

A short review on the strategy towards development of π -conjugated polymers with highly reversible p- and n-doping[☆]

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Abstract

In order to increase the n-doping ability and ensure the high stability of the reduced forms of π -conjugated polymers, we compare two alternative concepts for designing such polymers, based on polythiophene. The first is based on the introduction of an electron-withdrawing group in the 3-position of the thiophene ring, whereas the second approach involves the synthesis of subunits that possess high electron-withdrawing abilities and polymers comprising bi- or quatro-thiophene subunits in the polymeric backbone. Among the four polymeric films reported in this paper, exceptionally good results were obtained with polymethyl octyl thiophene oxadiazole (PMOThOD) film electrodes, which are composed of quatro-thiophene and oxadiazole subunits. The remarkable redox activity at low potentials of this polymer and the absence of charge trapping, as well as the charge trapping phenomena occurring with the other polymeric systems studied, are explained and discussed in light of both the cyclic voltammetric and electrochemical impedance spectroscopic studies of these systems. The conclusions reached are supported by modeling the impedance of the films' electrodes, which are subjected to charge trapping, either because of geometric or energetic non-homogeneities.

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1. Introduction

It is well known that π -conjugated polymeric films or coatings may undergo oxidation and reduction from their electrically neutral, non-conductive state, with the formation of electrochromic, highly conductive p- and n-doped states [1,2]. The process of p- and n-doping can occur easily in an electrochemical cell in suitable supporting electrolyte solutions (in most cases in aprotic organic solvents), using different electro-analytical techniques such as stationary or pulsed potentiostatic or galvanostatic modes, potentiodynamic measurements, cyclic voltammetry, etc. The doping reactions result in the formation of mobile electronic carriers in the polymer chains (either positively or negatively charged upon p- and n-doping, respectively), which are compensated electrostatically by the insertion of the anions or cations, respectively [1,2]. Mixed cation–anion insertions, complicated by the insertion of electrically neu-

tral solvent molecules, often take place [3,4]. These insertion processes affect considerably the stability of the emerging doped states of the polymer. However, it appears that even a more important impact on the stability is due to (i) the nature of the parasitic electrochemical processes of the supporting electrolyte solutions, and (ii) the intrinsic resistance of the doped polymeric states to overoxidation and overreduction, due to the features of their electronic structure. Overoxidation and overreduction processes can be defined as partially reversible (e.g., deactivation due to some charge trapping in the material bulk), or a completely irreversible process (e.g., degradation).

N-doping of π -conjugated polymers is known to suffer from the insufficient stability of the resulting reduced forms of the polymer, more than that in the case of p-doping [5]. In order to overcome this principal difficulty, we have elaborated on a strategy of using new, specially designed co-monomers, whose electron-accepting ability is finely tuned to the redox-behavior of the main π -conjugated polymer [6,7]. In this paper, we briefly summarize our efforts in this direction. We show that at least one of the mixed polymers, containing both quatro-thiophene and oxadiazole subunits, shows exceptional stability with respect

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to overreduction [6], greatly beyond the common stability of a variety of previously studied polymers [8–10].

2. Experimental

The electropolymerization procedure of pristine thiophene and 3-(3,4,5-trifluorophenyl)thiophene on Pt wires was previously reported in [11] and [12], respectively. As both monomers are soluble in acetonitrile (AN), this solvent was chosen for electropolymerization. Two other monomers, namely, 2,7-bis(4-octylthien-2-yl)-fluoren-9-one and dimethyldioctylquatrothiophene-*alt*-oxadiazole, are not soluble in AN, so the former was electropolymerized in CH_2Cl_2 [7], whereas the latter was electropolymerized in a mixture of CH_2Cl_2 and AN [6]. Since the polymers are deposited on the electrode in the p-doped state, their reduction to the neutral state must be carried out consecutively before studying their n-doping activity.

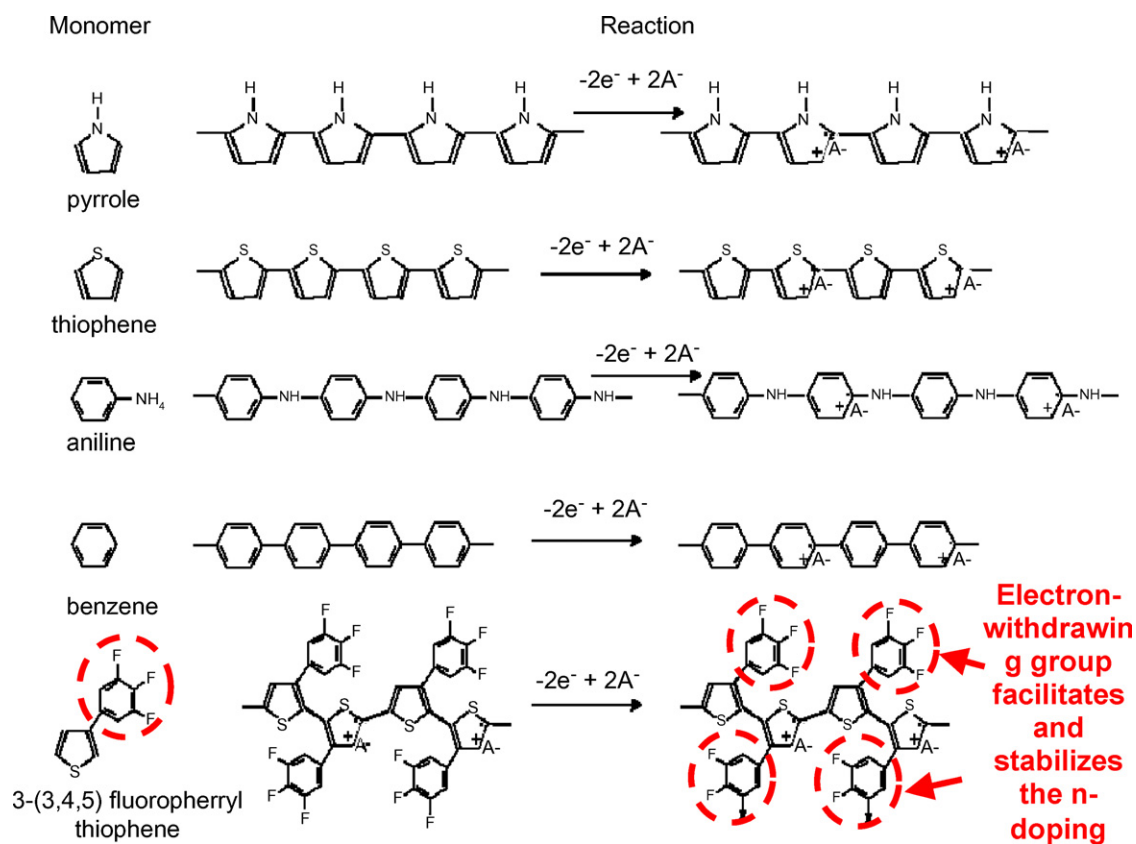
In the next step, the polymer deposited on the electrode was thoroughly washed with a pure solvent, dried, and transferred to another 3-electrode cell for cycling in a 0.25 M $\text{TEABF}_4/\text{sulfolane}$ solution. The sulfolane-based electrolytes were shown to be superior to other solutions, in that they ensure the stable and highly reversible doping of π -conjugated polymers, in particular in the n-doping domains (acetonitrile solutions were shown to be inferior in this respect [12,13]). The

working electrodes were Pt wires fused into glass tubes in the cells, and the WE were symmetrically surrounded by cylindrical Pt-foil counter-electrodes. We used $\text{Ag}/0.1 \text{ M AgNO}_3$ in AN reference electrodes, separated from the sulfolane solution by thin frit membranes. From time to time, the potential of these electrodes was compared with the standard potential of a Fc/Fc^+ couple (which appeared to be $-0.035 \text{ V vs. Ag}/\text{Ag}^+$). All the potentials quoted in this work refer to $\text{Ag}/\text{Ag}^+ 0.1 \text{ M RE}$. Cell assembly and electrochemical measurements were performed at room temperature under highly purified Ar atmosphere in glove boxes. The oxygen and water content did not exceed 2 ppm. Residual water in the dry solutions never exceeded 20 ppm, measured by Karl Fischer titration.

Cyclic voltammetry (CV) and electrochemical impedance (EIS) measurements were carried out using a computerized potentiostat–galvanostat Model 20 Autolab controlled by the GPES and FRA Version 4.9 Eco Chemie B.V. Software (Utrecht, The Netherlands).

3. Results and discussion

Scheme 1 summarizes the structural formulae of underivatized heterocyclic monomers (of important π -conjugated polymers) whose anodic oxidation leads to grafting the surface of current collectors with thin electronically conductive polymeric films. The high electronic conductivity of these films is due



Scheme 1. Typical simple (non-derivatized) monomers, whose anodic oxidations in aprotic solutions containing suitable supporting electrolytes result in the formation of doped, electronically conductive polymeric films, as indicated. Derivatization of the thiophene unit by a 3-(3,4,5-fluorophenyl) electron-withdrawing group is expected to stabilize and facilitate the n-doping reaction of the resulting polymer film.

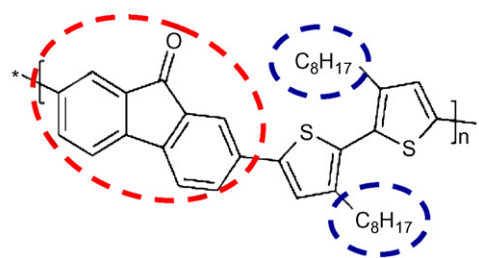
to the formation of positively charged mobile carriers accompanied by the concomitant insertion of anions from the supporting electrolyte solution, and very often of the solvent molecules. Two of the four conducting polymers presented in Scheme 1, namely, polypyrrole and polyaniline, cannot be n-doped since they have a relatively low standard potential for p-doping, so, in accordance with their relatively large bandwidths, their possible n-doping process is expected to occur at much lower potentials than those allowed by the electrochemical window of most (if not all) of the common polar aprotic electrolyte solutions relevant to such systems. The other two polymers, polythiophene (PTh) and polyparaphenylene, with relatively high oxidation potentials, can be n-doped, although the maximal possible charge (i.e., the practical redox capacity) is usually considerably lower than that for the corresponding p-doping processes (of the same polymers).

In order to improve the n-doping ability of PTh, the introduction of an electron-withdrawing group was suggested, e.g., (3,4,5-fluorophenyl), in the 3 position of the heterocycle [12,14–16] (see the bottom of Scheme 1), which was believed to facilitate and improve the n-dopability of the resulting polymeric film. This is because the above substitution in the heterocyclic ring may facilitate the onset of the polymer reduction (i.e., the start of the n-doping).

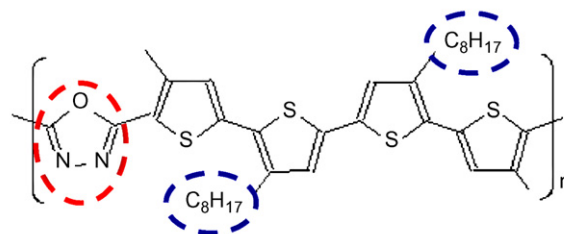
In recent years, in order to increase the efficiency and lifetime of organic polymer light-emitting devices (PLEDs) [17,18], an alternative concept, namely, the introduction of units with a high electron affinity directly onto the polymeric backbone of the parent π -conjugated polymer was developed [19–21]. The advantage to this concept was that this could be done by the synthesis of new co-monomers, containing units of the parent polymer and with the increased electron affinity. The fluorenone, as well as the oxadiazole groups embedded in the substituted bi- or quarto-thiophene backbone (see the top and the bottom of Scheme 2, respectively), seemed to lead to the desirable goal of improving the n-dopability of the related polymers [7].

Fig. 1a–d show the stable CV responses of the 4 selected polymers (based on polythiophene) within the potential ranges of their p- and n-doping (the potential refers to the Ag/Ag⁺ reference electrode). These CVs demonstrate how the substitutions described above increase the capacity of the n-doping processes of polythiophene-based polymers. The unsubstituted PTh film electrode clearly shows the beginning of the n-doping process when approaching the potential of -2.2 V. However, the maximal n-doping charge is about 1/10 of the maximal charge of the p-doping process of the same polymer at the comparable span of the potential ranges. This ratio, as was previously shown [11], may slightly depend on the mode of the film's preparation and the nature of the solvent used.

The ratio of the n-to-p-doping charge improves dramatically when going from the PTh to the P3FPhTh film electrode (see Fig. 1b) [12,13]. Due to the presence of the electron-withdrawing group in the polymeric structure, the beginning of both p- and n-doping shifts considerably towards more positive potentials, compared to those for the PTh film electrode. For a similar potential span, the maximal n-doping charge is comparable with that of the charge of the p-doping process.



poly[(2,7-fluoren-9-one)-*alt*-(5,5'-(3,3'-di-*n*-octyl-2,2'-bithiophene) abbreviated as PFDOBTh



poly(dimethyldioctylquattrothiophene-*alt*-oxadiazole) abbreviated as PMOThOD

Scheme 2. Repeated units of new, conducting polymers with a highly p- and n-doped capacity based on bi- (a) or quatrothiophene building blocks, (b) together with highly electron-withdrawing groups such as 9-fluorenone (a) and oxadiazole (b) in the polymer backbone.

When the polymeric film contains both a 9-fluorenone group with a high electron affinity and bithiophene units, the n-doping process of such a polymer, namely, PFDOBTh, begins at about -1.5 V (i.e., at a potential similar to that for the P3FPhTh, see Fig. 1c), and proceeds in two clearly separate steps. The first reduction results in the localization of the emerging anion-radicals of the fluorenone units, followed by the formation of anion-radicals in the bithiophene units at a lower potential, comparable with that for the n-doping process of the unsubstituted PTh film electrodes [7]. However, despite reaching a substantial increase in the n-doping charge of PFDOBTh, it can be seen in Fig. 1c that the related p-doping process is not efficient when the anodic limit exceeds 1 V.

Finally, as seen in Fig. 1d, the combination of oxadiazole and quarto-thiophene units in the polymer backbone allows the attainment of the highest ever reported n-doping capacity for n-doped polymers ($1.8e^-$ per repeat unit [6]), which is even higher than the maximum p-doping capacity (1.1 and $1.3e^-$ per repeated unit measured in solutions based on sulfolane or imidazolium ionic liquid, respectively [6]). A remarkable feature of the doping characteristics of this polymer is that the potential span between the end point of the p- and n-doping is about 4.2 V, and that both the anodic and cathodic currents, when reaching these potentials in the CV curves in Fig. 1d, tend to diminish to zero. This means the withdrawal of all of the available π -electrons during the p-doping, and a complete filling of the polymer chains (saturation) with π -electrons upon polymer n-doping.

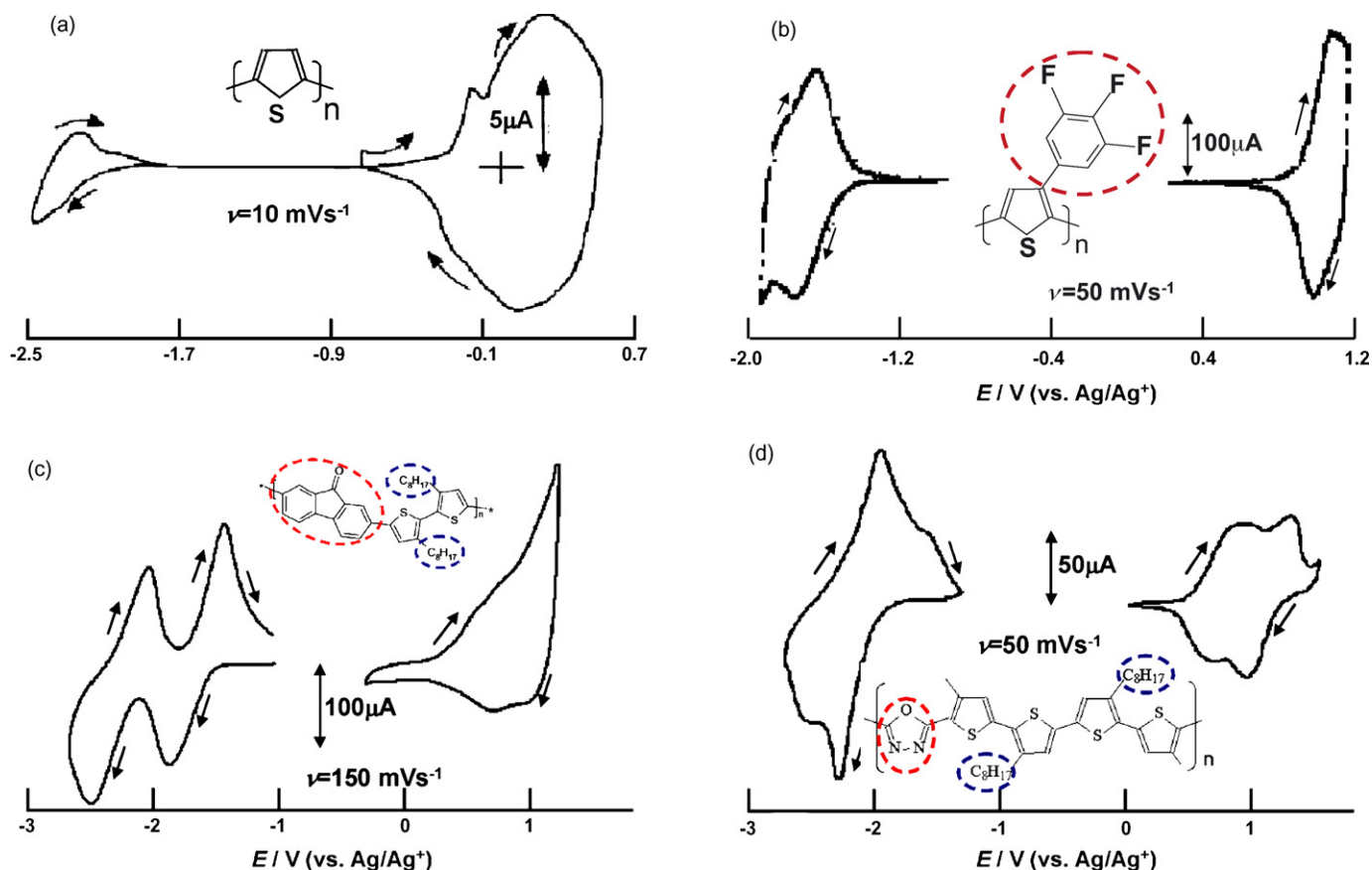


Fig. 1. Stable (steady-state) CV curves related to both p- and n-doping of four different polymers: (a) a simple polythiophene (PTh) film in 0.22 M TEABF₄/propylene carbonate solution; (b) A P3FPhTh film in 0.25 M TEABF₄/sulfolane solution; (c) PFDOBTh film 0.25 M TEABF₄/sulfolane solution; (d) PMOThOD in 0.15 M TBAPF₆/sulfolane solution. All potentials are referred to the same reference electrode (0.1 M Ag/AgNO₃ in acetonitrile separated from the electrolyte solution by a frit). The scan rates are indicated.

In Fig. 2a–d we address the important issue of the stability of the 4 selected polymeric films upon their consecutive cycling in the potential range typical to n-doping processes. The pristine PTh film electrode was cycled six times down to -2.50 , -2.55 and -2.60 V (Fig. 1a). It can be seen that during cycling the Faradaic efficiency of the n-doping process gradually improves, which can be assigned to a progressive trapping of the negatively charged electronic species in the film's bulk (see discussion below). Beyond the potential of -2.6 V, a rapid irreversible degradation of the PTh film was observed. Note also that the inserted cationic species belong almost exclusively to large tetraalkylammonium cations [11]. Even the negligible presence of the highly solvated, small size cations in a supporting electrolyte, such as Li⁺, results in the considerable trapping of the negatively charged electronic species (this can be classified as a kind of deactivation process, which is partially reversible, i.e., reactivation can be achieved after prolonged p-redoping in the same electrolyte solution) [11].

In contrast to the PTh films, the P3FPhTh film electrodes with the characteristic potential range of the n-doping shifted towards more positive values and show good Faradaic efficiency and very stable cycling (see Fig. 2b). Although the beginning of the n-doping process of the PFDOBTh film electrode (see Fig. 2c) is approximately the same as that for the P3FPhTh film,

its cut-off (vertex) potential shifts towards less positive values. As a result, the consecutive cycling of this electrode leads to a progressive charge trapping, and thus, the redox process of the moieties' low potentials (n-doping) shows a decrease in its redox-capacity related to both units (i.e., to the fluorenone and bithiophene) upon cycling.

As seen in Fig. 2d, the PMOThOD film shows very stable cycling when doped down to -2.7 V. This emphasizes the advantage of the combination of the fluorenone and the quaterthiophene units in the same polymeric backbone. Compared to the previous case of PFDOBTh, one can conclude that the value of the lowest vertex potential is not a reason in itself for the appearance of charge trapping and the diminution of the redox-capacity of the polymeric film. Most probably, the character of the initial electron-transfer and the accommodation of the electronic carriers of the more readily reduced subunit (i.e., on the fluorenone or oxadiazole) predetermines the subsequent facile reduction of the bi- or quaterthiophene units. The quaterthiophene co-unit is, in turn, more advantageous compared to that of the bithiophene co-unit, since it ensures a lower charge density and a more effective delocalization of the π -electron density in the conjugated system. In addition, the reduced oxadiazole unit probably has a better conjugation with the main quaterthiophene co-unit, and hence, a higher electronic con-

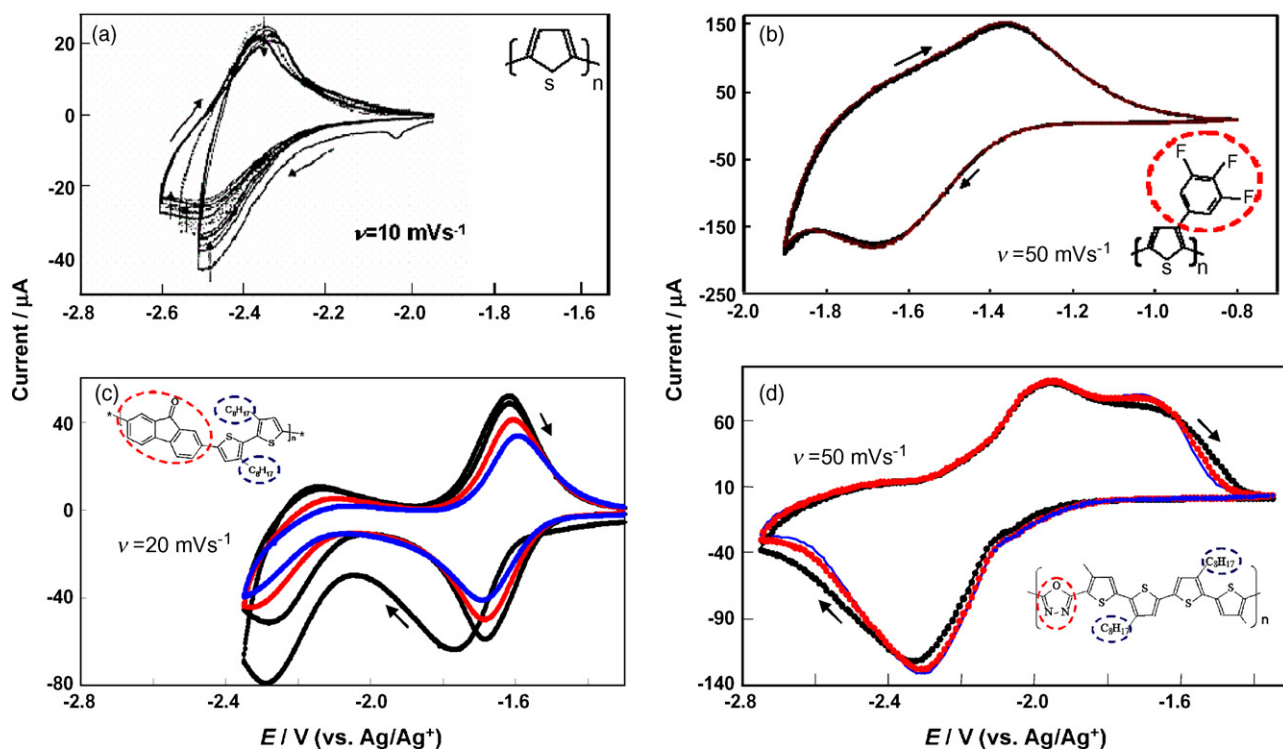


Fig. 2. Trapping of negatively charged carriers (and cations) probed by consecutive CV cycling of the four different polymers for (a) PTh, (b) P3FPhTh, (c) PFDOBTh, and (d) PMOThOD, respectively. For the PTh electrode, several cycles were performed with the gradually decreasing limiting potential (−2.50, −2.55 and −2.60 V). In panels c and d, the blue curve is the last, and the red curve is before the last cycle. Solution compositions are indicated in the caption to Fig. 1. The scan rates are shown in the panels. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

ductivity compared to the case of conjugation of the bithiophene and the fluorenone co-units.

In Fig. 2, the occurrence of charge trapping in the case of the PTh and PFDOBTh film electrodes was inferred from the improvement in the Faradaic efficiency of the n-doping reaction during their consecutive voltammetric cycling. We have confirmed this suggestion by direct impedance measurements. In the case of the PFDOBTh electrode, we chose the potential of −1.3 V (i.e., at the very beginning of the n-doping process) to probe the electrode impedance for the three situations: (i) for the fresh film (before the complete first n-doping process), (ii) after the complete n-doping process, and, finally, (iii) after the p-redoping process of the film which was first n-doped. The related Nyquist plots presented in Fig. 3a show very characteristic behavior, whereas in the case of the fresh film, the low-frequency impedance is essentially capacitive in its nature, and transforms into a rather large low-frequency semicircle after the first n-doping. This implies considerable charge trapping, as is explained below. However, the negatively charged carriers trapped can be easily removed in a consecutive process of p-doping, so that re-measuring the electrode's impedance (after an n-doping → p-doping cycle) at −1.3 V shows the expected capacitive character of the impedance spectra at the low frequency (i.e., monotonous behavior of Z'' vs. Z') which indicates the disappearance of the charge trapping phenomenon. In contrast, a similar test with the PMOThOD film electrode did not show any sign of charge trapping, since the low frequency impedance of this electrode

remains perfectly capacitive after the complete first n-doping (see Fig. 3b).

Until now, we demonstrated that the trapping of negatively charged mobile carriers depends on the electronic properties of the constituting redox-units or co-units. Fig. 4a shows another interesting aspect of the charge-trapping effect, namely, its dependence on the solvent viscosity. From the CV curves in Fig. 2b, it follows that the P3FPhTh film electrode is basically free of trapped charges as the n-doping is carried out in sulfolane electrolyte solutions. This is in agreement with the capacitive character of the low-frequency impedance of this electrode measured in this solution at a potential of −1.5 V (see the curve in the left corner of Fig. 4a marked by red circles). However, once the n-doping was conducted in a less polar and less viscous acetonitrile-based electrolyte [13], the low-frequency semicircle, characteristic of charge trapping, appears in the Nyquist plots of the P3FPhTh film electrode, similar to that of the PFDOBTh electrode in the sulfolane solution. Previously, we developed a model that may explain the origin of the low-frequency semicircle in Nyquist plots of different electroactive materials (not necessarily conducting polymers). This low-frequency semicircle can appear in Nyquist plots of electrodes due to the geometric non-homogeneity of the electroactive coating on the current collector, and/or a distribution of sites of different energy for doping (or intercalation) reactions [22]. The model developed to explain the appearance of a low frequency semicircle in the impedance spectra of redox polymers or intercalation electrodes (instead of the expected

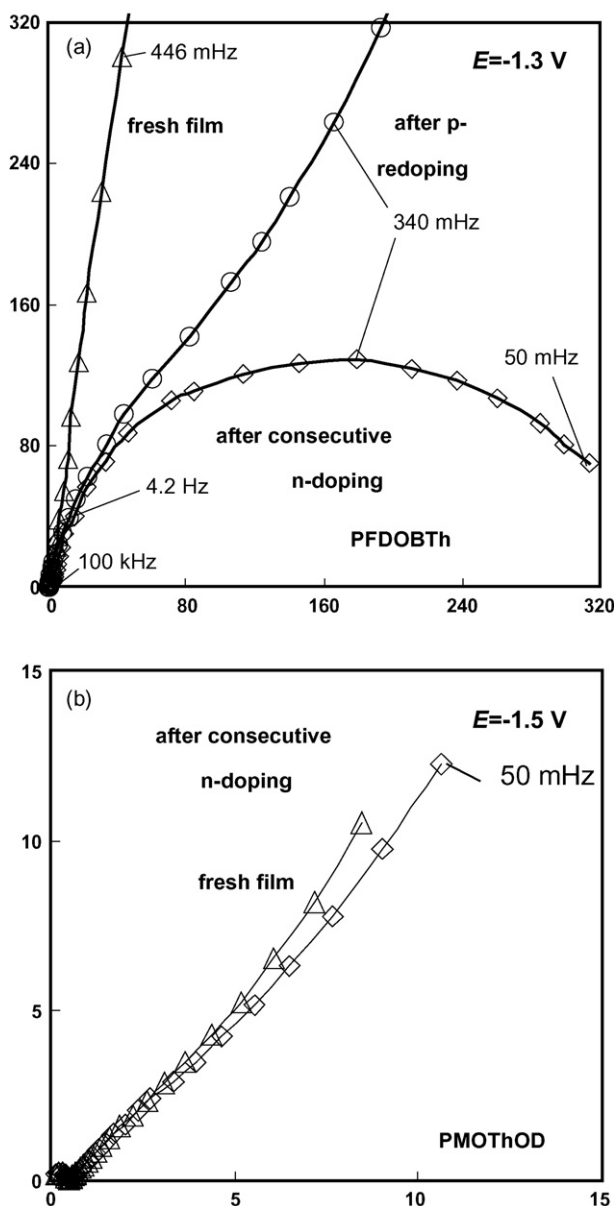


Fig. 3. (a) Nyquist plots measured with PFDOBTh at -1.30 V: (1) before the first n-doping (i.e., for freshly prepared film), (2) after consecutive n-doping; (3) after n-doping and p-redoping. Nyquist plots measured with PMOTHOD at -1.50 V: (1) before the first n-doping and (2) after consecutive n-doping.

low frequency capacitive behavior, which is a straight Z'' vs. Z' line) can be presented as an equivalent electrical circuit analogue, as shown in Fig. 4b. The most simplified presentation (analog) includes two possible sites for redox activity: one set of sites with deep interactions, i.e., high capacitive values, and one set of sites with relatively small interactions between the inserted ions and the active electrode material, to which low capacitive values can be attributed. Hence, the analogue circuit includes two R-C branches in parallel, as presented in Fig. 4b. Using a physically reasonable set of parameters, a good agreement between the experimental and model Nyquist plots was obtained (compare the curves marked by the open circles and the solid triangles in Fig. 4a). It seems that the high viscosity

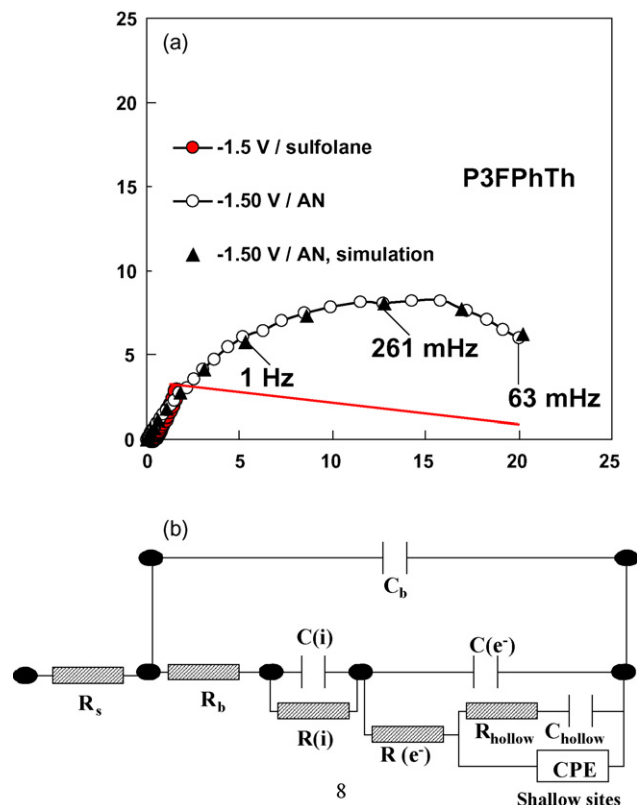


Fig. 4. (a) Nyquist plots measured with P3FPhTh at -1.50 V in sulfolane (the red-filled circles in the left corner of the graph) and acetonitrile-based electrolyte solutions (the open black circles). The curve with the filled black triangles shows the best fit of the curve calculated according to the model described in panel b to the experimental plot, related to acetonitrile solution. The fitting was performed using the equivalent electrical circuit analog for the systems with trapped charges shown in panel b (the model). The following parameters were used: $R_s = 32.1 \Omega$; $R_b = 0 \Omega$; $R_i = 4.0 \Omega$; $C_i = 3.7 \mu\text{F}$; $C_e = 0.8 \mu\text{F}$; $R_e = 18.0 \Omega$; $\text{CPE}-T = 0.028 \text{ mF}$; $\text{CPE}-P = 0.69$; $R_{\text{hollow}} = 27.0 \text{ k}\Omega$; $C_{\text{hollow}} = 2 \text{ mF}$. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

and polarity of sulfolane (as compared to that of acetonitrile) averages effectively the energies of the sites in the polymeric film for the insertion of the charge carriers (ions from the solution phase), and the penetration depths of charge carriers from the solution side, which results in a capacitive-like Nyquist plot in the low frequency domain. This reflects the average capacity of the electrode in the charge accumulation process, with no differentiation among the different sites (which leads to the typical charge-trapping effect and the related demonstrated by impedance spectroscopy). In contrast, the use of much less viscous and polar acetonitrile solutions allow the differentiation between different sites in terms of film's morphology and energy and thus demonstrates the inevitable non-homogeneity of these electrodes. This non-homogeneity of the various sites is reflected by the appearance of a large, low-frequency semicircle in the relevant Nyquist plots, which can be interpreted as a partial charge trapping by sites which processes suffer from more sluggish kinetics. This clearly demonstrates the duality of conducting polymers as mixed electronic-ionic conductors, which is strongly affected by the solvent nature (i.e., to what extent the molecules of the conventionally used solvents and the ions

really reach the entire bulk of the polymeric films during the doping process).

4. Conclusion

In this paper, we summarize our efforts directed towards the development of π -conjugated polymers with the high n-doping ability and high stability of the reduced forms of the polymers. Two alternative concepts based on (i) the introduction of an electron-withdrawing group in the 3-position of thiophene, and (ii) the introduction of electron-withdrawing moieties (subunits) together with π -conjugated bi- or quatro-thiophene subunits in the polymer backbone, are compared. Exceptionally good results were obtained for PMOThOD film electrodes, in which the π -conjugation includes both quatro-thiophene units and the highly electron-withdrawing oxadiazole subunits. We discuss herein the effect of trapping of the negatively charged electronic carriers measured by both cyclic voltammetry and electrochemical impedance spectroscopy, and suggest a model presented as an equivalent circuit analog that explains the characteristic impedance spectra. Both the electronic properties of the electron-withdrawing co-unit in the main backbone of the copolymer and the nature of the solvent used affect the intensity of the charge trapping phenomena upon the reversible doping of π -conjugated polymers.

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