

ELECTROCHEMICAL SYNTHESIS AND OPTICAL PROPERTIES OF POLYAMINOTHIAZOLE ON SEMICONDUCTIVE ELECTRODES

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Conductive polymers, since their discovery in 1970s, caused a revolution in various fields of science, due to their electronic properties and potential applications. They are already used as ion sensors, biosensors, electrochromic devices, electrochemical batteries and corrosion inhibitors [1-5].

Conducting polymers are one of the most interesting classes of low-dimensions (1D) materials. In contrast to conventional semiconductors the charge transport in these systems is realized by nonlinear topological excitations created in polymer chains as a result of Peierls instability, namely - solitons in trans-polyacetylene, polarons and bipolarons in conjugated polyarenes – poly-*para*-phenylene, polyaniline [6, 7] and others. These polymers exhibit a semiconductor property; the reason to that is the existence of the conjugated π -electron bonds. Conjugation of electron system of neighboring elementary link lead to charge delocalization along the polymer chain and possibility of charge transport both in one dimension, and in the three dimensions over the interchain transitions.

The presence of a conjugated system of π -electron bonds and aromatic fragments determines the optical absorption of conjugated polymer systems in the visible part of the spectrum, as well as near UV and IR regions of the spectrum. These properties can be used to create functional devices for various purposes, such as optical sensors, light valves, color indicators [8-10]. Recently, there is a growing interest in polymeric materials as active components of display coatings, light emitting diodes, recording and information storage systems. Both linear and nonlinear optical properties of polymers, their ability to change the spectrum under

the action of external factors (electrochromic, thermochromic, photochromic), radiation under the action of an electromagnetic field, the presence of photocurrent are important [11-16].

Electrically conductive polymers are easily reduced by *n*-doping or oxidation by *p*-doping. The polymer matrix itself can act as both a donor and an electron acceptor. External physical and chemical factors - temperature, radiation, electric or magnetic field, adsorption of gases, solvents or ions change the electronic properties of conjugated polymer systems - the energy of the band gap, the concentration of charge carriers and their mobility [6, 9].

Family of conducting polymers is very widely and includes different classes of polyconjugated compounds – polyacetylene and its derivatives, polyanilines, polythiophenes, polypyrroles, polyphenylene and polyphenylenevinylene, polyethylenedioxythiophenes etc. [6-10]. However, the conducting polymers with heteroatoms in the aromatic rings and in the link of conjugation are studied unsatisfactorily. One of such polymers is poly(2-aminothiazole) that contained sulfur and nitrogen atom in pentacyclic fragment connecting via amino group [17-20].

Poly(2-aminothiazole) (PAT) is an electrically conductive conjugated polymer that exhibits a range of interesting properties. Materials based on poly(2-aminothiazole) adsorb heavy metal ions from aqueous and organic medium. Bıyıkoğlu M. and Çiftçi H. developed a method of selective adsorption of Ag(I) ions from wastewaters using poly(2-aminothiazole) [1]. Selective concentration or even determination of one of the ions in the presence of others is possible using poly(2-aminothiazole). Regina M. Takeuchi and André L. Santos reported about new chemically modified solid paraffin-based carbon paste electrode that was developed using 2-aminothiazole organofunctionalized silica for Cu²⁺ determination in commercial ethanol fuel samples. Interference effects of other ions decreased in that order Ni > Zn > Cd > Pb > Fe, but they did not limit the usage of the method [21]. One more example is determination of Ag(I) with PAT-Pt electrode in the range of 2.8·10⁻⁷ – 20.4·10⁻⁵ mol L⁻¹ Ag(I) concentrations [22]. Ni(II), Co(II) and Al(III) ions did not interfere in the

Ag(I) determination, however Cu(II) had significant effect. In addition, 2-aminothiazole is biologically active compound with antimicrobial, antitumor properties. According to Shiv Jee Kashyap [23] thiazole ring is a part of the active substances of many drugs. Some 2-aminothiazole derivatives have anticorrosion properties [4] that can be present in poly(2-aminothiazole) too. High biological activity of aminothiazole which contains in its structure heteroatoms of nitrogen and sulfur and is able to form complexes with ferric ions, making it a potential candidate for modeling the behavior of magnetic centers in metalloprotein [17, 18]. However, despite the prospects of applications of PAT the number of publications on its synthesis and usage of the polymer itself is limited.

Synthesis of poly(2-aminothiazole) can be carried out by two methods: chemical synthesis and electrochemical. Chemical synthesis is mostly carried out in organic solvents with various oxidants as initiators of reaction [24-25]. Polyaminothiazole with specific volume conductivity near 3×10^{-4} Sm/cm was synthesized by the oxidative polymerization of aminothiazole in the organic solvents under action of ferrum (III) chloride [19]. Xia Wang and Hua Zou in their works conduct synthesis in aqueous solution with three different oxidants: CuCl_2 , FeCl_3 and $(\text{NH}_4)_2\text{S}_2\text{O}_8$. The results suggested that the monomer higher yields were achieved when CuCl_2 was used as the oxidant [24]. Also they have studied 2-aminothiazole polymerization in nitromethane (CH_3NO_2) using FeCl_3 as an oxidant [26]. There are works that report about polymerization in 1,4-dioxane with benzoyl peroxide as initiator [27]. Chemical synthesis allows to obtain a great amount of polymer, even for industrial purposes. Several strategies were reported for the nanostructured conducting polymers synthesis, including template-free, template-based and physical approaches, for example, electrospinning [3].

Electrochemical polymerization is a simple method to produce thin polymer films on different surfaces (directly on the electrode surface). This method enables to influence the resulting polymer properties by reaction conditions [8, 9]. Moreover, electrochemical synthesis technique allows obtaining a simultaneously doped conductive polymer, the deposition of insoluble polymers and easy electrochemical

control of the film thickness [9]. Anodic synthesis conditions are found that provide the polyaminothiazole conducting film synthesis on platinum and tin dioxide electrodes in organic solvents [20]. IR and electron spectroscopy studies show that the polymer synthesized on the electrode surface contains fragments of π -conjugated bonds that provide optical absorption in the visible region and electrochemical activity of the film.

Electrochemical synthesis can be conducted in three different regimes: galvanostatic, when current is constant, potentiostatic, when potential is constant, voltammetry, when potential is function of time. Cyclic voltammetry allows to study processes that take place on electrode surface during the polymerization.

Transparent conductive layers are required to fabricate highly efficient elements of optoelectronic devices using organic semiconductors (conjugated polymers). Currently, the so-called "conductive glass" is widely used - glass plates with a layer of SnO_2 oxide applied on them [28, 29]. The most common are also transparent conductive layers for flat panel displays, electroluminescent and electrochromic displays, OPV and OLED - these are thin films of indium-tin oxide [29]. Particularly interesting (for creating optical sensors) is deposition active films on the optically transparent electrodes, for example, SnO_2 electrode. The main achievement in the manufacture and application of transparent semiconductor electrodes is the combination of high transparency in the visible part of the spectrum with sufficient conductivity.

Therefore, the aim of our research is to find optimal ways of electrochemical synthesis of poly(2-aminothiazole) by cyclic potential scanning or voltammetry (CVA) on different electrodes, especially transparent ones, and to study properties of the obtained films.

Experimental

Materials

2-aminothiazole (CAS-number: 96-50-4; $M=100.14$ g/mol; UOSlab,) – light yellow crystals; soluble in water (100 g / l at 20°C), alcohols, diethyl ether.

Electrolytes - sulfuric acid (fixanal), ammonium oxalate monohydrate ($(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$, >98%), lithium perchlorate LiClO_4 (Aldrich).

Solutions were prepared either with distilled water or with acetonitrile as organic solvent.

Electrochemical cell consisted of a three-electrode system: as a model working electrode platinum sheet ($S = 2 \text{ cm}^2$) was used, also optical transparent electrode covered with conductive SnO_2 layer was used ($S = 2 \text{ cm}^2$); as a reference electrode – saturated Ag/AgCl ; as a counter electrode – platinum mesh. Electrochemical measurements were performed using a potentiostat MTechPGP-550M with special software connected to a PC. All results were recorded on the monitor screen and processed by Origin 6. Synthesis was conducted at a sweep rate 20 mV/s in potential range from -0,2 V to 1,5 V in aqueous solution and in potential range from -0,2 V to 2,5 V in organic solvent. Thin films of PAT were obtained from 0,1 M aqueous monomer solution at the presence of 0,3M ammonium oxalate or 0,5 M sulfuric acid as electrolytes or from 0,1 M monomer solution at the presence of 0,1 M lithium perchlorate in acetonitrile.

In order to get information about electrochemical activity of PAT films, cyclic voltammograms at different sweep rates at the presence of 0,3M ammonium oxalate, 0,5 M sulfuric acid or 0,1 M LiClO_4 (acetonitrile solution) as electrolytes were obtained.

To study optical properties of PAT, spectral measurements were carried out on photocolorimeter KFK-3 in the range of wavelength 310-990 nm.

Doping was carried out as follows: $\text{K}_3[\text{Fe}(\text{CN})_6]$ as a dopant, electrode surface with PAT film on it was soaked for 20 min at 0,005 M solution of dopant, further it was pulled out, washed with distilled water and used for investigation.

Results and discussion

During the polymerization in the organic solvent thin light-yellow film of PAT was formed on the surface of SnO_2 transparent electrode. According to multiple cyclic voltammogram (Fig.1) electrooxidation of monomer occurred during first potential sweep at the peak potential $E = 2,0 \text{ V}$ (versus Ag/AgCl reference). Later this

peak disappeared due to the film formation on the electrode surface and further oxidation takes place on the surface of formed electroactive film at $E = 1,2$ V.

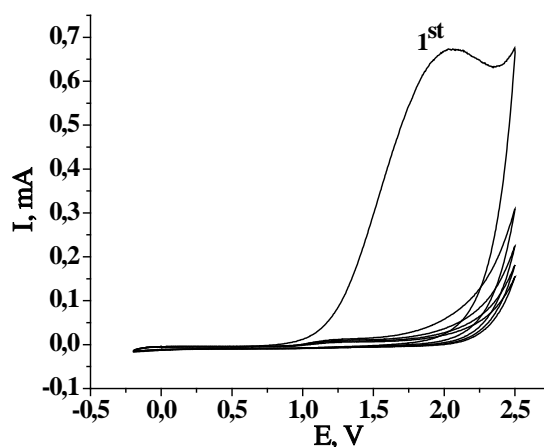


Fig. 1. CVA obtained in the electrochemical polymerization process from 0,1 M acetonitrile solution of monomer at the presence of 0,1 M lithium perchlorate on SnO_2 electrode.

Behavior of 2-AT polymerization from aqueous solution at the presence of 0,3M ammonium oxalate as electrolyte is slightly different. As can be seen from multiple cyclic voltammograms (Fig.2) peak potentials shift to the left comparatively with those from acetonitrile. Electrooxidation of monomer on the optical transparent electrode covered with conductive SnO_2 layer occurred the peak potential $E = 1,0$ V (versus Ag/AgCl reference) (Fig.2a), while on the platinum sheet (Fig.2b) it shifts more to the $E = 0,8$ V. Oxidation of 2-aminothiazole was more active on platinum sheet in sulfuric acid medium that can be seen from higher current value during the first potential sweep (Table 1).

Table 1. Oxidative potential of 2-AT at the different conditions

Material of electrode	Solvent	Electrolyte	Oxidative potential, V	Peak current, mA/cm^2
SnO_2	Acetonitrile	0,1 M LiClO_4	2,0	0,34
SnO_2	Water	0,3 M $(\text{NH}_4)_2\text{C}_2\text{O}_4$	1,0	0,54
Pt	Water	0,3 M $(\text{NH}_4)_2\text{C}_2\text{O}_4$	0,8	0,27
Pt	Water	0,5 M H_2SO_4	1,2	0,85

Like from the organic solvent peak disappeared after the first cycle and further oxidation occurred at $E = 0,7 \text{ V}$ (at both electrodes). This new peak corresponds to the formed electroactive film on the electrode surface. The potential value did not change in subsequent cycles on both electrodes and from both solvents, while peak current slightly changed to the bigger values with every cycle, that means that film was slowly growing. As a result light yellow film was formed on the electrode surface as well. At the presence of sulfuric acid as electrolyte polymerization from aqueous solution happen only on Pt electrode at $E = 1,2 \text{ V}$ (Fig.3).

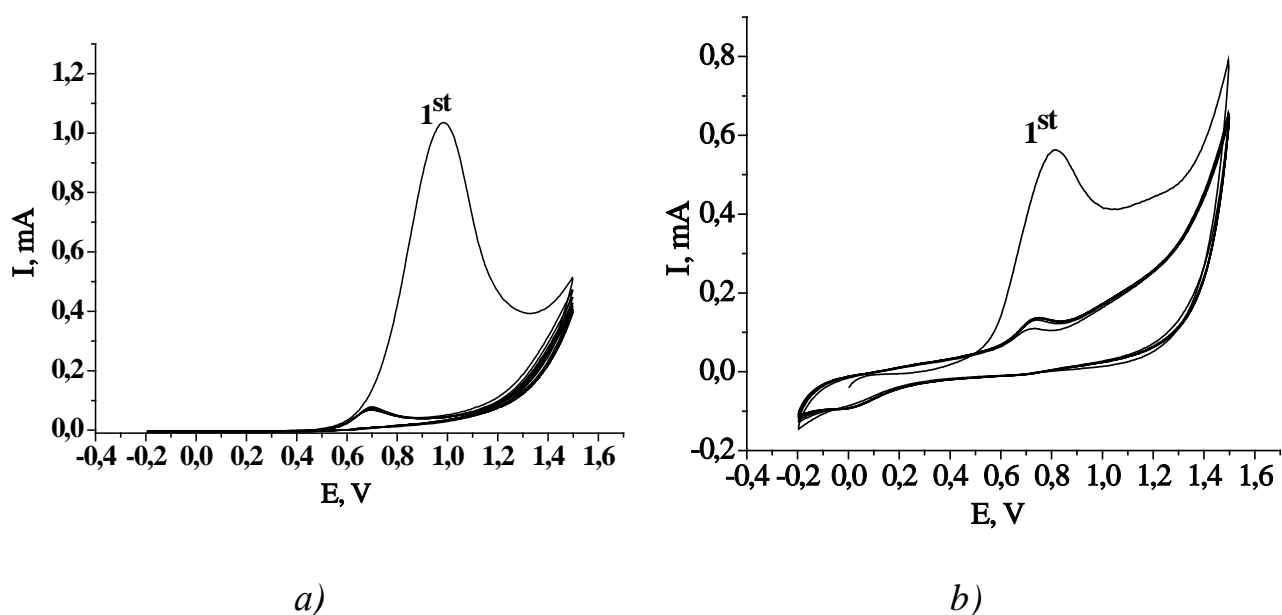


Fig. 2. CVA obtained in the electrochemical polymerization process from 0,1 M aqueous solution of monomer at the presence of 0,3 M $(\text{NH}_4)_2\text{C}_2\text{O}_4$ on SnO_2 electrode (a) and on platinum sheet (b).

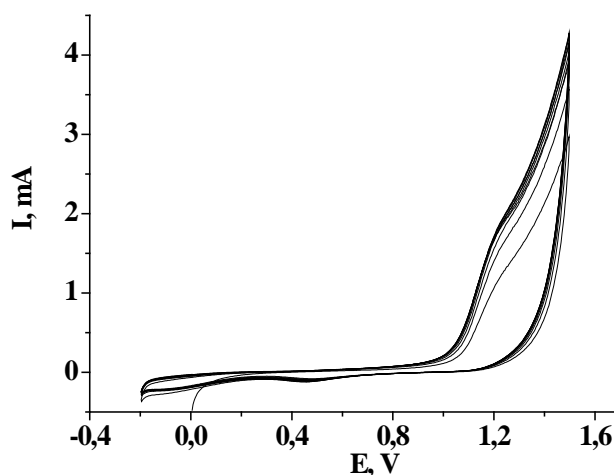


Fig. 3. CVA obtained in the electrochemical polymerization process from 0,1 M aqueous solution of monomer at the presence of 0,5 M H_2SO_4 on Pt electrode.

The look of multiple cyclic voltammograms can be explained by suggested in the literature [30] mechanism of polymerization (Fig.4). In the first stage, that corresponds to the first cycle, as a result of monomer oxidation the formation of cation-radical on amino group takes place. In the following stages, that correspond to following cycles, the growth of polymer film begins because of the recombination of radicals with simultaneous deprotonation. After that rearrangement with one more deprotonation occurs.

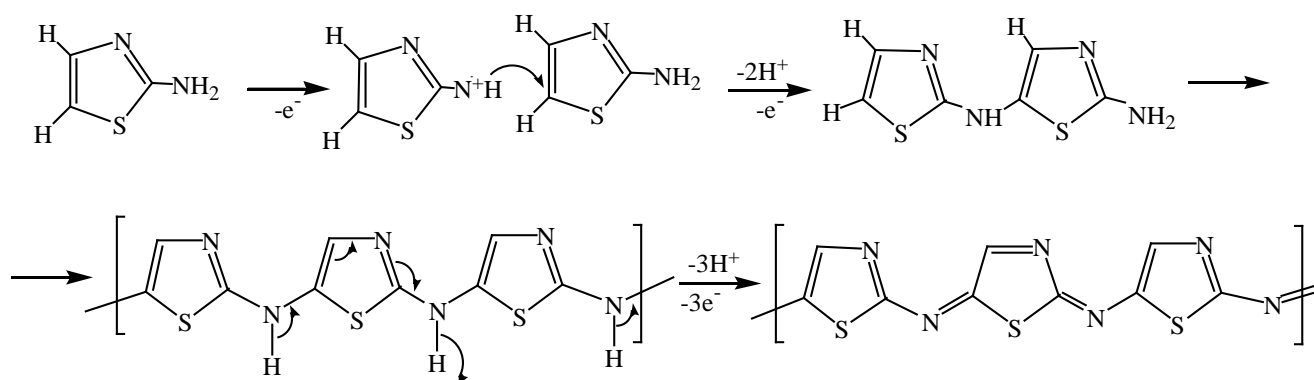


Fig. 4. Mechanism of electrooxidation and oxidative coupling of 2-aminothiazole

Films of PAT obtained from both solvents show electrochemical activity as in cyclic voltammograms in the present of different electrolytes peaks occur. Dependence between the peak current and the scan rate was studied at the presence of 0,1 M $LiClO_4$ in acetonitrile solution for the film obtained from organic solvent on transparent electrode and at the presence of 0,5M sulfuric acid or 0,3 ammonium oxalate in aqueous solution for the film obtained on platinum electrode. The peak current does not depend on scan rate in organic solvent on transparent electrode while it increased with increasing of scan rate in both cases in aqueous solution. (Fig.5). The electrolyte nature can significantly affect electrochemical activity. As can be seen from cyclic voltammograms one more zone of electrochemical activity at $E = 0,25$ V appears (Fig.5a) at the presence of sulfuric acid. Sulfuric acid as a strong acid acts as a dopant for PAT film, that leads to intensifying of electrochemical activity and film conductivity.

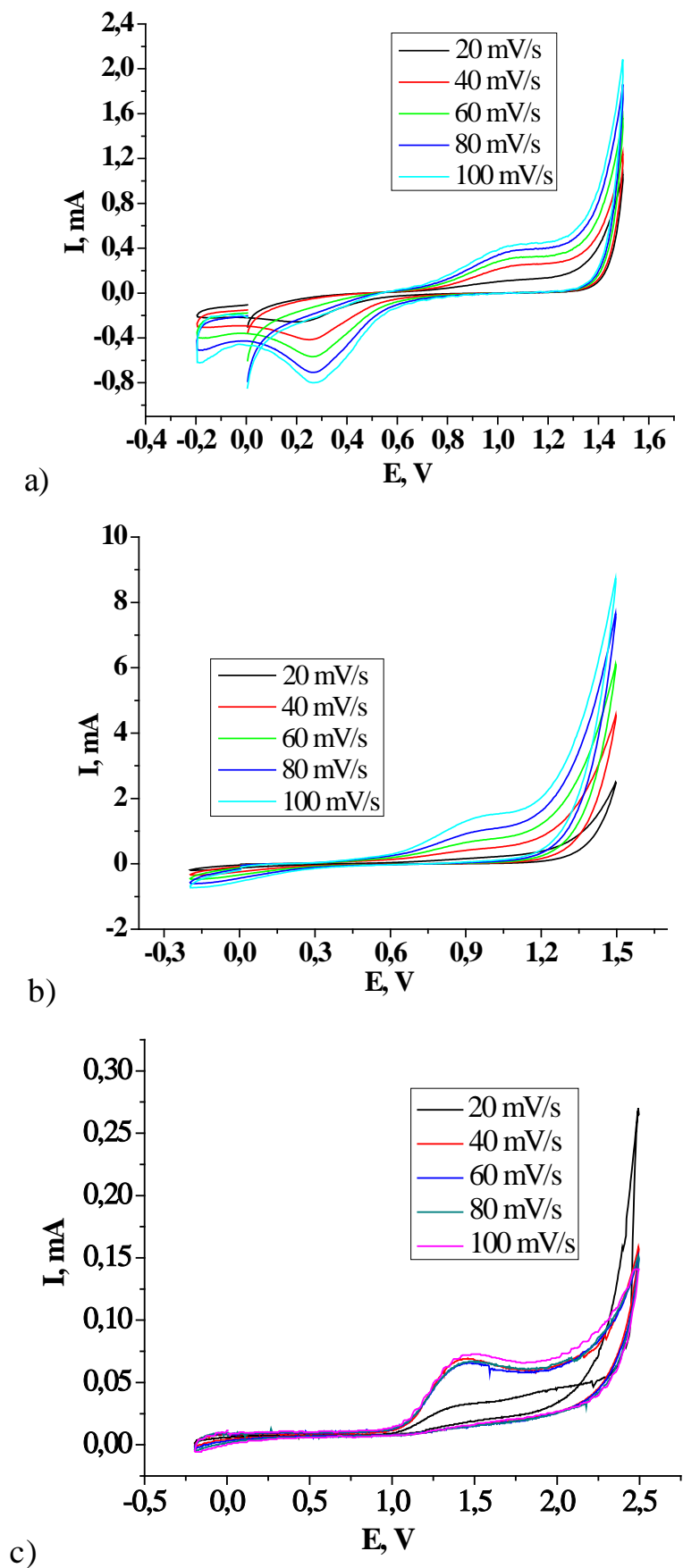


Fig. 5. CV of PAT films at different scan rates at the presence of *a)* 0,5 M H_2SO_4 , aqueous solution, *b)* 0,3 M $(\text{NH}_4)_2\text{C}_2\text{O}_4$, aqueous solution (platinum sheet), *c)* 0,1 M LiClO_4 , acetonitrile solution (transparent electrode)

For the film in the acetonitrile medium, where current values did not change with increasing of the scan rate, electrochemical processes probably can be limited by slow kinetics stage [31, 32]. Linear dependence between the peak current and the scan rate at 0.5 degree was obtained at the presence of H₂SO₄ and (NH₄)₂C₂O₄. This can be explained by diffusion limitation of electrochemical processes in PAT film (Fig.6).

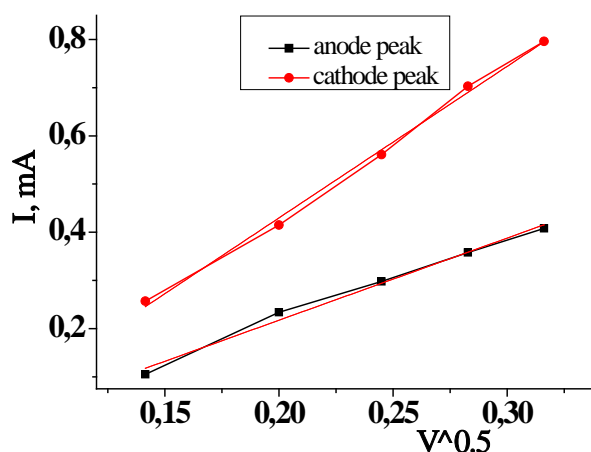


Fig. 6. Dependence between the peak current and the scan rate at 0.5 degree at 0,5 M H₂SO₄(anode and cathode peaks)

This allows to apply the model of semi-infinite diffusion and Randles - Shevchik equation (1) to determine effective coefficients of charge diffusion in polymer film [8].

$$i_p = 2,69 \cdot 10^5 n^{3/2} S D_{ef}^{1/2} \nu^{1/2} C^* \quad (1)$$

Equation for calculating the concentration of active centers

$$C^* = Q/nFSl, \quad (2)$$

(where n is the number of electrons involved in the redox process, S is the area of the film (cm²), C^* is the concentration of active centers in the film (mol/cm³), Q is the total charge calculated by integrating the anode or cathode part of the CV curve obtained at a slow scan speed, l is the film thickness (cm), F is the Faraday constant, C).

To determine the concentration of active centers in the PAT film, graphical integration of CVA curves was performed using the mathematical program Origin6.0. To calculate the concentration of active centers, the anode and cathode branches of the CVA curve, obtained at $\nu = 20$ mV/s, were used, under conditions when the largest number of redox centers of the polymer layer have time to oxidize or reduce.

As can be seen from the values of the diffusion coefficients, the rate of electronic transfer is higher for the anode process at the presence of both electrolytes, as confirmed by the higher diffusion coefficient (Table 2).

Table 2. Electrochemical activity of PAT films

Medium	Process	E, V	$C^* \cdot 10^4$, mol/cm ³	D_{ef} , cm ² /sec
0,5 M H ₂ SO ₄	Cathodic	0,25	2,47	$5,6 \cdot 10^{-10}$
	Anodic	1,03	2,69	$1,37 \cdot 10^{-8}$
0,3 M (NH ₄) ₂ C ₂ O ₄	Anodic	0,93	7,32	$2,03 \cdot 10^{-8}$

To interpret the optical absorption spectra, it is necessary to have data on the electronic structure and type of charge carriers. The transitions of electrons from the valence band to the conduction band cause the formation of holes in the valence band, and the motion of electrons in turn causes a curvature in the polymer matrix that accompanies the motion of the electron. The electron and the polarizing field it forms are called polarons. When another electron appears in the polymer matrix, it forms bipolaron due to dimerization paired with polaron [6]. The presence of polaron and bipolar direction depends on the level of doping of the conjugated polymer.

The optical spectra of PAT films obtained from both solvents are slightly different, however characteristic of conjugate polymer systems (Fig. 7). For example, the absorption inherent in such systems within wavelengths of 400 -500 nm, which extends into the UV range, and is associated with (π - π^*) electronic transition of aromatic structures in the conjugate system. The intense band of the film obtained in aqueous solution with a maximum at 600 nm, characteristic of the electronic

transition ($n-\pi^*$ in imino-quinoid structures) of polyaminoaromatic compounds. Absorption in the range of 850-900 nm, extending to the near IR region, corresponds to absorption of delocalized charge carriers in the film of PAT [33].

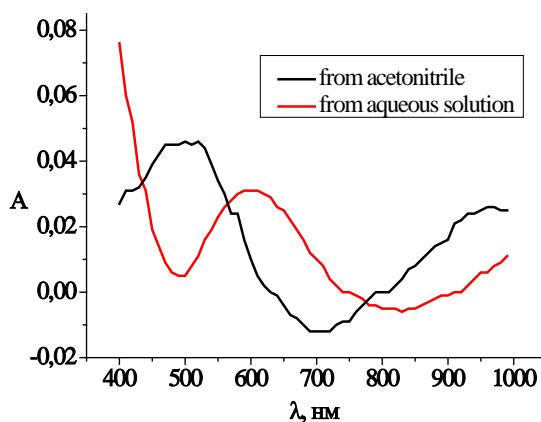


Fig. 7. Absorption spectrum of PAT films obtained from different solvents

Doping of conjugated polymers can lead to useful outcome, for example, such as changes in electroactivity or spectrum characteristics [34]. Potassium ferricyanide $\text{K}_3[\text{Fe}(\text{CN})_6]$ is a dopant that attracts a lot of attention as is used in many amperometric biosensors as an agent for electron transfer. As was investigated complex compound $\text{K}_3[\text{Fe}(\text{CN})_6]$ has significant effect on optical characteristics of thin films obtained from aqueous solution. After doping a hypsochromic shift of the central band is observed by almost 50 nm (from 600 to 550 nm) and intensity of this band increases considerably. Also new bands at approximately 680 nm and 890 nm appear that can be associated with the formation of free charge carriers [33-35].

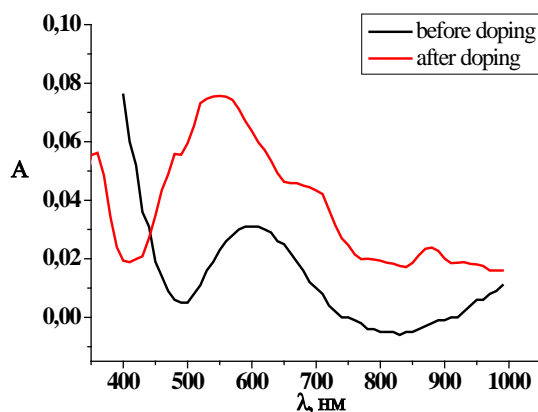
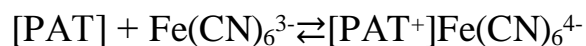


Fig. 7. Absorption spectrum of PAT film before and after doping with $\text{K}_3\text{Fe}(\text{CN})_6$

Changes in spectrum are due to the course of redox processes. We can assume that dopant acts as oxidant mainly, considering the hypsochromic shift of the absorption maximum. As a result, part of neutral fragments of PAT turn into oxidized, while the iron ion changes degree of oxidation from +3 to +2:



Formation of PAT-Fe(CN)₆ complex can probably explain appearance of the absorption maximum at 870 nm as well as considerable hypsochromic shift of absorption maximums.

Conclusion

The process of electrooxidation of 2-aminothiazole (2-AT) on Pt and semiconductor SnO₂ electrode in aqueous and organic medium were investigated by cyclic voltammetry method. It is found that oxidative potential of 2-AT monomer on SnO₂ electrode in organic solvent is near 2,0 V that significantly higher than in aqueous solutions (1,0 V). The process is accompanied by electroactive polymer film formation on the electrode surface. On the base of peak current dependences on sweep rate in 1/2 degree values of the effective diffusion coefficients (D_{ef}) for charge transport were calculated. It is established that the rate of electron transfer in the PAT film is higher for the anode process in both oxalate and sulfuric acid electrolytes and is in the range D_{ef} = (1,4-2)10⁻⁸ cm²/s. The optical absorption spectra of obtained films on the transparent SnO₂ electrodes are typical as for organic semiconductor and conjugated polymer systems. Doping of PAT film with complex K₃[Fe(CN)₆] has significant effect on optical characteristics of thin films obtained from aqueous solution. Obtained results will be used in further study of properties and fields of application of poly(2-aminothiazole).

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