Electrochimica Acta 59 (2012) 567–572

Contents lists available at SciVerse ScienceDirect

Electrochimica Acta

journal homepage: www.elsevier.com/locate/electacta

Electrochemistry and spectroelectrochemistry of a novel selenophene-based monomer

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ARTICLE INFO

Article history:
Received 8 July 2011
Received in revised form 3 November 2011
Accepted 4 November 2011
Available online 15 November 2011

Keywords:
Synthesis
Properties
Spectroelectrochemistry
X-ray analysis

ABSTRACT

A stereocontrolled synthesis of a novel selenophene-based monomer namely (*E*,*E*)-1,4-dimethoxy-2,5-bis[2-(selenophen-2-yl)ethenyl]benzene has been successfully performed starting from 1,4-dimethoxybenzene. Its spectra, electrochemistry and polymerization have been studied.

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1. Introduction

In recent years there has been a rapid development of studies related to the production of new materials for electrochromic devices [1], electronic papers, sensors, radio frequency identification tags [2], polymeric light emitting diodes, organic photovoltaic devices [3,4], and organic single crystal field-effect transistors [5]. An important group of compounds that may have potential application are oligomers [6,7] or polymers of thiophene and their derivatives [8]. Oligomers of thiophene are well known to possess good electrodonating properties [9], considerable mobility of charges and substantial crystallinity [10]. Surprisingly, up to 2005 only little attention has been given to polymers of selenophene and its derivatives even though selenophene has lower oxidation potential in compare to its sulfur or oxygen analogs. In the last few years a number of papers on polymers bearing selenophene have grown rapidly [11–17] and first electrochromic device where a selenophene-containing polymer was used as an active layer was described in 2009 [18]. Few years ago, regioregular poly(3-hexylselenophene) (P3HS) [19,20] have been reported for organic electronic applications, with higher mobility than analog of polythiophenes (P3HT) [21,22] Poly(9,90-n-dioctylfluorene-alt-biselenophene) (F8Se2) [23] and poly(5,50-bis(3-dodecylthiophene-2-yl)2-20-biselenophene) (PDT2Se2) [24] revealed an order of magnitude larger field-effect mobility than their sulfur analogs, when used as active layers in OFETs. For this reason, in recent years an increasing interest in oligomers, polymers and copolymers containing selenophene moiety has been observed. Of the many compounds synthesized and studied may be mentioned: 1,3-diarylbenzo[c]selenophenes [25], benzo[1,2-b:4,5-b']diselenophenes [26], selenopheno[3,2-b]thiophene [27] or 9,9-dialkylfluorene capped benzo[c]thiophene/benzo[c]selenophene derivatives [28]. Attracted much interest: poly(3,4-ethylenedioxyselenophene) (PEDOS; PEDOT analog) [29–31], poly(fluorene-co-selenophene) [13] and poly(9,9′-dioctylfluorene-alt-biselenophene) [21]. Very recent review on poly(selenophene) and its derivatives was done by Patra and Bendikov [32]. Selenophene derivatives have a lower oxidation potentials than the thiophenes but higher than tellurophenes analogs in series Te < Se < S [33,34]. In comparison with tellurophene analogs selenophene derivatives are more stable [35,36].

Formerly, it was shown that monomers with conjugated system of multiple bonds bearing two terminal residues of chalcogens (furan or thiophene) with free positions in C5 (Fig. 1, structure **1**) are able to electropolymerize leading to linear PPV-type conducting systems that show strong electroluminescence [37–40]. Now we present a synthesis and some properties

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doi:10.1016/j.electacta.2011.11.021

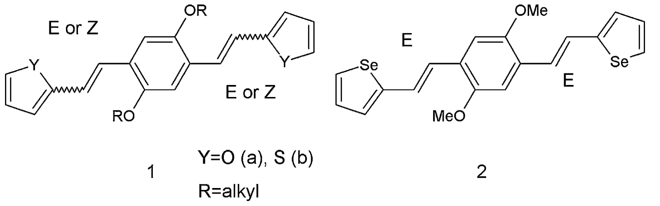


Fig. 1. Structures of monomers with conjugated system of multiple bonds and two terminal residues of furan (**1a**), thiophene (**1b**) or selenophene (**2**).

of compound **2** being (EE) selenophene analog of compounds **1**.

2. Experimental

2.1. Synthesis

2.1.1. General

All solvents were dried and then distilled before the use. Other commercial substances and reagents were used without purification. Melting points (not corrected) were determined on Boetius HMK apparatus. NMR spectra were taken in $CDCl_3$ with TMS as an internal reference by Varian XL-300 at 300 MHz for ^1H and at 75.5 MHz for ^{13}C. EA results were obtained using Perkin-Elmer CHN automatic analyzer. X-ray structural data have been obtained on the Gemini A Ultra diffractometer. The structures were determined by means of direct methods and refined by the full-matrix least squares technique.

2.1.2. Preparation details

*2.1.2.1. (E,E)-1,4-dimethoxy-2,5-bis[2-(selenophen-2-yl)ethenyl]benzene (**2**).* This compound was synthesized following a slightly modified procedure known for the synthesis of (*E*,*E*)-2,5-bis(2,5-dihexylstyryl)-1,4-dimethoxybenzene [39,40]. Preparation procedures of intermediate materials (selenophene-2-carboxyaldehyde and tetraethyl [2,5-dimethoxy-1,4-bis(methylene)]diphosphonate) can be obtained on request from authors as an supplementary additional material.

A cold solution of selenophene-2-karboxyaldehyde (0.36 g, 2.28 mmol) and tetraethyl [2,5-dimethoxy-1,4-bis(methylene)]diphosphonate (0.50 g, 1.14 mmol) in dry DMF (5 mL) was added dropwise to a solution of *t*-BuOK (0.79 g, 7.05 mmol) in DMF (10 mL) at 0 °C. The mixture was stirred without cooling for 2.5 h and then poured into cold water (50 mL) acidified with conc. HCl (1 mL). The obtained solution was extracted with dichloromethane (3 × 25 mL) and organic extracts were dried over $MgSO_4$. The solvents were evaporated under diminished pressure and the residue purified by column chromatography over silica gel using hexane:benzene (4:1) as eluent to give (*E*,*E*)-1,4-dimethoxy-2,5-bis[2-(selenophen-2-yl)ethenyl]benzene (0.15 g, 0.34 mmol, 30%) as orange crystals (m.p. 224–226 °C). ^1H NMR δ (300 MHz, $CDCl_3$): 3.91 (6H, s, CH_3); 7.04 (2H, s, ArH), 7.13 (2H, d, $J = 15.9$ Hz, CH vinyl); 7.20–7.24 (4H, m, C_4H_3Se, C^4H, C^3H); 7.33 (2H, d, $J = 15.9$ Hz, CH vinyl); 7.83–7.85 (2H, m, C_4H_3Se, C^5H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 56.42 ($O\underline{C}H_3$); 109.38 (Ar, CH); 124.51 (C vinyl); 124.96 (C vinyl); 126.28 ($Ar\underline{C}CH$); 128.84 (C_4H_3Se, C^5); 129.00 (C_4H_3Se, C^4); 130.27 (C_4H_3Se, C^3); 150.00 (C_4H_3Se, C^2); 151.59 (Ar, $\underline{C}OAlk$). UV (CH_2Cl_2): $\lambda = 414.5$ nm; $\varepsilon = (4.66 \pm 0.29) \times 10^4$; $\lambda = 355.0$ nm; $\varepsilon = (1.77 \pm 0.12) \times 10^4$; $\lambda = 293.0$ nm; $\varepsilon = (0.82 \pm 0.07) \times 10^4$ [dm^3/mol cm].

The structure of product was additionally proved by X-ray measurements of a monocrystal of (*E*,*E*)-1,4-dimethoxy-2,5-bis[2-(selenophen-2-yl)ethenyl]benzene (**2**). Crystallographic data (excluding structure factors) for the structure of **2** has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC 812456. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 01223 336033 or e-mail: deposit@ccdc.cam.ac.Uk).

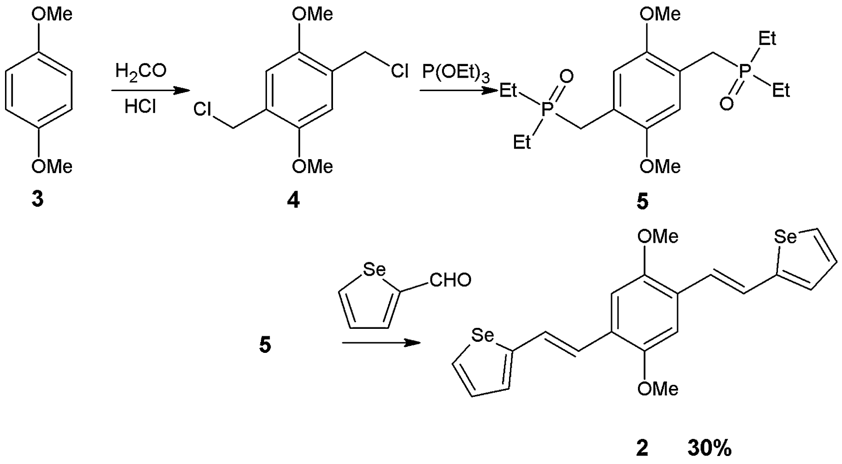
2.2. Electrochemical measurement

The solutions of compound **2** with the concentration of 1.0 mM were used for cyclic voltammperommetry measurements. Electrochemical studies were conducted in 0.1 M solution of Bu_4NBF_4 (Sigma Aldrich 99%) in anhydrous dichloromethane at room temperature. The electrochemical investigations were carried out using Eco Chemie Company's AUTOLAB potentiostat "PGSTAT20". The results were collected using GPES (General Purpose Electrochemical System) software. The electrochemical cell comprised of platinum wire with 1 mm diameter of working area as working electrode, Ag wire – calibrated versus ferrocene/ferrocinium redox couple – as a quasi-reference electrode and platinum coil as auxiliary electrode. Cyclic voltammperometry measurements were conducted at 50 mV/s potential rate.

3. Results and discussion

A stereocontrolled three steps synthesis of a novel selenophene-based monomer namely (*E*,*E*)-1,4-dimethoxy-2,5-bis[2-(selenophen-2-yl)ethenyl]benzene (**2**) has been successfully performed starting from 1,4-dimethoxybenzene (**3**) as it is shown in Scheme 1. Compound 3 was treated with paraformaldehyde and gaseous hydrogen chloride in the solution of glacial acetic acid and concentrated hydrochloric acid to give 2,5-bis(chloromethyl)-1,4-dimethoxybenzene (**4**). The latter compound on heating at with ca 20% excess of triethoxyphosphine 150 °C afforded 2,5-bis(diethylphosphononomethyl)-1,4-dimethoxybenzene as a white powder in 30% yield.

During our former work on preparation of several compounds of type **1**, we treated bis(chloromethyl)-1,4-dialkoxybenzenes with triphenylphosphine (instead of triethoxyphosphine used here); the respective substitution products obtained in form of salts, were then condensed with appropriate 2-chalcogenecarbaldehydes. Such method in all cases lead to mixtures of stereoisomers differing in configuration of substituents at the double bonds. Separation of stereoisomers was always troublesome [37–41]. After all, ZZ isomers were rather unstable and in solutions quickly isomerized to give equilibrium mixtures. All separated steroisomers underwent electropolymerization showing two oxidation peaks. For the stereoisomers, first oxidation peaks appeared at different potentials decreasing in the following order ZZ, EZ, EE. Only small differences in oxidation potentials could be spotted for the second peaks what might suggest isomerization prior to oxidation. Polymers obtained from the stereoisomers did not show distinct differences what seemed to support the former suggestion [39]. Therefore, a stereocontrolled condensation of **5** with 2-selenophenecarbaldehyde leading to the most stable EE isomer was a method of choice for the synthesis of **2** even if yields achieved would be lower in comparison with reaction using triphenylphosphine derivatives. Indeed, the reaction afforded the expected EE isomer as the only product. Its structure was proved by ^1H NMR spectrum analysis (two doublets 2H $J = 15.9$ Hz at 7.13 and 7.33 corresponding to two pairs of vinyl groups of E configuration). This finding was confirmed by X-ray crystallography of **2** monocrystal (crystal dimensions 0.38 mm × 0.27 mm × 0.12 mm). 3058 reflections were collected using Cu Kα radiation within 3 h 13 min at 100 K. Program used to solve structure was: SHELXS97 (Sheldrick [42]); program used to refine structure: SHELXL97 (Sheldrick [43]). ORTEP projection is shown in Fig. 2. The shape of molecules of



Scheme 1. A stereocontrolled three steps synthesis of (*E*,*E*)-1,4-dimethoxy-2,5-bis[2-(selenophen-2-yl)ethenyl]benzene (**2**).

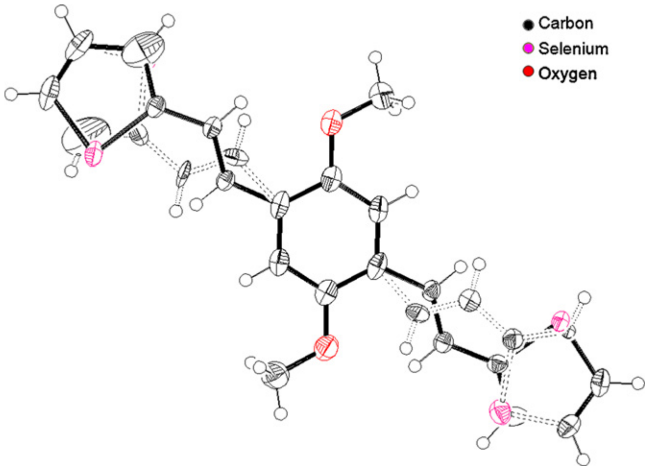


Fig. 2. Molecular structure of (*E*,*E*)-1,4-dimethoxy-2,5-bis[2-(selenophen-2-yl)-ethenyl]benzene (**2**) showing 50% probability displacement ellipsoids obtained by ORTEP 3. A dominant conformation in the crystal is indicated by thicker lines.

monomer **2** allows relatively efficient crystal packing. The packing mode is shown in Fig. 3 in a projection along the α-axis.

Refinement of F^2 was calculated against all reflections. The weighted *R*-factor *wR* and goodness of fit *S* were based on F^2, conventional factors *R* were based on *F*, with *F* set to zero for negative F^2. The following results were obtained: final *R* indices [>2sigma(*I*)], $R_1 = 0.0449$, $wR_2 = 0.1168$ and final *R* indices (all data), $R_1 = 0.0476$, $wR_2 = 0.1211$.

For the sample, Mo provided better data than Cu in terms of R_1 [>2sigma(*I*)]. R_1 (all data) was similar in both cases while the intensity of the Cu data was vastly greater than that for Mo, with a value for *I*/sigma of 60 compared to 25. The ellipsoids for the Mo data set were generally less well behaved than for Cu.

The selenophene rings in the crystal of monomer **2** (Fig. 3) are disordered over two orientations. Taking into account the extent of disorder in this crystallographic structure of **2**, a final R_1 of 4.49% is an excellent result. Similar phenomena disorders in ring orientations were detected e.g., in structures of all 1,4-dialkoxy-2,5-bis[2-(thiophene-2-yl)ethenyl]benzenes studied by X-ray crystallography and thus can be consider as typical for compounds of such type [37,41,44].

Synthesized compound **2** was oxidized during the multielectron process shown in Fig. 4, with not clearly distinguishable individual peaks, but coalescing ones to give a first maximum at 0.46 V and the next, very broad at 0.95–1.05 V. During the reduction the increase of current is observed in potential range 0.5–0.7 V indicating that the process of catalytic polymerization showing a prominent peak for the reduction of the forming polymer, showing up at the potential

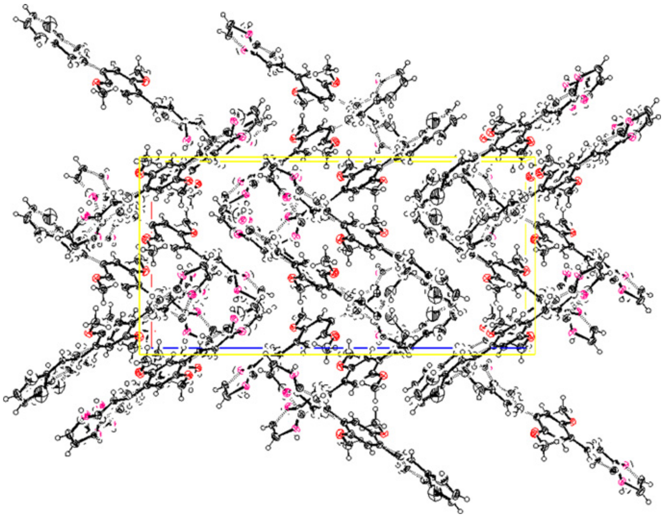


Fig. 3. Crystal packing, shown in a projection along the crystallographic α-axis.

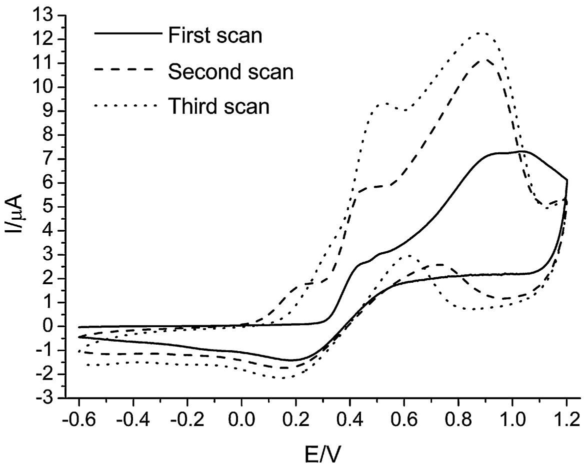


Fig. 4. Cyclic voltammograms of electropolymerization of compound **2** in wide oxidation range. Measure conditions: scan rate 50 mV/s, Ag wire – calibrated versus ferrocene/ferrocinium redox couple as a quasi-reference electrode.

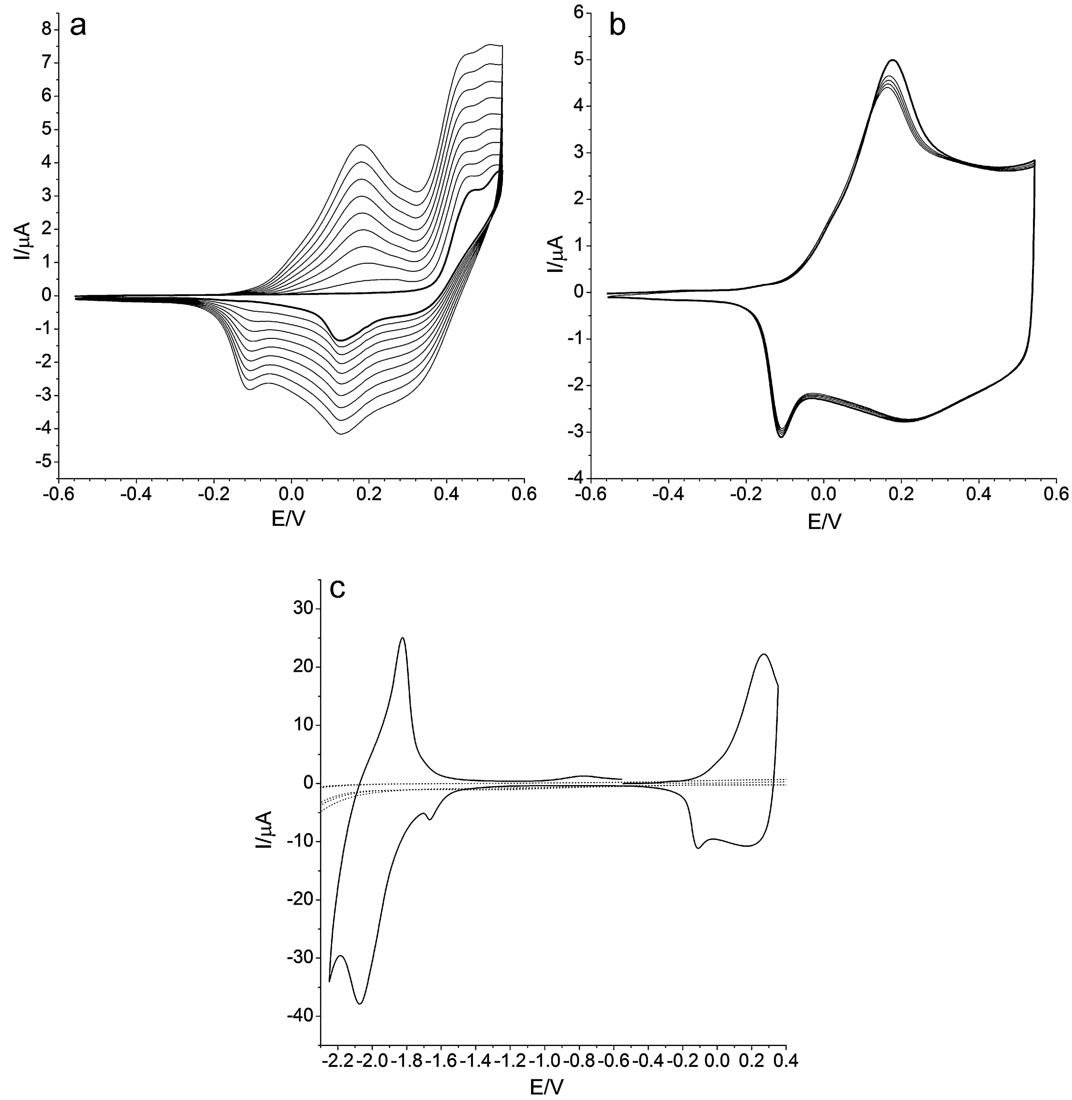


Fig. 5. Cyclic voltammograms of electropolymerization (a) of **2**, doping–dedoping process of obtained polymeric film in monomer free medium (b) and redox process of polymeric film in monomer free medium (c). Measure conditions: scan rate 50 mV/s, Ag wire – calibrated versus ferrocene/ferrocinium redox couple as a quasi-reference electrode.

of 0.2 V. The next two scans shows the oxidation of the polymer at potentials of 0.2 V prior to monomer oxidation peak, height of which clearly increases confirming the autocatalytic effect of the polymer formed on the electrode.

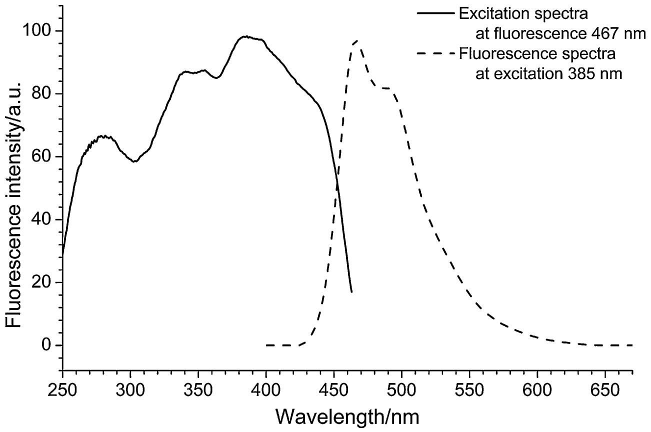


Fig. 6. A comparison of monomer **2** fluorescence spectra and excitation spectra.

The best properties of the polymer were obtained by limiting the potential of polymerization to approximately 0.6 V thus slightly higher than the oxidation potential of monomer (**2**), as shown in Fig. 5a. A regular increase of the polymer layer was observed when the oxidation potential was 0.2 V and the reduction one equal to 0.18 V. The sharp reduction peak at the potential of −0.11 V is responsible for a violent change of conductivity of the polymer at the transition from the conductive phase to nonconductive one.

By transferring the electrode with deposited polymer layer into an electrolyte solution without the monomer, voltammetric curve shown in Fig. 5b was obtained. It is evident that the shape of the polymer curve very well matches those obtained during the polymerization what may suggest practically quantitative polymerization of monomer **2** with only very small amounts of possible by-products. The rapid decrease in current after return of potential

Table 1
Electrochemical and optical band-gap.

	Homo (eV)	Lumo (eV)	Eg El. (eV)	Eg Op. (eV)
Poly(**1b**)	−5.26	−3.33	1.93	1.92
Poly(**2**)	−4.79	−3.09	1.70	1.67

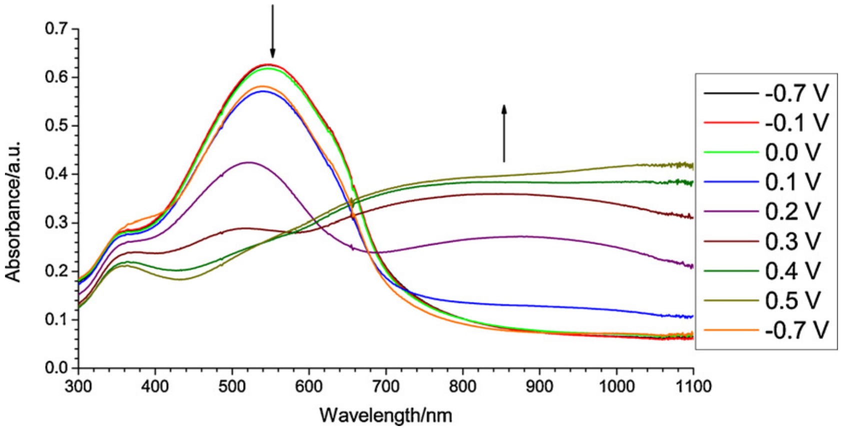


Fig. 7. UV–Vis spectra recorded during electrochemical oxidation of poly(**2**) film.

at 0.6 V demonstrates that the resulting polymer is characterized by high electrical conductivity. Next voltammetric cycles run at the same practical curves showing high electrochemical stability of the polymers at the investigated range of potentials. In addition to the oxidation, monomer **2** can also be reduced, as shown in Fig. 5c, yielding reversible redox system located at the potential ca. −2.0 V. This allowed us to determine so called electrochemical band gap, which is equal to 1.70 eV. Comparing the results obtained here with data for thiophene analog one can see that the energy gap width for the thiophene polymer is higher amounting to 1.93 eV.

Fluorescence spectra of monomer **2** registered in dichloromethane solution are illustrated in Fig. 6. These observations are consistent with the absorption spectroscopy data. The monomers exhibited a strong peak at 467 and 492 nm when excited by 385 (Fig. 6).

In order to find relation between redox processes and UV–Vis absorption the spectroelectrochemical properties of polymer of **2** prepared on ITO coated glass electrode was investigated. The UV–Vis set of spectrum recorded during the polymer oxidation revealed the absorption band (400–700 nm) correlated with the polymer (Fig. 7), gradually losing its intensity as the applied potential is increased. In the same time, the new defined absorption band occurred (700–1100 nm Fig. 7), owing the formation of radical cation (polaron). Formation of other charge carriers (probably bipolaron band) occurs with absorption band formation from 950 nm to beyond 1100 nm. Under oxidation polymers layer from gold color turns to dark violet.

Thank to electrochemical characterization of polymers by analyzing oxidation and reduction processes it is possible to estimation of electrochemical band-gap value. In correlation to the molecular band, oxidation peak is related to the HOMO band and reduction peak is related to the LUMO band. Electrochemical band-gap value is the range between those peaks (Table 1). The lowest electrochemical and optical band-gap have polymer containing biselenophene unit.

4. Conclusions

Presented results show that (*E*,*E*)-1,4-dimethoxy-2,5-bis[2-(selenophen-2-yl)ethenyl]benzene (monomer **2**) can be obtained by condensation of 2-selenophenecarbaldehyde with tetraethyl [2,5-dimethoxy-1,4-bis(methylene)]-diphosphonate as the solely isolated isomer. Its structure as EE isomer can be proved both by the presence of two doublets at 7.13 and 7.33 (with $J = 15.9$ Hz) corresponding to two pairs of E vinyl protons groups in analysis of ^{1}H NMR spectrum and by analysis of X-ray measurements results of a monocrystal of **2**.

Monomer **2** readily undergoes electropolymerization to afford product of excellent stability. Both monomer **2** and its polymer exhibit photoluminescent properties what could enable them to qualify for the group of materials of the potential applications in optoelectronics.

Acknowledgments

X-ray measurements was performed courtesy of Oliver Presly, Ph.D., Program Marketing Manager, XRD; Oxford Diffraction Ltd. – *now Agilent Technologies*.

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