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Electrochemical and spectral properties of meta-linked 1,3,5-tris(aryl)benzenes and 2,4,6-tris(aryl)-1-phenoles, and their polymers

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ABSTRACT

We present electrochemical and spectral properties of symmetric monomers 1,3,5-tris(aryl)benzenes and 2,4,6-tris(aryl)-1-phenols and their polymers. These compounds contain thienyl, furyl or EDOT moieties attached to central benzene or phenol ring at the meta-position, synthesized by a Stille cross-coupling procedure. All monomers are electroactive and undergo electropolymerization creating thin films on an electrode surface. Polymers with meta-linkages were obtained by electrochemical oxidation. Detailed cyclic voltammetry and in situ UV–vis spectroelectrochemistry show that polymers with hydroxy groups exhibit higher conductivity and better stability than with benzene core. Interesting and different behavior occurs for 2,4,6-tris(2-thienyl)-1-phenol, for which the characteristic, sharp, redox peak is observed.

1. Introduction

Blue light-emitting materials have been the subject of extensive studies in recent years. The band gap of the materials can be tuned by attaching substituents to a polymer backbone or introducing the design of π-conjugated linkages which include nonconjugated carbon bridges. For example the presence of meta-linkage units along polymer chain effectively interrupts π-conjugation [1]. M-phenylenes units reveal great linearity of optical properties, inversion of conjugation length, and enhancement of photoluminescence efficiency [2]. Blue light-emitting materials can be synthesised on the base of this polymers.

It has previously been reported in the literature, that branched oligothiophenes and oligothienylbenzenes act as monomers in cross-linked semiconducting polymers [3–5], and as components of conjugated dendrimers [6–10]. This type of polymers can be characterized by a three-dimensional structure, which is on the sake of their conductivity [11]. Moreover dendrite structures exhibit interesting properties like energy transfer and photochemical antenna function [12].

Herein, we describe a convenient and simple method for creating conjugated polymers, namely poly(arylbenzene)s, based on thiophene, furan and EDOT linkages. Aside from a typical application of polymers for high surface area materials, such as gas storage or catalyst support, the conjugated polymers may find new applications in the field of organic electronics or optoelectronic compounds. The conjugated polymers in general, and polythiophenes in particular, have found widespread use in organic–electronic devices, such as organic light-emitting diodes (OLEDs) [13], organic field-effect transistors (OFETs) [14], organic solar cells, and photovoltaic cells [15,16].

Symmetrical molecules can be used as central cores for various oligomers and to decrease the length of the step synthesis. *C*3-symmetric compounds based on a di- and tri-thienylbenzene core and symmetrical conjugated oligo(phenylene ethynylene)s are useful in the design of organic electroluminescent devices. Suzuki, Stille, Kumada, and Negishi coupling reactions are well established [17,18]. In this paper, we present a series of various *C*3-symmetric compounds which were synthesized by Stille cross-coupling procedure. The obtained compounds are characterized by a high yield (84–92%).

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We performed electrochemical and spectroscopic studies. We focused on oxidative polymerization of the monomers and thoroughly elucidated optical and electrochemical properties of polymers in dependence of thiophene, EDOT, and furan moieties. It is also worthy to mention, that oligomers containing hydroxyl groups provide both a good stability and a high conductivity to the film.

2. Experimental

2.1. Synthesis of monomers

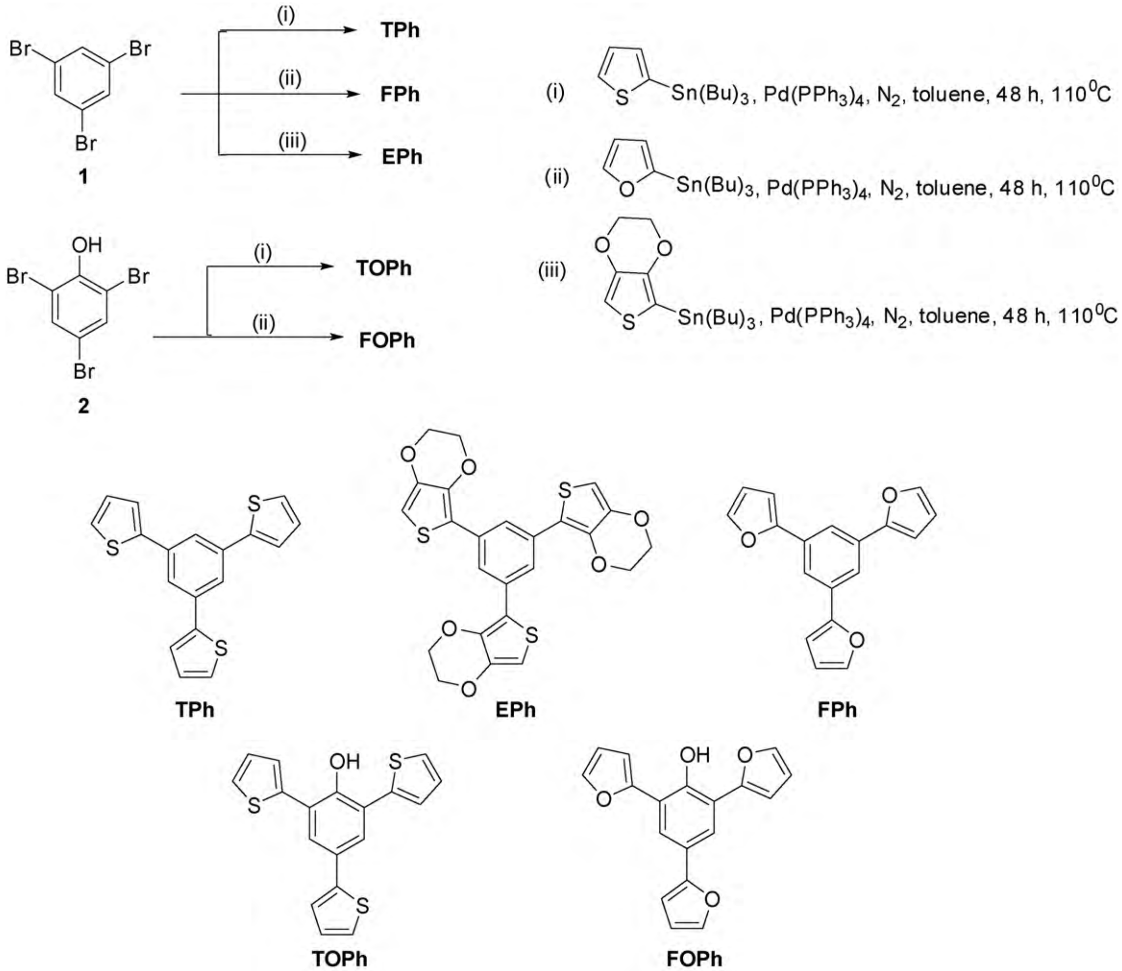
All chemicals, reagents and solvents are commercially available and were used without further purification, except toluene, which was distilled over sodium/benzophenone. 2-(Tributylstannyl)-3,4-ethylenedioxythiophene was synthesized according to a previously reported method [19]. The structures have been characterized by ^{1}H NMR and ^{13}C NMR in one of our previous papers [20].

2.2. General procedure for the preparation of 1,3,5-tris(aryl)benzene (TPh, FPh, EPh) and 2,4,6-tris(aryl)-1-phenol (TOPh, FOPh)

To 1,3,5-tribrombenzene (**1**) (2.0 mmol) or 2,4,6-tribrom-1-phenol (**2**) (2.0 mmol) in a 250 mL round two-bottom flask under nitrogen into an anhydrous toluene (150 mL) was added 2-(tributylstannyl)arene (6.6 mmol) and Pd(PPh$_3$)$_4$ (0.460 g, 0.4 mmol). The resulting mixture was stirred for 48 h in 110 °C. After this time the mixture was cooled down to the room temperature. Water was added and the resulting solution was extracted with 3 × 50 mL portions of CHCl$_3$. The combined organic layers were washed with 50 mL of brine, dried over MgSO$_4$ and evaporated to until a dark brown oil appeared. The crude product was purified by a column chromatography (silica gel, hexane/AcOEt, 10:1). The palladium-catalyzed Stille cross-coupling reaction of compound **1** or **2** with 2-(tributylstannyl)arylenes gave the desired products: 1,3,5-tris(2-thienyl)-benzen (TPh), 1,3,5-tris(2-furyl)-benzene (FPh), 1,3,5-tris[2-(3,4-ethylenedioxythienyl)]-benzene (EPh), 2,4,6-tris(2-thienyl)-1-phenol (TOPh), 2,4,6-tris(2-furyl)-1-phenol (FOPh) (Scheme 1) with an yield of 84–92%. All the monomers were obtained as white, green, yellow, and brown crystals and characterized by spectroscopic methods. All spectral data confirm their molecular structures.

2.3. Electrochemistry

Electrosynthesis and studies on polymer films were performed on Ecochemie AUTOLAB potentiostat–galvanostat model PGSTAT20. The results were analyzed using GPES software (General Purpose Electrochemical System). Cyclic voltammetry (CV) was used for electrochemical measurements. In order to perform all experiments three-electrode arrangement was employed. Polymer films were synthesized on the platinum wire at a scan rate 50 mV/s. An Ag pseudo-reference electrode was used and its exact potential



Scheme 1. Synthesis of monomers: (a) 1,3,5-tris(2-thienyl)-benzene (TPh), (b) 1,3,5-tris(2-furyl)-benzene (FPh), (c) 1,3,5-tris[2-(3,4-ethylenedioxythienyl)]-benzene (EPh), (d) 2,4,6-tris(2-thienyl)-1-phenol (TOPh), (e) 2,4,6-tris(2-furyl)-1-phenol (FOPh).

was measured versus ferrocene. Platinum wire served as a counter electrode.

Spectroelectrochemical measurements were carried out using a UV–vis spectrophotometer Hewlett Packard 8452A. The target polymer was synthesized on the indium–tin-oxide (ITO) coated quartz electrode, acting as a 'working electrode'. UV–vis spectra of polymer films were recorded during p-doping. ITO potential was scaled versus ferrocenu before film polymerization.

All experiments were performed in acetonitrile (POCH 99.8%) or dichloromethane (POCH 99.8%), in presence of 0.1 M tetrabutylammonium tetrafluoroborate (Aldrich) as an inert electrolyte.

3. Results and discussion

3.1. Cyclic voltammetry

All monomers under potentiodynamic conditions were electrochemically active. Fig. 1 shows oxidation of monomers in CH_3CN. Different processes during oxidation were recorded in their dependence on both the terminal arm moieties and presence of hydroxyl group attached to benzene ring. Monomers undergo irreversible oxidation at least in two step reaction. Cyclic voltammetry curves of 1,3,5-tris(2-furyl)-benzene (FPh) are considerably different from 2,4,6-tris(2-furyl)-1-phenol (FOPh). Hydroxyl group decreases the first oxidation potential E_m^{ox} at about 0.56 V, nevertheless we observed at least one more oxidation peak. The same effect occurs during oxidation of 1,3,5-tris(2-thienyl)-benzene (TPh) and 2,4,6-tris(2-thienyl)-1-phenol (TOPh), a similar shift in oxidation potentials is visible, as in the case of furyl derivatives. Among monomers with benzene core 1,3,5-tris[2-(3,4-ethylenedioxythienyl)]-benzene, the (EPh) monomer exhibits the lowest E_m^{ox} of approximately 0.81 V, while for monomer with thienyl $E_m^{ox} = 1.02$ V, and with furyl groups the highest $E_m^{ox} = 1.13$ V is observed. Similar situation occurs for monomers with phenol core. The values of E_m^{ox} for TOPh and FOPh amount to $E_m^{ox} = 0.55$ V, and $E_m^{ox} = 0.57$ V, respectively.

All monomers were irreversibly oxidized already in the first oxidation step. In the case of FPh cyclic voltammetry (Fig. 2a) yellow film was formed on the electrode surface at the beginning of first oxidation peak at 0.91 V, with the highest oxidation peak in the first scan. The peak decreases during all next scans. However, the reduction peak is not observed. It indicates the formation of a poor conductive polymer on the electrode surface. In the case of FOPh (Fig. 2b) a different behavior is observed. Namely, the first 10 scans reveal an increase of the peak of interest in every subsequent scan,

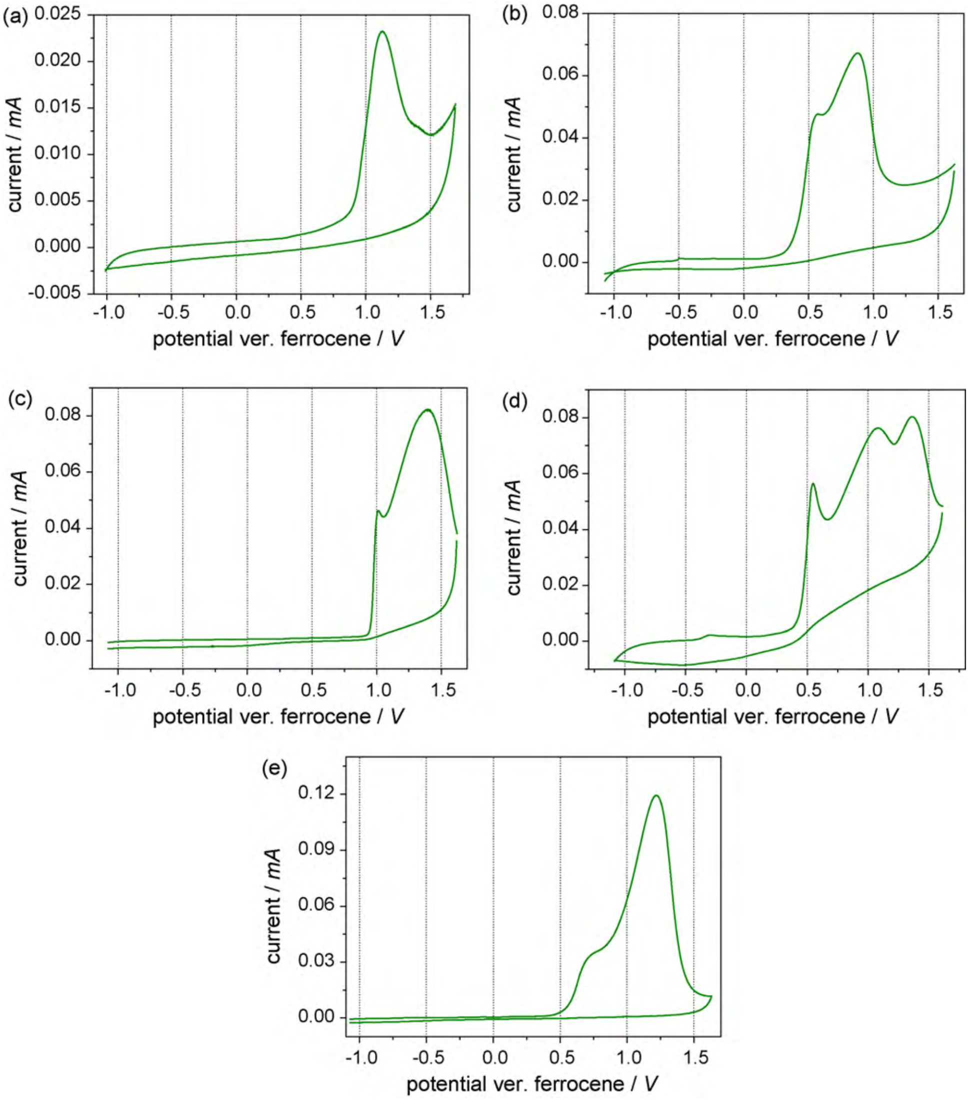


Fig. 1. Cyclic voltammograms obtained in wide range of potentials on Pt electrode; potential sweep rate $\nu = 50$ mV/s; 1 mM monomer solutions in 0.1 M Bu_4NBF_4 in CH_3CN: (a) FPh, (b) FOPh, (c) TPh, (d) TOPh, (e) EPh.

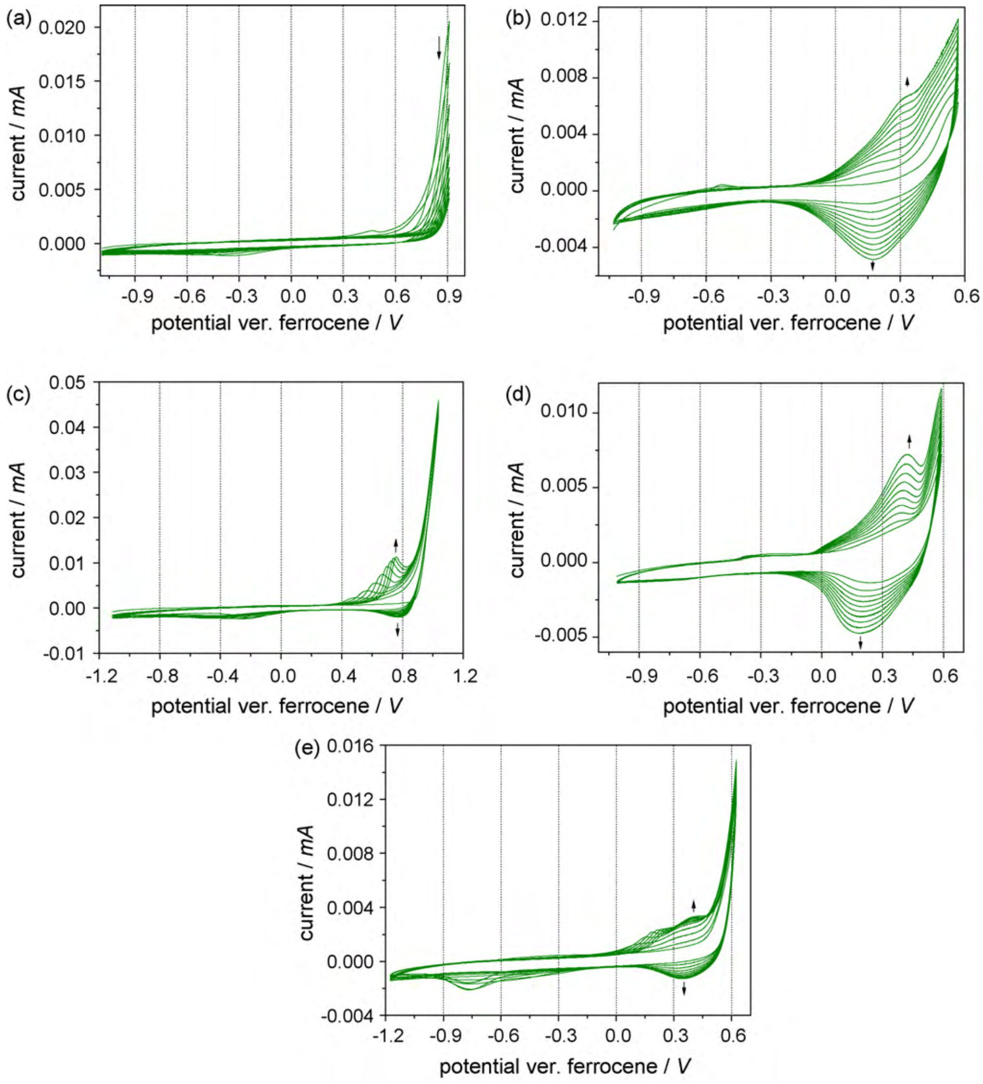


Fig. 2. Cyclic voltammograms obtained during polymerization on Pt electrode; potential sweep rate $\nu = 50$ mV/s; 1 mM monomer solutions in 0.1 M Bu_4NBF_4 in CH_2Cl_2: (a) FPh, (b) FOPh, (c) TPh, (d) TOPh, (e) EPh.

while the oxidation onset appears at the value of the oxidation potential of −0.15 V. Hydroxyl substituent considerably changes the monomer behavior during electropolymerization.

The oxidation of TPh (Fig. 2c) results in covering of the working electrode with a light yellow film. After the second scan a new peak appears at 0.49 V, which reveals a subsequent shift towards a higher potential according to the number of scans, achieving at the stage of the 10th scan the value of 0.76 V. During the whole experiment the value of the current has to be stabilized due to a poor conductivity of a polymer film. The formation of a black film is observed during polymerization of TOPh (Fig. 2d). A new oxidation peak appears during subsequent scans at values of the potential being in the range of 0.36–0.42 V. Oxidation onset and reduction peak appear at the oxidation potential values of −0.07 V and 0.18 V, respectively. An increased value of the current even after 100 scans indicates much better conductivity of a film being formed, than it takes place for TPh. In case of TPh the largest increase of the current occurs after the first scan, while afterwards this effect is strongly hampered. Replacement of central benzene ring by phenol results not only in decreasing the oxidation potential but also considerable strengthens the film conductivity of copolymers with furan or thiophene moieties. The result of CV performed on EPh is slightly different from the previously described monomers (Fig. 2e). In the second scan a peak with maximum at approximately 0.34 V and in the third scan an additional peak at 0.07 V appear. In every subsequent scan the peak undergoes a shift towards the higher potential. During the reverse cycles we observe a peak at 0.35 V due to dedoping of polymers. The peak at −0.76 V originates from deprotonation of σ-dimers. However, one could observe peaks also for FPh at −0.32 V and TPh at −0.34 V. It was impossible to obtain stable polymers at the second peak, owing to their degradation during polymerization in this potential range. According to the well known electropolymerization mechanism, the coupling between thiophene, furan or EDOT moieties in 5-position was expected [21].

There are some differences in the increase of both polymer films and oxidation potentials, depending on the used solvent (Fig. 3). Oxidation potentials of monomers in CH_3CN are lower than in CH_2Cl_2 (Table 1). Only in the case of FPh (Fig. 3a) the situation is different, CH_2Cl_2 lowers E_m^{ox} at about 0.08 V. However, the shape of voltammetric curves and compound behavior during polymerization reveal the similar tendency. The second scan of FOPh in CH_3CN (Fig. 3b) introduce a new peak (the oxidation onset at −0.19 V), which increases with following scans, but the clear maximum is not observed. Polymerization of TPh in CH_3CN (Fig. 3c) reveals both a less intense increase of the current, and a smaller shift towards higher potential values according to subsequent scans. The con-

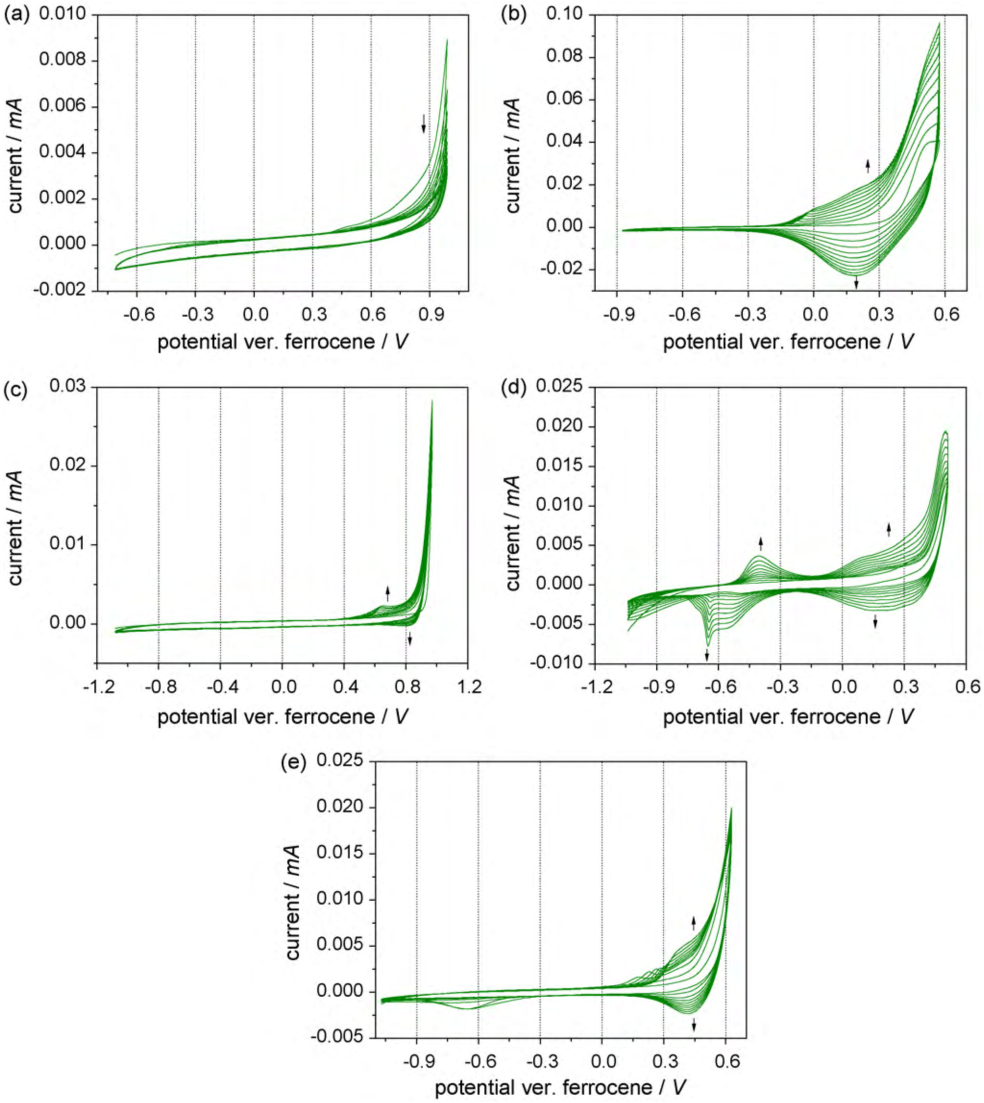


Fig. 3. Cyclic voltammograms obtained during polymerization on Pt electrode; potential sweep rate $v = 50$ mV/s; 1 mM monomer solutions in 0.1 M Bu_4NBF_4 in CH_3CN: (a) FPh, (b) FOPh, (c) TPh, (d) TOPh, (e) EPh.

cerned range edges of 0.52 V and 0.65 V are related to the second and the 10th scan, respectively. In the case of poly(TOPh), the influence of solvent polarity used in electropolymerization is of the main interest (Fig. 3d). Regarding CH_3CN, in the second scan an additional redox system appears at the oxidation potential of −0.40 V simultaneously with the corresponding reduction peak at −0.65 V. Moreover, the monomer oxidation potential peak shifts towards lower values of the potential according to the number of scans starting from the value of 0.55 V and achieving 0.50 V in the 10th scan. This indicates the formation of a very well conducting layer. This feature facilitates efficiently the oxidation of monomers. In the case of EPh we observe very similar behavior in both solvents (Fig. 3e).

Fig. 4 illustrates all cyclic voltammetry curves for obtained polymers. The anodic cycle of poly(FPh) in CH_2Cl_2 reveals a not clear oxidation peak (Fig. 4a), positioned close to the monomer oxidation potential. However, this peak disappears after only few scans. This confirms, that the poly(FPh) film has poor stability and conductivity, and a low molecular mass. The cycling of the poly(FOPh) film performed in the polymerization potential range in a monomer free electrolyte solution reveals an oxidation peak located at about 0.35 V (oxidation onset at 0 V) and a reduction peak at 0.22 V (Fig. 4b). A sharp drop in the current value after changing polarization direction indicates the formation of the conducting layer. In addition, the obtained polymer is quite stable, as evidenced by recording repetitive voltammetric waves during multiple doping and de-doping processes. A similar influence of hydroxyl substituent occurs in the case of thiophene copolymers. The polymer of TPh (Fig. 4c) has oxidation potential of 0.87 V, comparing to the monomer is smaller by 0.23 V, what indicates short conjugation length. Moreover, poly(TPh) is middle stable. In the first anodic cycle of poly(TOPh) (Fig. 4d) there is an oxidation peak located at the

Table 1
Oxidation potential values of monomers and corresponding polymers in different solvents, where: E_m^{ox} is the monomer oxidation potential; E_p^{ox} is the polymer oxidation potential.

Compound	Solvent			
	CH_3CN E_m^{ox} [V]	CH_2Cl_2 E_m^{ox} [V]	CH_3CN E_p^{ox} [V]	CH_2Cl_2 E_p^{ox} [V]
TPh	1.02	1.10	0.69	0.87
FPh	1.13	1.05	[a]	[a]
EPh	0.81	0.89	0.51	0.43
TOPh	0.55	0.66	−0.41[b]	−0.41[b]
FOPh	0.57	0.66	0.25	0.40

[a] Ill-defined peak.
[b] Potential value determined from the second scan.

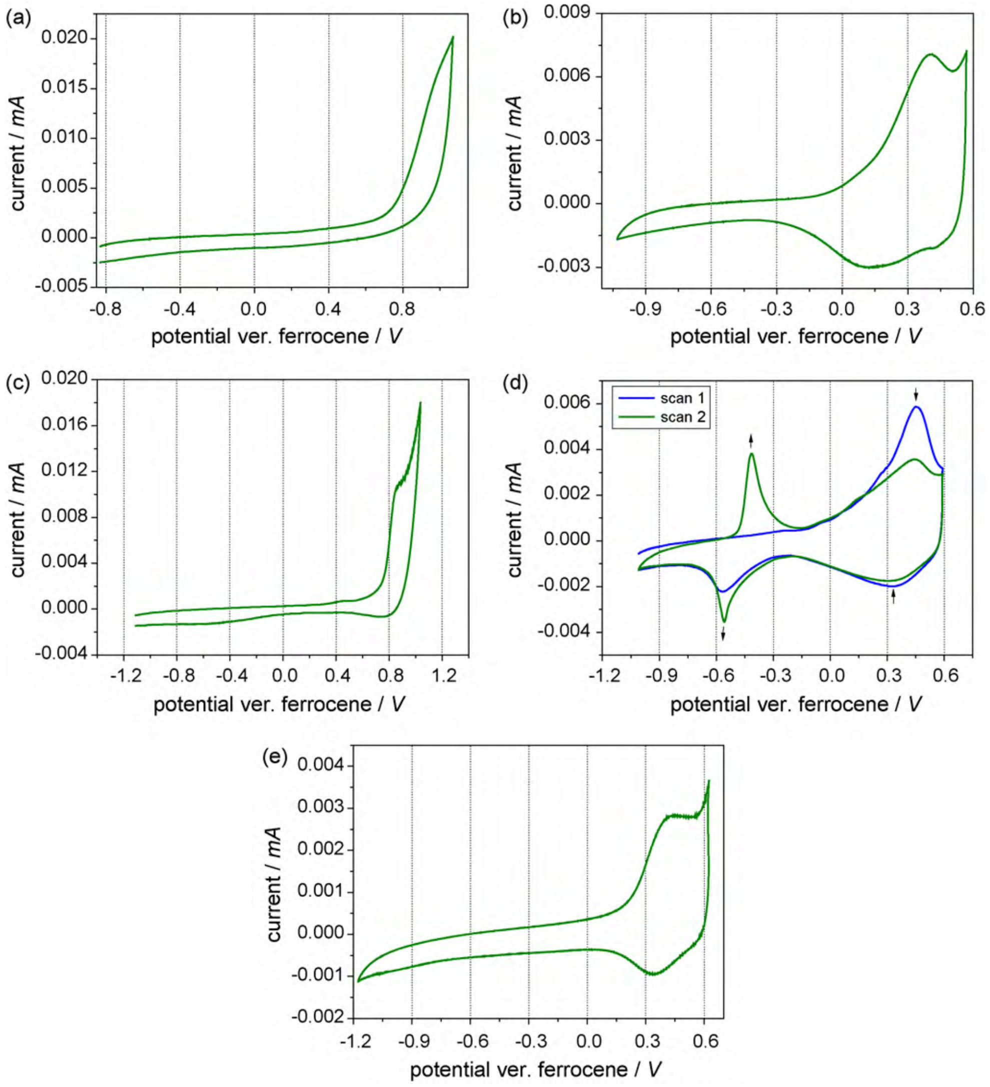
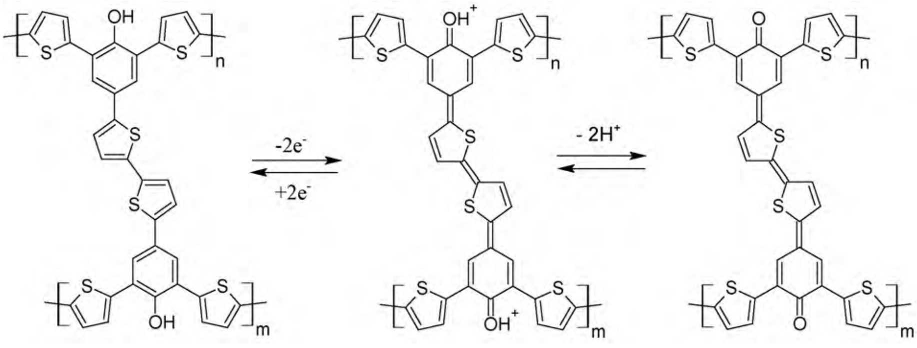


Fig. 4. Cyclic voltammograms performed on thin polymer films on Pt electrode; potential sweep rate $\nu = 50$ mV/s; in 0.1 M Bu_4NBF_4 in CH_2Cl_2: (a) poly(FPh), (b) poly(FOPh), (c) poly(TPh), (d) poly(TOPh), (e) poly(EPh).

potential 0.46 V, while in subsequent scans the additional oxidation peak appears at the potential of −0.41 V. However, the total charge consumed during the oxidation process remains almost the same. The first sharp and reversible spike in the cyclic voltammogram after which the current decreases, at the polymer poor conductivity in range −0.3 V to 0.0 V, might indicate a phase transformation [22] or pure redox reaction between particular hydroquinone–quinoine like states [23]. A small insertion of strong acid leads to the disappearance of the redox system that may confirm an appearance of the structure, which is shown in Scheme 2. The second, broad polymer oxidation peak indicates a variety of chain lengths. Clarification of this process comprises the subject for further studies. The poly(TOPh) film is much more stable during multiple doping and de-doping processes, than the poly(TPh) one. This confirms,



Scheme 2. Proposed mechanism of the redox reaction during oxidation of poly(TOPh) in the range from −0.9 V to −0.3 V.

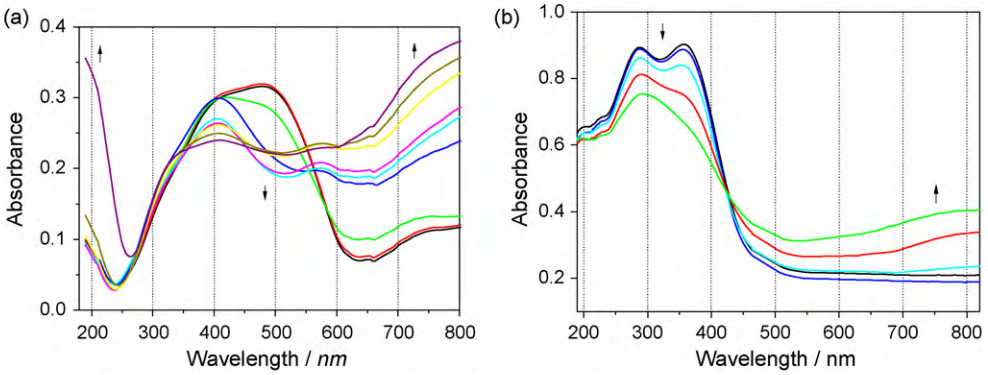


Fig. 5. UV–vis spectra recorded during oxidation of polymer films on ITO electrode in 0.1 M Bu_4NBF_4 solution in CH_3CN of: a) poly(TOPh), potentials from −0.9 V to 0.7 V in intervals of 0.2 V; (b) poly(FOPh), potentials from −0.3 V to 0.5 V in intervals of 0.2 V.

that a phenol core improves the electric properties of the polymers and their stability, when comparing to a benzene core. In the case of poly(EPh) (Fig. 4e) we observe the oxidation onset at 0.14 V, and the maximum at 0.43 V. After switching of the scanning direction, a sharp drop in the current and the minimum at 0.34 V are observed.

3.2. UV–vis spectroscopy

Fig. 5a shows the spectrum of poly(TOPh) recorded at different values of potential representing various oxidation levels. However, one can conclude from the spectrum, the total film de-doping after synthesis is not visible at values of the potential of −0.9 V and −0.7 V. Poly(TOPh) in the neutral state absorbs the light in a broad range, and has at least two absorption maxima located at $\lambda_{max1} = 401$ nm and $\lambda_{max2} = 484$ nm. This may indicate the presence of at least two polymer fractions with different effective conjugation lengths. At the higher values of the potential, which correspond to the characteristic sharp voltammetric peak, both the decrease in the absorption maximum, and formation of a new band in a higher wavelength range are observed. This confirms the formation of oxidation products, probably polarons. A further increase of the electrode potential up to a value of the second oxidation peak is accompanied by a change in the shape of the spectrum with the decay at 400 nm. This originates from the products with the shortest conjugation length, and an increase in polaron or bipolaron band intensity.

UV–vis spectra of poly(FOPh) are presented in Fig. 5b. The polymer in its neutral state has two absorption maxima at $\lambda_{max1} = 288$ nm and $\lambda_{max2} = 363$ nm. The analysis of the spectra during oxidation reveals a decrease in the absorption π–π* transition band, and an increase in the oxidation product absorption. Moreover, a stable isosbestic point is observed at 423 nm.

Following empirical studies, the absorption maxima of the thienyl substituted benzenes change proportionally to the conjugation length. One can find in the literature [24,25], that the number of conjugated double bonds (n) of oligothiophenebenzenes is estimated by Eq. (1), where E_{max} (eV) is the energy at the absorption maximum.

$$n = \frac{7.32}{E_{max} - 2.26} \tag{1}$$

Substituting the values of E_{max} for TPh ($E_{max} = 4.16$ eV) and TOPh ($E_{max} = 4.11$ eV) monomers, the calculated values of n are equal to 3.95 and 3.85, respectively. Eq. (1), when applied to the polymers, gives the values of $n = 4.11$ and $n = 24.24$ for poly(TPh) and poly(TOPh). The analysis of these results leads to the conclusion, that there is a lack of coupling among the thiophene arms, and a negligible conjugation between the benzene rings of TPh, TOPh monomers and poly(TPh). However, the behavior of poly(TOPh) is quite different. Large number of conjugated bonds indicate, that hydroxyl groups enable coupling among the thiophene arms at the meta-position. On the other hand, the quinoine structure of the connector between two polymer arms, which is shown in Scheme 3 assures that there is much better conjugation than meta-substitution in benzene. The analysis of Fig. 6a and b confirms the theoretical calculations. While the spectra of monomers TPh and TOPh are similar, the spectrum of poly(TOPh) differs significantly from that of poly(TPh).

Fig. 7 illustrates, that the poly(EPh) film has two absorption maxima at 330 nm and 492 nm. The first one is related to the light

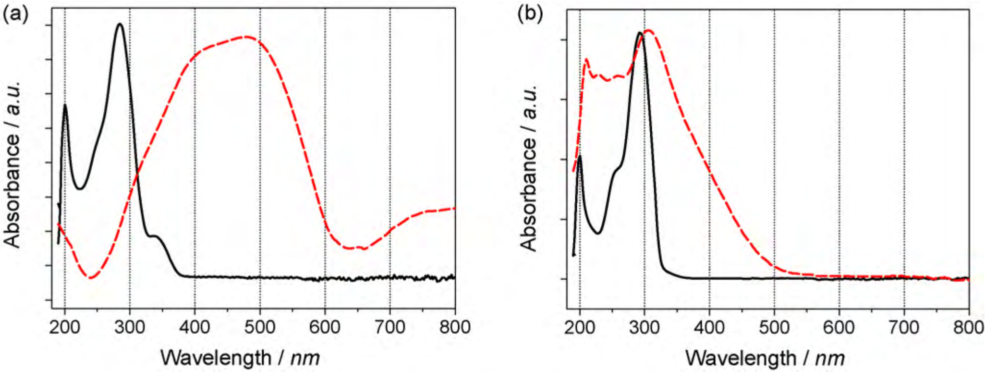
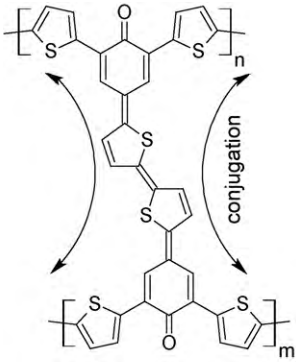


Fig. 6. UV-Vis spectra of monomers in CH_3CN and polymer films of: a) (—) TOPh (— —) poly(TOPh); b) (—) TPh (— —) poly(TPh). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)



Scheme 3. Proposed conjugation paths in poly(TOPh) in its oxidized state at approximately −0.3 V.

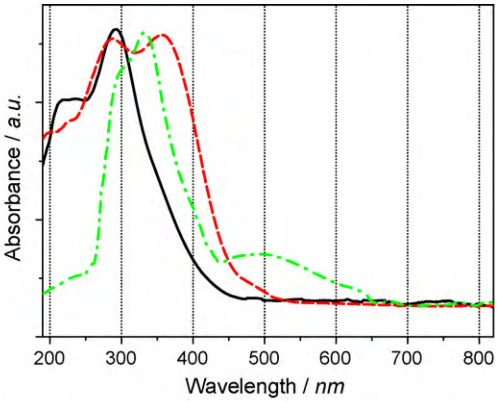


Fig. 7. UV-Vis spectra performed on polymer films: (—) poly(FPh), (— —) poly(FOPh) and (— • —) poly(EPh). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

absorption by a polymer in its neutral state, while the second one arises from its oxidized form. In fact, the oxidized form appears because polymer does not undergo entirely the de-doping process. In the case of poly(FPh) spectra one maximum at 291 nm is present, while for poly(FOPh) two maxima at 288 nm and 363 nm are observed. The energy gap values amount to 3.34 eV, 2.8 eV and 2.74 eV for poly(FPh), poly(TPh) and poly(EPh), respectively. Taking under consideration the polymers with hydroxyl groups, the value of E_g is lowered significantly achieving 2.73 eV and 2.00 eV for poly(FOPh) and poly(TOPh), respectively. This may be attributed to the strong influence of a hydroxyl substituent on the aryl arms, not only by decreasing the oxidation potential, but also by increasing the effective conjugation length between the aryl arms. This coupling is present only in polymers and suggests that the conjugation itself can expand beyond the hydroxyl groups through thiophene or furan moieties.

4. Conclusion

All monomers containing thienyl, furyl and EDOT groups were successfully synthesized and characterized by spectroscopic methods. The cyclic voltammetry method was successfully applied, because all of target compounds are electroactive and undergo electropolymerization creating thin films directly on the surface of electrodes.

Generally, the application of the meta-linkages is rarely reported. However, in some cases meta-substitution is desired, mainly due to the better control of the energy gap (E_g). We obtained poly[1,3,5-tris(aryl)benzene]s with relatively large E_g, which is required for blue light-emitting materials. In addition, following the data set resulting from optical and electrochemical measurements, we conclude, that oligomers containing EDOT groups are much more stable and probably also better conducting, than thienyl, or furyl analogues. The hydroxyl substituent has a significant influence on the electrochemical and spectroelectrochemical properties of oligoarylbenzenes. In the whole class of these compounds the decrease of oxidation potential and energy gap is observed. On the other hand, this substituent enables coupling among particular aryl arms, which are connected at the meta-position. Thus, this leads to the improvement of the polymer overall stability and significant enhancement of polymer conductivity. The overall analysis from point of view of chemistry, electrochemistry supported by spectroscopic methods confirms, that the compounds described in this paper could be considered as a material for the whole class of organic–electronic devices.

Acknowledgments

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