side groups is quite a topical problem. Meanwhile, pyridin-2-ylimidazo[1,5-*a*]pyridine arouses particular interest as an n-type electron transporting building block due to high electron affinity, good thermal stability, and easy synthesis. Presumably, more pronounced electron-withdrawing properties of the pyridin-2-ylimidazo[1,5-*a*]pyridine ring compared with quinoline or quinoxaline ring would enhance the electron transport in polymers. In turn, the introduction of triphenylamine moieties having high hole conductivity and luminescent properties into conjugated polymer macromolecules would improve the electron–hole balance.

Polymers containing triarylamine (TAA) structures [13, 14] are among the most widely used hole-transport materials, because they are readily oxidized to give stable radical cations. The inclusion of TAA segments into a polymer chains may also improve their solubility and glass transition temperature. However, to our knowledge, donor–acceptor polymers with triphenylamine in the backbone and pyridin-2-ylimidazo[1,5-*a*]pyridine in the side chain have been unknown so far. These facts provide the opportunity to combine good processability and transport properties of charge carriers within the same macromolecule without deteriorating the luminescence efficiency.

This paper reports the synthesis of several new polyfluorene derivatives with triphenylamine moieties in the backbone and pyridin-2-ylimidazo[1,5-*a*]pyridine side groups having high thermal and oxidative stability and good mechanical and film-forming properties. The spectral and electroluminescence properties of LED structures of the obtained polymers were studied.

For the preparation of new copolyfluorenes, bis(4-bromophenyl)-[4'-(1"-pyridin-2-ylimidazo[1,5-*a*]pyridin-3-yl)phenyl]amine (**3**) was first synthesized. This

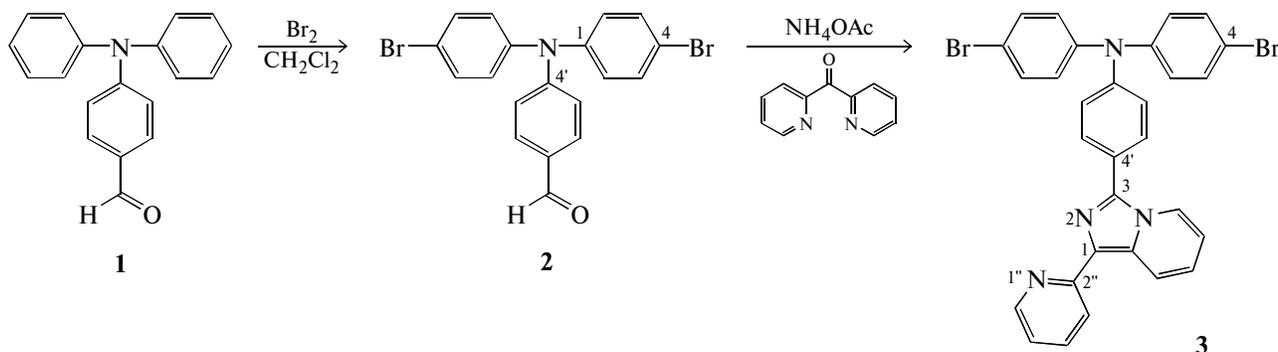
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compound contains triphenylamine groups, which enhance the hole transport, and bulky electron-withdrawing bipyridyl analogue, namely, pyridin-2-ylimidazo[1,5-*a*]pyridine moieties, which increase the electron affinity of the target macromolecular structures. Compound **3** was synthesized by bromination of

commercially available 4-(diphenylamino)benzaldehyde **1** and subsequent condensation of the resulting 4'-bis[(4-bromophenyl)amino]benzaldehyde **2** with 2,2'-dipyridyl ketone and NH_4OAc in glacial acetic acid to give target monomer **3** (Scheme 1).

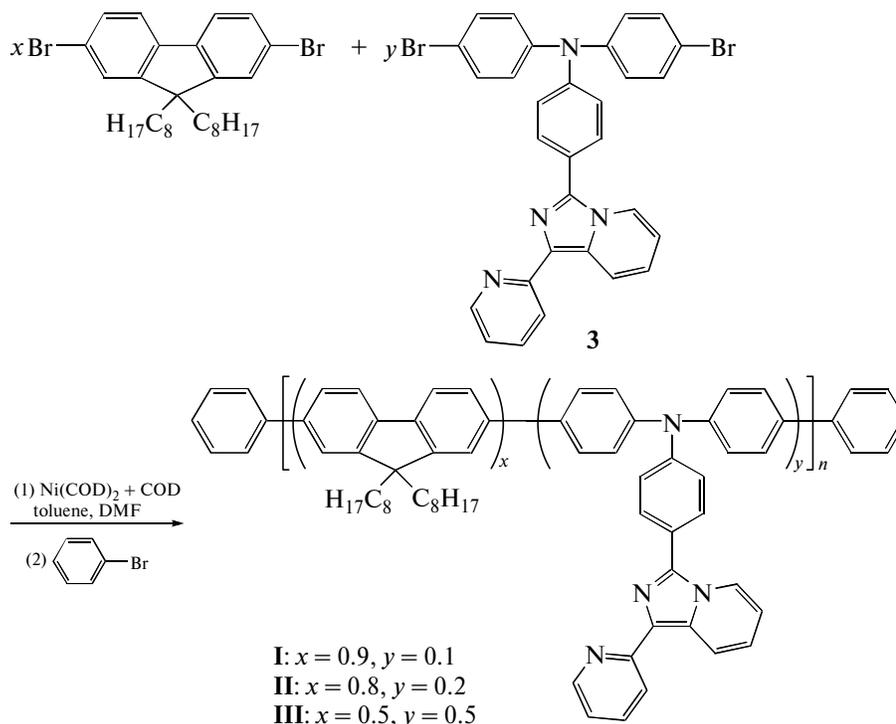


Scheme 1.

The composition and structure of the intermediate compounds and target product **3** were confirmed by elemental analysis data and ^1H and ^{13}C NMR spectroscopy. In particular, the ^1H NMR spectrum of **3** exhibits five doublets, two doublets of doublets, two triplets, and one multiplet in the low-field region ($\delta_{\text{H}} = 8.7\text{--}6.6$ ppm), which correspond to phenyl, pyridyl, and imidazopyridyl protons (Fig. 1a). In the ^{13}C NMR spectrum of product **3**, the range δ_{C} of

154.77 to 113.83 ppm contains nineteen signals, eight of these being due to the key quaternary carbon atoms (Fig. 1b).

The Yamamoto co-polycondensation of the obtained bipolar aromatic dibromide **3** and 2,7-dibromo-9,9-dioctylfluorene catalyzed by $\text{Ni}(\text{COD})_2$ in a toluene–DMF mixture afforded new conjugated copolyfluorenes **I–III** with different contents of the monomer units: 10, 20, and 50 mol % (Scheme 2).



Scheme 2.

The structures of polymers **I–III** were confirmed by ^1H NMR data. In the region $\delta_{\text{H}} = 8.75\text{--}6.73$ of the ^1H NMR spectrum, in addition to strong broadened signals for the fluorene aromatic protons, there are also well-resolved multiplets centered at $\delta_{\text{H}} = 8.30$, 8.33, 7.97, and 6.72 ppm, which are due to the monomer units of **3** present in the chain (Fig. 2). These results confirm that the triphenylamine and pyridin-2-ylimidazo[1,5-*a*]pyridine moieties were successfully introduced in the polymer chain. The $\delta_{\text{H}} = 2.11$, 1.13, and 0.80 ppm signals correspond to the aliphatic protons of the octyl groups in position 9 of the fluorene unit. The integrated intensity ratio of the aromatic and aliphatic protons corresponds to the assumed structures for all polymers and confirms the required content of unit **3** in the backbone. The mole percents of monomer units **3** in polymers **I**, **II**, and **III** are 10.1, 20.8, and 51.1 mol. %, respectively, as found from the data of elemental analysis.

All polymers are soluble in common organic solvents such as DMF, DMSO, THF, dimethylacetamide, toluene, and chloroform. Films with tensile strength of 83–90 MPa (Table 1) were obtained from the solutions. The number- and weight-average molecular weights and polydispersities of polymers **I–III** determined by gel permeation chromatography (elution with THF) vary in the ranges of $(2.19\text{--}4.17) \times 10^4$ (M_n), $(4.14\text{--}8.46) \times 10^4$ (M_w), and 1.89–2.03, respectively.

The thermal and thermooxidative characteristics of copolyfluorenes **I–III** were studied by thermomechanical (TMA) and thermogravimetric (TGA) analyses; the results are summarized in Table 1. All polymers exhibit high thermal stability. The glass transition temperatures of copolymers (T_g) found from TMA data are between 103 and 200°C. The temperatures of 10% weight loss ($T_{10\%}$) determined by TGA in air and under argon are in the ranges of 401–422 and 409–445°C, respectively. It follows from Table 1 that the introduction of bipolar heteroaromatic units into the polymer chain results in higher T_g and higher thermal stability of the copolyfluorenes compared with homopolyfluorene (HPF) ($T_g = 66^\circ\text{C}$, $T_{10\%} = 410^\circ\text{C}$), and these values tend to increase with an increase in the molar content of monomer units **3** in the backbone. The introduction of 50 mol. % of monomer units **3** into the polyfluorene chain made the largest contribution to the increase in the glass transition temperature of polymer **III**, which becomes as high as 200°C with simultaneous maximum increase in the thermal stability with respect to other copolymers synthesized.

The optical properties of polymers **I–III** were studied by UV and fluorescence spectroscopy; the

results are summarized in Table 2. The absorption spectra of copolymers **I–III** (Fig. 3) show similar patterns, the peaks being in the range of $\lambda = 370\text{--}387$ nm. As the content of comonomer **3** in the backbone increases, the polymer absorption band shifts hypsochromically.

The forms and positions of the bands are apparently related to low-energy $\pi\text{--}\pi^*$ electron transitions in the polyconjugated macromolecular chain. The short-wavelength shift relative to that of HPF ($\lambda_{\text{max}}^{\text{abs}} = 390$ nm) is due to substituents that affect the polyconjugation length of copolymers **I–III**, particularly, somewhat decrease the polyconjugation length compared with the parent polyfluorene.

All polymers **I–III** exhibit intense photoluminescence (PL), which is manifested as two bands in the blue region, which do not differ much from those in the fluorescence spectrum of HPF and have maxima at 417–419 and 433–442 nm (which are also the same as for HPF). Their structure does not practically change upon excitation of the system either at the absorption maximum or at a shorter wavelength, i.e., in the absorption region of substituents. These bands should be assigned to 0–0, 0–1, and 0–2 intrachain singlet transitions in HPF. Thus, nonradiative transfer of the excitation energy to the polyconjugated chain occurs in the macromolecular system, apparently, according to the Forster mechanism, because the absorption and PL spectra substantially overlap, which is true for all of copolymers **I–III**. The introduction of monomer unit **3** has almost no influence on the positions of the principal PL bands of the copolyfluorenes, and only a considerable increase in the intensity of the longer-wavelength band compared to the shorter-wavelength band occurs upon increase in the molar content of the monomer units **3**.

From the onset of absorption spectra of **I–III** in solution ($\lambda_{\text{ons}}^{\text{ads}}$), the optical width of the forbidden gap (E_g^{opt}) was calculated; the results are presented in Table 2. The band gap of the polymers in question remains almost invariable upon changes in the structure and molar amount of comonomer **3** introduced in the polyfluorene backbone.

The electrochemical properties of **I–III** were studied by cyclic voltammetry; the results are also presented in Table 2. The cyclic voltammograms of the copolymers exhibit anodic oxidation peaks at positive voltage; no cathodic reduction peaks are present at negative voltage. From the potential of the onset of oxidation ($\lambda_{\text{ons}}^{\text{ox}}$), the energies of the highest occupied molecular orbital (HOMO) for **I–III** (Table 2) were found in the following way:

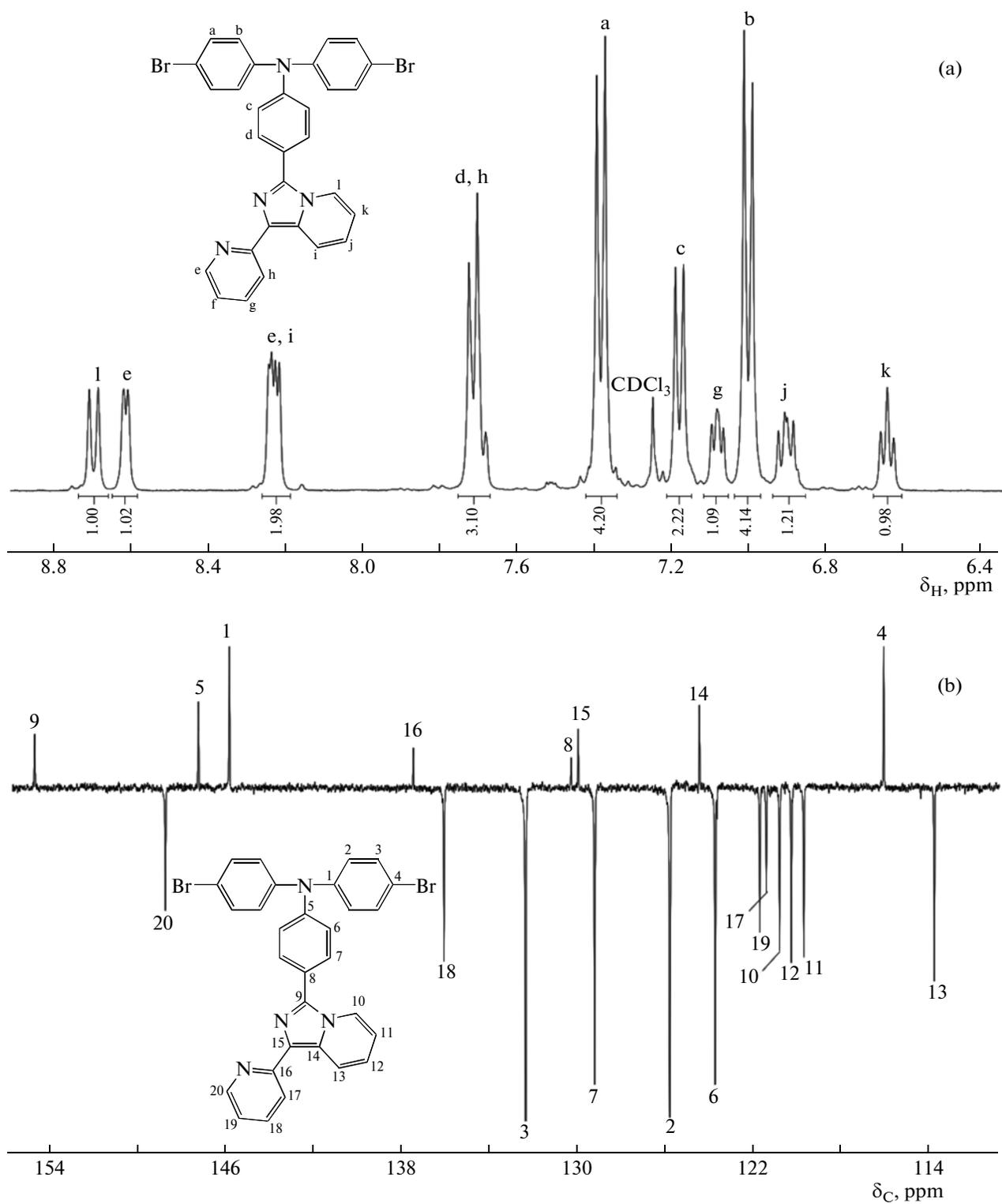


Fig. 1. (a) ^1H NMR and (b) ^{13}C NMR spectra of bis(4-bromophenyl)-[4'-(1''-pyridin-2''-ylimidazo[1,5-*a*]pyridin-3-yl)phenyl]amine **3** in CDCl_3 .

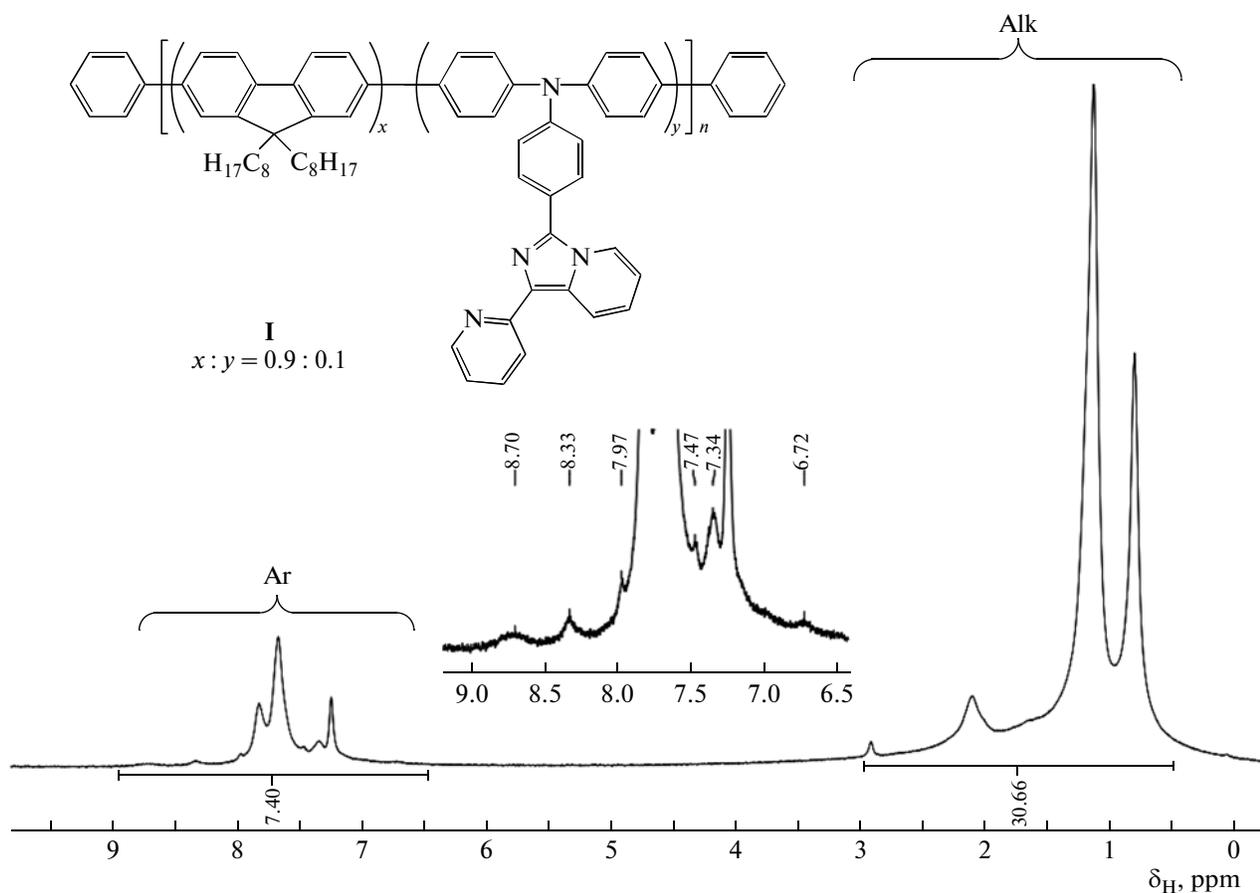


Fig. 2. ^1H NMR spectrum of copolymer **I** in CDCl_3 .

$$E^{\text{HOMO}} = E_{\text{Fc}}^{\text{HOMO}} + e(E_{\text{ons}}^{\text{ox}} - E_{\text{ons}(\text{Fc})}^{\text{ox}} - E_{\text{el}}),$$

where $E_{\text{Fc}}^{\text{HOMO}} = -4.8$ eV, $E_{\text{el}} = 0.21$ eV, $E_{\text{ons}(\text{Fc})}^{\text{ox}} = 0.23$ eV (found from cyclic voltammetry data for ferrocene used as the standard). Thus, $E^{\text{HOMO}} = -4.36$ eV + $eE_{\text{ons}}^{\text{ox}}$. The lowest unoccupied molecular orbital (LUMO) energies were calculated as $E^{\text{LUMO}} = E^{\text{HOMO}} + E_{\text{g}}^{\text{opt}}$. It follows from Table 2 that the HOMO energy of **I–III** gradually increases with increase in the content of monomer units **3**.

In order to study the effect of monomer units **3** introduced into the polyfluorene backbone on the electroluminescence properties of the obtained copolymers, LED structures with a hole injection layer based on a dispersion of polyethylenedioxythiophene stabilized by polystyrene sulfonate (PEDOT/PSS) and a thin electron injection layer based on lithium fluoride were fabricated. On voltage application on the LED structures, blue EL was observed (Fig. 3) with color coordinates given in Table 3 and in Fig. 4a. The EL spectra are shifted to longer wavelengths relative to PL bands, which is usual for organic materials.

The EL spectra of copolyfluorenes **I** and **II** are broad intense bands with peaks at 476 and 469 nm, respectively (Fig. 3), which corresponds to blue radiation with color coordinates $x = 0.191$, $y = 0.249$ (for **I**, Table 3). An increase in the content of monomer units **3** in the macromolecule entails a slight hypsofluoric shift of the principal EL band (by 7 nm for copolymers **I** and **II**).

The brightness of devices based on copolymers **I** and **II** and on previously prepared HPF (used for comparison) was measured to characterize the emission properties of the LED structures. The dependence of brightness on the applied voltage is presented in Fig. 4b.

It follows from the data that a device based on the active layer consisting of pure HPF has a brightness of 1.4 Cd/m^2 at 10 V. Light emitting diodes based on copolymers **I** and **II** had brightnesses of 5.9 and 7.3 Cd/m^2 at the same voltage. The introduction of 10 and 20 mol. % of monomer units **3** into the HPF structure increased the brightness of emission 4.2- and 5.2-fold, respectively.

Table 1. Molecular-weight and thermal characteristics of copolyfluorenes I–III

Polymer	$M_n \times 10^{-4}$	$M_w \times 10^{-4}$	M_w/M_n	$T_g, ^\circ\text{C}$	$T_{10\%}, ^\circ\text{C}^*$	Film properties	
						σ, MPa	$\epsilon, \%$
I	4.17	8.46	2.03	103	$\frac{401}{409}$	89	6
II	2.19	4.14	1.89	174	$\frac{412}{426}$	83	7
III	2.78	5.39	1.94	200	$\frac{422}{445}$	90	5

* In air, above the line, and under argon, below the line.

Table 2. Optical and electrochemical properties of copolyfluorenes I–III

Polymer	$\lambda_{\text{max}}^{\text{abs}}$	$\lambda_{\text{max}}^{\text{PL}}$	$\lambda_{\text{ons}}^{\text{abs}}$	$E_{\text{ons}}^{\text{ox}}, \text{V}$	E^{HOMO}	E^{LUMO^*}	E_g^{opt}
	nm				eV		
I	387	419; 440	415	1.28	−5.64	−2.65	2.99
II	382	419; 442	418	1.15	−5.51	−2.54	2.97
III	370	417; 433	420	1.10	−5.46	−2.51	2.95

* The LUMO values were found from the difference of E_g^{opt} and E^{HOMO} .

Thus, we synthesized electroactive polymers of a new type having photo- and electroluminescence properties incorporating donor–acceptor groups as functional substituents. The partial replacement of dialkylfluorene by the multifunctional monomer unit **3** in the copolyfluorene backbone gave rise to some advantages, in particular, higher thermal and chemical stability, higher electron affinity within the same molecule without deterioration of the emission properties of polyfluorenes, considerable improvement and extension of electron-transport and electroluminescence properties. Moreover, replacement of alkyl substituents by bulky heteroaromatic moieties reduces the aggregation and phase separation, which are inherent in poly(dialkylfluorene) macromolecules in the production of such devices. The electroactive conjugated copolyfluorenes based on bipolar monomers can serve as effective electroluminescence materials in polymeric LEDs. Moreover, pyridin-2-ylimidazo[1,5-*a*]pyridine moieties contained in copolymers I–III, like 2,2'-bipyridine, exhibit chelating ability towards a

broad range of metal ions and are attractive as recognition receptors. Thus, copolyfluorenes I–III can also be used as chemosensors for various analytes.

EXPERIMENTAL

Synthesis of Monomers

4'-Bis[(4-bromophenyl)amino]benzaldehyde (2) was prepared by a reported procedure [15]. Yield 5.38 g (72%). Mp = 152–154°C; lit. [15]: Mp = 156–157°C.

$^1\text{H NMR}$ (CDCl_3 , 400 MHz, δ , ppm): 9.82 (s, 1H), 7.70 (d, $J = 8.8$ Hz, 2H), 7.43 (dd, $J = 9.3, 2.5$ Hz, 4H), 7.06–6.98 (m, 6H).

$^{13}\text{C NMR}$ (CDCl_3 , 100 MHz, δ , ppm): 190.29, 152.22, 144.87, 132.80, 131.26, 129.99, 127.26, 120.29, 117.95.

For $\text{C}_{19}\text{H}_{13}\text{Br}_2\text{NO}$ anal. calcd. (%): C, 52.93; H, 3.04; N, 3.25; Br, 37.07.

Found (%): C, 52.72; H, 2.99; N, 3.32; Br, 37.25.

Bis(4-bromophenyl)-[4'-(1''-pyridin-2''-ylimidazo[1,5-*a*]pyridin-3-yl)phenyl] amine (3). 2,2'-Bipyridyl ketone (0.21 g, 1.16 mol) and glacial acetic acid (7.0 mL) were charged into a 25 mL three-necked round-bottom flask equipped with a reflux condenser, argon inlet, and a magnetic stirrer. The mixture was stirred at room temperature until the solid completely dissolved, and 4'-bis[(4-bromophenyl)amino]benzaldehyde **2** (1.0 g, 2.32 mmol) and NH_4OAc (0.45 g, 5.80 mmol) were added. The reaction mixture was stirred at 80°C under argon for 1.5 h and cooled to

Table 3. Electroluminescence properties of copolyfluorenes I–III

Polymer	$\lambda_{\text{max}}^{\text{el}}, \text{nm}$	Color coordinates	
		x	y
I	476	0.191	0.249
II	470	0.206	0.309
III	462	0.213	0.348

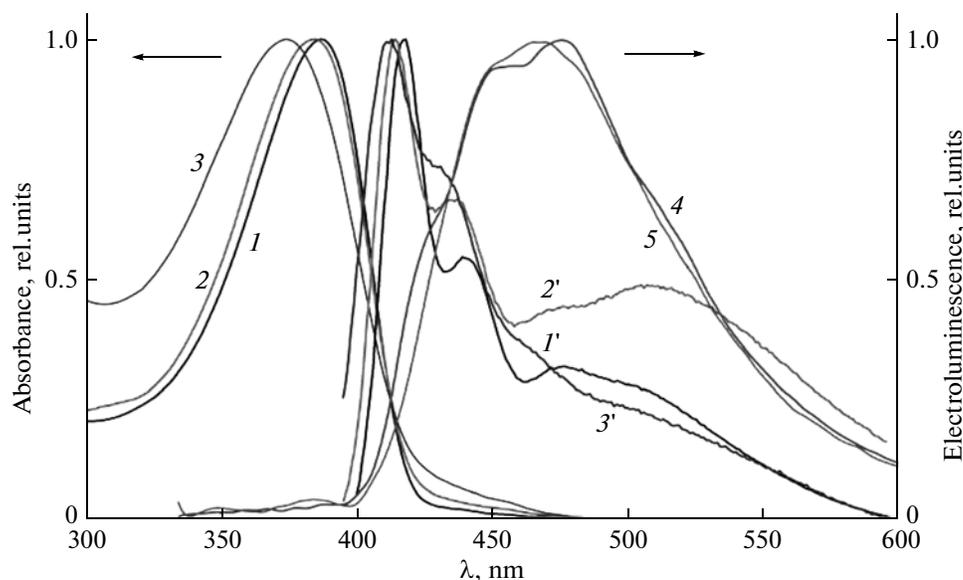


Fig. 3. Absorption spectra of polymers (1) I, (2) II, and (3) III; photoluminescence spectra of (1') I, (2') II, and (3') III in chloroform ($c = 1 \times 10^{-5}$ mol/L); and electroluminescence spectra of (4) I and (5) II in a film.

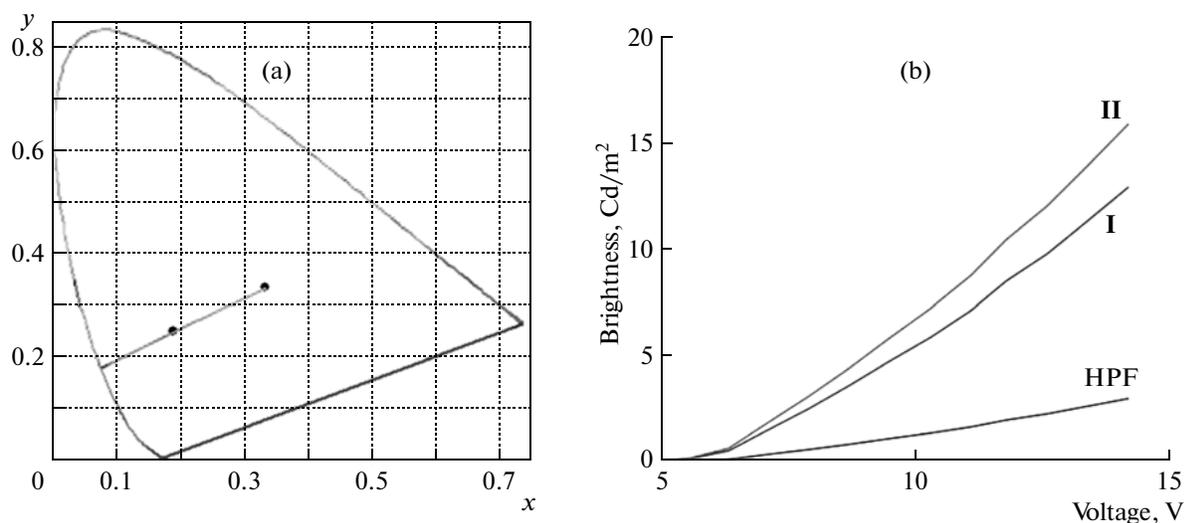


Fig. 4. (a) Color coordinates of a light emitting diode device based on polymer II and (b) brightness of the electroluminescence of copolymers I and II and homopolyfluorene (HPF) vs. applied voltage.

room temperature. The mixture was poured into 100 mL of ice water and extracted with CH_2Cl_2 . The organic layer was washed with doubly distilled water, dried with MgSO_4 , and concentrated on a rotary evaporator. The product was purified by column chromatography (elution with toluene : ethyl acetate = 1 : 1) to give yellow crystals. Yield 0.57 g (82%). Mp = 174–175°C.

^1H NMR (CDCl_3 , 400 MHz, δ , ppm): 8.70 (d, $J=9.2$ Hz, 1H), 8.61 (d, $J=4.2$ Hz, 1H), 8.23 (dd, $J=7.4, 3.5$ Hz, 2H), 7.70 (t, $J=8.6$ Hz, 3H), 7.38 (d, $J=$

8.7 Hz, 4H), 7.18 (d, $J=8.5$ Hz, 2H), 7.12–7.05 (m, 1H), 7.00 (d, $J=8.7$ Hz, 4H), 6.91 (dd, $J=9.0, 6.5$ Hz, 1H), 6.64 (t, $J=6.7$ Hz, 1H).

^{13}C NMR (CDCl_3 , 100 MHz, δ , ppm): 154.77, 148.82, 147.32, 145.91, 137.54, 136.14, 132.43, 130.04, 129.29, 125.87, 124.52, 123.81, 121.78, 121.48, 120.88, 120.34, 119.77, 116.13, 113.83.

For $\text{C}_{30}\text{H}_{20}\text{N}_4\text{Br}_2$ anal. calcd. (%): C, 60.42; H, 3.38; N, 9.40.

Found (%): C, 60.69; H, 3.27; N, 9.07.

Synthesis of Copolymers

Copolyfluorene I. A 25 mL three-necked flask equipped with a reflux condenser, a magnetic stirrer, and argon inlet was charged with Ni(COD)₂ (1.000 g, 3.636 mmol), 2,2'-bipyridine (0.5600 g, 3.636 mmol), and 1,5-cyclooctadiene (0.3933 g, 3.636 mmol). Dry DMF (5 mL) was added, and the mixture was stirred at 80°C for 30 min. A solution of 2,7-dibromo-9,9-dioctylfluorene (0.7898 g, 1.44 mmol) and bis(4-bromophenyl)-[4'-(1"-pyridin-2"-ylimidazo[1,5-*a*]pyridin-3-yl)phenyl]amine (**3**) (0.095 g, 0.16 mmol) in a 4 : 1 toluene–DMF mixture (12.5 mL) was added through a septum. The mixture was stirred at 80°C for 48 h, and bromobenzene (0.06 g, 0.39 mmol) was added. The mixture was stirred for more 5 h, cooled to room temperature, and poured into a 2 : 1 methanol–concentrated HCl mixture (250 mL). The precipitate was filtered off, dissolved in chloroform, and reprecipitated with methanol. The polymer was purified by methanol and acetone extraction in a Soxhlet apparatus for 24 h and dried in vacuum at 70°C. Yield 75%.

¹H NMR (CDCl₃, 400 MHz, δ_H, ppm): 8.75–7.73 (m, Ar H), 2.93–0.48 (m, Alk H).

For **I** anal. calcd. (%): C, 88.55; H, 9.66; N, 1.79.

Found (%): C, 88.19; H, 9.71; N, 1.63.

Copolymers **II** and **III** were synthesized in a similar way.

Copyfluorene II. Yield 81%. ¹H NMR (CDCl₃, 400 MHz, δ_H, ppm): 8.72–7.68 (m, Ar H), 2.97–0.52 (m, Alk H).

For **II** anal. calcd. (%): C, 88.06; H, 9.04; N, 2.90.

Found (%): C, 87.82; H, 9.18; N, 2.78.

Copyfluorene III. Yield 77%. ¹H NMR (CDCl₃, 400 MHz, δ_H, ppm): 8.73–7.75 (m, Ar H), 2.94–0.50 (m, Alk H).

For **III** anal. calcd. (%): C, 85.87; H, 7.27; N, 6.86.

Found (%): C, 85.36; H, 7.36; N, 6.72.

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