New Conjugated Monomers and Oligomers Derived from Chalcones and Containing Thiophene, Pyrrole, and Pyrimidine Fragments

A. Yu. Bushueva^{*a*}, E. V. Shklyaeva^{*b*}, and G. G. Abashev^{*a*,*b*}

^a Institute of Technical Chemistry, Ural Branch, Russian Academy of Sciences, Perm, Russia ^b Natural Science Institute, Perm State University, Perm, Russia

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Abstract—4New hybrid systems based on a series of chalcones and containing pyrimidine, thiophene, and pyrrole fragments were synthesized, and their electrochemical properties were studied.

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Conjugated polymers exhibiting semiconductor properties acquired very high importance recently as active materials for optical devices such as polymeric light-emitting diodes (PLEDs), displays, photovoltaic cells, and lasers, and also for field transistors and sensors [1-13].

A particular place among conjugated polymers is occupied by polymers that contain in the conjugation chain electron-deficient and electron-excessive heterocycles simultaneously. Introduction of a pyrimidine ring into the center of a banana-shaped molecule strongly alters the physical properties of the molecule such as mesomorphism, fluorescence, and solvatochromism [14]. π -Deficient heterocycles can be readily reduced, and polymers prepared from such comonomers are readily n-doped. Introduction into the pyrimidine structure of external π -excessive heterocycles allows preparation of polymers and oligomers that can be both *n*- and *p*-doped. Such compounds are of much interest, because, e.g., in fabrication of emitting devices smaller number of polymer layers in the device structure is required, which makes the device more efficient [15].

In π -conjugated materials, pyrimidines can be used as electron-deficient fragments in the center of the molecule, with electron-donor fragments linked to them. Previously we prepared novel promising materials of the structure D–A–D (donor–acceptor–donor), containing electron-excessive thiophene and electrondeficient pyrimidine rings. By their electrochemical polymerization, we prepared polymer films on the surface of an indium–tin oxide (ITO) working electrode [16]. Compounds in which pyrimidine fragments are in a common conjugation chain with other heterocycles and with aromatic rings have also been synthesized [17, 18].

The goal of this study is preparation of polymeric materials using chalcone derivatives containing pyrimidine, thiophene, and pyrrole fragments.

The use of *p*-aminoacetophenone as methylene component in preparation of chalcones allowed the range of new conjugated monomers to be considerably expanded. The starting chalcone, (4-aminophenyl)-3-(thiophen-2-yl)prop-2-en-1-one (1), was prepar-ed by heating of thiophenecarbaldehyde with *p*-amino-acetophenone in the presence of an aqueous alkali solution (Scheme 1).

The reaction of 1 with 2,5-dimethoxytetrahydrofuran yielded pyrrole 2. Its reaction with guanidine sulfate, followed by addition of hydrogen peroxide [19], yielded pyrimidine 3 containing in the conjugation chain electron-excessive substituents: thiophen-2-yl in position 4 and p-(1H-pyrrol-1-yl)phenyl in position 6 of the pyrimidine ring. To prepare pyrimidine 5 containing



Here a, d is treatment with 2,5-dimethoxytetrahydrofuran and acetic acid; b, c is treatment with guanidine sulfate and H_2O_2 .

two pyrrole rings, we also used chalcone **1**, from which we initially prepared 2-amino-4-(aminophenyl)-6-(thiophen-2-yl)pyrimidine **4**. The reaction of **4** with 2,5-dimethoxytetrahydrofuran yielded 2-(1*H*-pyrrol-1-yl)-4-[4-(1*H*-pyrrol-1-yl)phenyl]-6-(thiophen-2-yl) pyrimidine (**5**) (Scheme 1).

Oligomers and polymers containing a 3,4-ethylenedioxythiophene fragment, which has a lower oxidation potential than thiophene and readily undergoes electrochemical polymerization, play an important role in the development of diverse electronic devices. Therefore, it was interesting to replace in chalcone 1 the thiophene ring by the ethylenedioxythiophene ring. With 3,4-ethylenedioxythiophenecarbaldehyde taken instead of thiophenecarbaldehyde, following Scheme 1, we prepared 1-(4-aminophenyl)-3-(3,4ethylenedioxythiophen-2-yl)prop-2-en-1-one (6) and 1-[4-(pyrrol-1-yl)phenyl]-3-(3,4-ethylenedioxythiophen-2-yl)prop-2-en-1-one (7). From these compounds, in turn, we synthesized 2-amino-4-(4aminophenyl)-6-(3,4-ethylenedioxythiophen-2-yl) pyrimidine (8) and 2-(pyrrol-1-yl)-4-[4-(pyrrol-1-yl) phenyl]-6-(3,4-ethylenedioxythiophen-2-yl)pyrimidine (9). Replacement of thiophenecarbaldehyde by 2-pyrrolecarbaldehyde led to the synthesis of chalcone 1-(4-aminophenyl)-3-(1H-pyrrol-2-yl)prop-2-en-10. 1-one. By the reaction of *p*-aminoacetophenone with 2,5-dimethoxytetrahydrofuran in refluxing acetic acid, we prepared 1-acetyl-4-(pyrrol-1-yl)benzene (11), whose condensation with 2-pyrrolecarbaldehyde in an alcoholic alkaline medium yielded chalcone 12 (Scheme 2).

The use of *p*-aminoacetophenone in the Paal-Knorr reaction also allows preparation of conjugated monomers, oligomers, and polymers containing various heterocycles in the conjugation chain. Starting 1,4-di(thiophen-2-yl)butane-1,4-dione from and *p*-aminoacetophenone, we synthesized a substituted acetophenone, 1-(4-acetylphenyl)-2,5-di(thiophen-2yl)-1*H*-pyrrole **13**. By its condensation in an alcoholic alkaline medium, we prepared chalcones containing thiophene (14) and 3,4-ethylenedioxythiophene (15) fragments. For chalcone 15, this synthesis failed. Therefore, it was synthesized by the Paal-Knorr reaction from 1,4-di(2-thienyl)butane-1,4-dione and chalcone 6 in xylene in the presence of *p*-toluenesulfonic acid (TsOH) (Scheme 3).

Electrochemical polymerization of conjugated monomers is one of the main procedures for direct preparation of polymer films in both doped and neutral states on electrode surfaces. Thiophenes are used as terminal groups of the monomer most frequently; pyrroles are also used for this purpose. For example, polymers containing pyridine and bipyridine electrondeficient fragments in the center of the monomer were prepared by this procedure. Generally, polymers with electron-deficient heterocycles located in the center of





14

the monomer are less stable in the *p*-doped state than in the *n*-doped state [20, 21]. The electrochemical behavior of chalcones was studied previously in [22–24]. It was shown that chalcones undergo irreversible oxidation in the anodic region and reduction and dimerization in the cathodic region. The possibility of preparing polymer films based on chalcones containing carbazole fragments was demonstrated, and their electrochromic properties and redox stability was examined.

13

EXPERIMENTAL

We used the following materials: thiophene, 4-aminoacetophenone, 2,5-dimethoxytetrahydrofuran (Alfa Aesar), silica gel (Lancaster), and pyrrole (Aldrich). The ¹H NMR spectra were recorded on a Mercury plus 300 spectrometer, with HMDS as internal reference. Electrochemical studies were performed on an IPC-compact device with an EM-04 electrochemical sensor. Mass-spectrometric analysis was performed with an Agilent Technologies 6890N/5975B gas chromatograph–mass spectrometer (HP-5ms column, 30×0.25 mm, 0.25 µm, carrier gas He, ionization by electron impact, 70 eV). The UV spectra were recorded with an SF 2000 spectrophotometer (OKB Spektr). The reaction progress and the product purity were monitored by TLC on Silufol UV-254 plates.

15

 $1-R_1-3-R_2$ -Prop-2-en-1-ones (chalcones) 1, 2, 6, 7, and 10 (general procedure). A flask equipped with a stirrer was charged with 150 ml of a 2% NaOH

RUSSIAN JOURNAL OF APPLIED CHEMISTRY Vol. 83 No. 8 2010

solution, 0.03 mol of appropriate ketone, and 0.03 mol of appropriate aldehyde. The mixture was refluxed for 2 h. The precipitate formed after cooling was filtered off and washed on the filter with a double excess of cold water. The chalcones obtained were purified by recrystallization from ethanol or by column chromatography on silica gel (eluent CH_2Cl_2).

1-(4-Aminophenyl)-3-(thiophen-2-yl)prop-2-en-1-one (1). Yield 64%, mp 94–96°C. ¹H NMR spectrum (CDCl₃, δ, ppm, *J*, Hz): 4.14 br.s (2H, NH₂), 6.68 d (2H, Ph, *J* 8.7), 7.06 t (1H, CH-Th, *J* 3.6), 7.31–7.38 m (2H, SC<u>CH-</u>Th, CO–<u>CH</u>=CH), 7.87–7.93 m (4H, SCH-Th, Ph, CO–CH=<u>CH</u>). IR spectrum, v, cm⁻¹: 3450 (NH₂), 1625 (C=O), 1596 (C=C), 1557 (C=C).

1-(4-Aminophenyl)-3-(3,4-ethylenedioxythiophen-2-yl)prop-2-en-1-one (6). Yield 84%, mp 222–225°C. ¹H NMR spectrum (CDCl₃, δ , ppm, *J*, Hz): 4.10 s (2H, NH₂), 4.23 m (2H, OCH₂), 4.32 m (2H, OCH₂), 6.43 s (1H, Th), 6.68 d (2H, Ph, *J* 8.7), 7.29–7.34 d (1H, CH=<u>CH</u>–CO, *J* 15.3), 7.78–7.83 d (1H, <u>CH</u>=CH–CO, *J* 15.0), 7.88 d (2H, Ph, *J* 8.7). IR spectrum, v, cm⁻¹: 3447 (NH₂), 1639 (C=O), 1595 (C=C), 1570 (C=C).

1-[4-(Pyrrol-1-yl)phenyl]-3-(thiophen-2-yl)prop-2-en-1-one (2) was prepared by refluxing for 1 h 1.15 g (0.005 mol) of chalcone 1 and 0.66 g (0.005 mol) of 2,5-dimethoxyTHF in a minimal amount of glacial CH₃COOH. After that, the reaction mixture was cooled and poured into ice-cold water. The precipitate that formed was filtered off, dried in air, and purified by recrystallization from ethanol or by column chromatography (silica gel, eluent CH₂Cl₂). Yield 62%, mp 140–141°C. ¹H NMR spectrum (CDCl₃, δ , ppm, J, Hz): 6.39 t (2H, pyrrole, J 2.25), 7.09 t (1H, CH-Th, J 3.6), 7.17 t (2H, pyrrole, J 2.25), 7.31–7.37 m (2H, SCCH-Th, CH=CH-CO), 7.43 d (1H, SCH-Th, J 5.1), 7.49 d (2H, Ph, J 8.7), 7.94–7.99 d (1H, CH=CH–CO, J 15.3), 8.08 d (2H, Ph, J 8.7). Mass spectrum, m/z (I, %): 279.00 [M⁺] (100.0), (C₁₇H₁₃NOS, M_{calc} 279.36). IR spectrum, v, cm⁻¹: 1650 (C=O), 1600 (C=C), 1570 (C=C). UV spectrum (CH₂Cl₂), λ_{max} 246.0, 350.9 nm.

3-(3,4-Ethylenedioxythiophen-2-yl)-1-[4-(pyrrol-1-yl)phenyl]prop-2-en-1-one (7) was prepared from 1.44 g (0.005 mol) of 1-(4-aminophenyl)-3-(3,4ethylenedioxythiophen-2-yl)prop-2-en-1-one (6) by the procedure similar to preparation of chalcone **2**. The product was chromatographed on silica gel (eluent CH_2Cl_2). Yield 78%, mp 236–238°C. ¹H NMR spectrum (CDCl₃, δ , ppm, *J*, Hz): 4.25 m (2H, OCH₂), 4.34 m (2H, OCH₂), 6.39 t (2H, pyrrole, *J* 1.95), 6.50 s (1H, Th), 7.17 t (2H, pyrrole, *J* 2.1), 7.31–7.36 d (1H, CH=<u>CH</u>-CO, *J* 15.3), 7.48 d (2H, Ph, *J* 8.7), 7.84– 7.89 d (1H, <u>CH</u>=CH–CO, *J* 15.0), 8.08 d (2H, Ph, *J* 8.4). IR spectrum, v, cm⁻¹: 1650 (C=O), 1610 (C=C), 1585 (C=C), 1560 (C=C). UV spectrum (CH₂Cl₂), λ_{max} 245.0, 370.8 nm.

1-(4-Aminophenyl)-3-(1*H***-pyrrol-2-yl)prop-2en-1-one (10). ¹H NMR spectrum (CDCl₃, δ, ppm,** *J***, Hz): 4.10 br.s (2H, NH₂), 6.33 m (1H, pyrrole), 6.63 m (1H, pyrrole), 6.94 m (2H, pyrrole), 7.15 d (1H, COCH,** *J* **15.3), 7.70 d (1H, CH,** *J* **15.3), 7.80 dd (2H, phenyl,** *J* **8.7), 7.92 dd (2H, phenyl,** *J* **8.7), 8.82 (1H, NH).**

1-(4-Acetylphenyl)-2,5-di(2-thienyl)-1*H*pyrrole (13). To a suspension of 1.25 g (0.005 mol) of 1,4-di(thiophen-2-yl)-1,4-butanedione and 0.95 g (0.007 mol) of p-aminoacetophenone in 85 ml of absolute toluene, we added 0.11 g (0.00058 mol) of *p*-toluenesulfonic acid monohydrate. The mixture was refluxed for 24 h (the reaction progress was monitored by TLC), cooled, and washed with water. The organic layer was dried over Na₂SO₄, the solvent was distilled off, and the dark solid residue was chromatographed on silica gel (eluent CH₂Cl₂). Yield 5%, mp 176–177°C. ¹H NMR spectrum (CDCl₃, δ , ppm, *J*, Hz): 2.63 s (3H, COCH₃), 6.50 d (2H, thiophene, *J* 3.6), 7.07–7.09 dd (2H, thiophene, *J* 5.1), 7.34–7.37 dd (2H, phenyl, *J* 9.01), 7.98 (2H, phenyl, *J* 7.84).

1-{4-[2,5-Di(2-thienyl)pyrrol-1-yl]phenyl}-3-(2-thienyl)prop-2-en-1-one (14) was prepared similarly to **13** and purified by column chromatography (silica gel, eluent CH₂Cl₂). Yield 3%, mp 226–228°C. ¹H NMR spectrum (CDCl₃, δ, ppm, *J*, Hz): 6.51 d (3H, 3SC<u>CH</u>-Th, *J* 3.75), 6.54 s (2H, 2CH-pyrrole), 6.81 t (3H, 3CH-Th, *J* 3.6), 7.08 d (3H, 3SCH-Th, *J* 5.1), 7.32–7.37 d (1H, CO-<u>CH</u>=CH, *J* 15.0), 7.39 d (2H, Ph, *J* 8.4), 7.96–8.01 d (1H, CO–CH=<u>CH</u>, *J* 15.3), 8.03 d (2H, Ph, *J* 9.0). IR spectrum, *v*, cm⁻¹: 1719 (C=O), 1578 (C=C). UV spectrum (CH₂Cl₂), λ_{max} 270.9, 339.9 nm.

1-{4-[2,5-Di(2-thienyl)pyrrol-1-yl]phenyl}-3-(3,4ethylenedioxythiophen-2-yl)prop-2-en-1-one (15). A suspension of 0.13 g (37 mmol) of 1-(4-acetylphenyl)-2,5-di(2-thienyl)-1*H*-pyrrole (13) and 0.06 g (37 mmol) of 3,4-ethylenedioxythiophene-2-carbaldehyde in 20 ml of methanol was heated until the reactants fully dissolved. Then the mixture was cooled to room temperature, and a solution of 0.56 g of KOH in 10 ml of methanol was added. The mixture was refluxed for 5 h, and the precipitate that formed was filtered off, dried in air, and chromatographed on a column (silica gel, eluent CH₂Cl₂). Yield 37%, mp 197–199°C. ¹H NMR spectrum (CDCl₃, δ , ppm, *J*, Hz): 4.25 m (2H, OCH₂), 4.35 m (2H, OCH₂), 6.48 d (2H, 2SC<u>CH</u>-Th), 6.51 s (1H, SCH-Th), 6.54 s (2H, 2CH-pyrrole), 6.81 t (2H, 2CH-Th, *J* 3.6), 7.07 d (2H, 2SCH-Th, *J* 5.1), 7.33–7.38 d (1H, CO–<u>CH</u>=CH, *J* 15.0), 7.37 d (2H, Ph, *J* 8.7), 7.85–7.90 d (1H, CO–CH=<u>CH</u>, *J* 15.6), 8.03 d (2H, Ph, *J* 9.0).

1-[4-(Pyrrol-1-yl)phenyl]-3-(pyrrol-2-yl)prop-2-en-1-one (12) was prepared similarly to **15** from 4-(pyrrol-1-yl)acetophenone (0.12 g, 0.00065 mol) and pyrrole-2-carbaldehyde (0.06 g, 0.00065 mol). The product was chromatographed on silica gel (eluent acetone : hexane = 1 : 3). Yield 12%, mp 207–208°C. ¹H NMR spectrum (CDCl₃, δ, ppm, *J*, Hz): 6.34 m (1H, HNC<u>CH</u>-pyrrole), 6.39 t (2H, pyrrole, *J* 2.25), 6.73 m (1H, CH-pyrrole), 7.00 m (1H, HN<u>CH</u>-pyrrole), 7.10– 7.15 d (1H, CH=<u>CH</u>–CO, *J* 15.3), 7.18 t (2H, pyrrole, *J* 2.25), 7.48 d (2H, Ph, *J* 9.0), 7.71–7.76 d (1H, <u>CH</u>=CH– CO, *J* 15.3), 8.06 d (2H, Ph, *J* 9.0), 8.82 s (1H, NH). UV spectrum (CH₂Cl₂), λ_{max} 321.9, 374.8 nm. Mass spectrum, *m/z* (*I*, %): 262.1 [M⁺] (100.0), (C₁₇H₁₄N₂OS, *M*_{calc} 262.3).

2-Amino-4-R₁-6-R₂-pyrimidines 3–5 and 8 (general procedure). A mixture of 0.025 mol of appropriate 1-R₁-3-R₂-prop-2-en-1-one, 4.10 g (0.019 mol) of guanidine sulfate, and 23 ml of 50% aqueous KOH solution in 60 ml of ethanol was refluxed with stirring for 1 h, after which 9 ml of a 33% H₂O₂ solution was added dropwise under the same conditions over a period of 1 h. The hot reaction mixture was poured into ice-cold water, and the precipitate that formed was filtered off, dried, and chromatographed on silica gel (eluent CH₂Cl₂ or acetone : hexane = 1 : 2).

2-Amino-4-[4-(pyrrol-1-yl)phenyl]-6-(2-thienyl) pyrimidine (3). Yield 34%, mp 221–223°C. ¹H NMR spectrum (CDCl₃, δ , ppm, *J*, Hz): 5.14 br.s (2H, NH₂), 6.38 t (2H, pyrrole, *J* 2.25), 7.14 t (1H, CH-Th, *J* 3.9), 7.16 t (2H, pyrrole, *J* 2.1), 7.36 s (1H, pyrimidine), 7.48 d (1H, SC<u>CH</u>-Th, *J* 5.1), 7.50 d (2H, Ph, *J* 8.7), 7.78 d (1H, SCH-Th, *J* 3.6), 8.11 d (2H, Ph, *J* 9.0). Mass spectrum, *m*/*z* (*I*, %): 318.10 [M⁺] (100.0), (C₁₈H₁₄N₄S, *M*_{calc} 318.40). UV spectrum (CH₂Cl₂), λ _{max} 246.0, 301.9, 357.9 nm.

2-Amino-4-(4-aminophenyl)-6-(2-thienyl)

pyrimidine (4). Yield 34%, mp 221–223°C. ¹H NMR spectrum (CDCl₃, δ , ppm, *J*, Hz): 5.05 br.s (4H, 2NH₂), 6.75 d (2H, Ph, *J* 8.7), 7.13 t (1H, CH-Th, *J* 4.5), 7.28 s (1H, pyrimidine), 7.45 d (1H, SC<u>CH</u>-Th, *J* 5.1), 7.74 d (1H, SCH-Th, *J* 3.6), 7.90 d (2H, Ph, *J* 8.4). Mass spectrum, *m/z* (*I*, %): 268.05 [M⁺] (100.0), (C₁₄H₁₂N₄S, *M*_{calc} 268.34). UV spectrum (CH₂Cl₂), λ_{max} 235.0, 291.9, 343.9, 354.9 nm.

2-(Pyrrol-1-yl)-4-[4-(pyrrol-1-yl)phenyl]-6-(2thienyl)pyrimidine (5). Yield 19%, mp 155–157°C. ¹H NMR spectrum (CDCl₃, δ , ppm, *J*, Hz): 6.37 t (2H, pyrrole, *J* 2.25), 6.40 t (2H, pyrrole, *J* 2.1), 7.18–7.21 m (2H, SC<u>CH</u>-Th, CH-Th), 7.53 d (2H, Ph, *J* 8.7), 7.56 t (2H, pyrrole, *J* 2.4), 7.69 s (1H, pyrimidine), 7.89 d (1H, SCH-Th, *J* 3.6), 7.95 t (2H, pyrrole, *J* 2.4), 8.25 d (2H, Ph, *J* 8.4). Mass spectrum, *m*/*z* (*I*, %): 368.05 [M⁺] (100.0), (C₂₂H₁₆N₄S, *M*_{calc} 368.46). UV spectrum (CH₂Cl₂), λ_{max} 256.0, 302.9, 336.9, 357.9 nm.

2-Amino-4-(4-aminophenyl)-6-(3,4-ethylenedioxythiophen-2-yl)pyrimidine (8). Yield 13%, mp 227–230°C. ¹H NMR spectrum (CDCl₃, δ , ppm, *J*, Hz): 4.28 m (2H, OCH₂), 4.41 m (2H, OCH₂), 5.01 s (4H, 2NH₂), 6.05 s (1H, Th), 6.50 s (1H, pyrimidine), 6.61 d (2H, Ph, *J* 9.0), 6.73 d (2H, Ph, *J* 8.7). UV spectrum (CH₂Cl₂), λ_{max} 246.0, 283.9, 349.9 nm.

2-(Pyrrol-1-yl)-4-[4-(pyrrol-1-yl)phenyl]-6-(3,4ethylenedioxythiophen-2-yl)pyrimidine (9). Yield 18%, mp 46–48°C. ¹H NMR spectrum (CDCl₃, δ , ppm, *J*, Hz): 4.30 m (2H, OCH₂), 4.46 m (2H, OCH₂), 6.35 t (2H, pyrrole, *J* 2.4), 6.40 t (2H, pyrrole, *J* 2.25), 6.60 s (1H, Th), 7.18 t (2H, pyrrole, *J* 2.1), 7.53 d (2H, Ph, *J* 8.7), 7.93 t (2H, pyrrole, *J* 2.4), 8.01 s (1H, pyrimidine), 8.25 d (2H, Ph, *J* 8.7). UV spectrum (CH₂Cl₂), λ_{max} 259.9, 302.9, 353.9, 368.2 nm.

4-(Pyrrol-1-yl)acetophenone (11). A mixture of 0.68 g (0.005 mol) of 4-aminoacetophenone and 0.66 g (0.005 mol) of 2,5-dimethoxytetrahydrofuran was refluxed in a minimal amount of glacial CH₃COOH for 1 h. After cooling, the mixture was poured into cold water and extracted with CH₂Cl₂ (3×75 ml). The organic layers were combined, washed with water, saturated aqueous NaHCO₃ solution, and again water and dried over Na₂SO₄. The solvent was evaporated, and the dark residue was chromatographed on silica gel (eluent CH₂Cl₂). Yield 50%, mp 118–120°C. ¹H NMR spectrum (CDCl₃, δ , ppm, *J*, Hz): 2.61 s (3H, CH₃), 6.38 t (2H, pyrrole, *J* 2.25), 7.16 t (2H, pyrrole, *J* 2.1), 7.45 d (2H, Ph, *J* 9.3), 8.02 d (2H, Ph, *J* 9.0).

CONCLUSION

Previously unknown pyrimidines and pyrroles containing both donor and acceptor heterocycles in the conjugation chain were synthesized. Films of a series of conjugated polymers on the surface of a glassy carbon electrode were prepared, and their redox stability was studied.

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