Preparation and Characterization of Poly(5-aminoindole) by using Electrochemical Quartz Crystal Nanobalance Technique

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Abstract

The electrochemical quartz crystal nanobalance (EQCN) was employed to study the electropolymerization of 5-aminoindole on platinum electrodes in acidic media. Potentiostatic or potential cycling electrooxidation of 5-aminoindole below 0.62 V vs. SCE leads to the formation of uniform, yellow, electrochemically active polymeric films. A scheme of the redox transformations of poly(5-aminoindole) which involves protonation-deprotonation steps accompanying the electron transfer is suggested. At higher positive potentials, further oxidation takes place resulting in different blue-purple, indigo-type materials which remain attached to the metal surface but show decreased or no redox activity.

Keywords: 5-aminoindole; electropolymerization; electrochemical quartz crystal nanobalance; redox behavior; overoxidation

1. Introduction

After more than three decades, the preparation, characterization and application of electrochemically active polymeric films are still in the foreground of research activity in electrochemistry.[1,2] This intense interest is understandable since several applications in the fields of energy storage, electrocatalysis, organic electrochemistry, bioelectrochemistry, photoelectrochemistry, electroanalysis, sensors, electrochromic displays, microsystem technologies, electronic devices, microwave screening and corrosion protection, etc. have been accomplished.[1,2] Showing similarities to pyrrole, experiments with indole were performed also as early as 1982.[3] Indole and indole derivatives have been studied thoroughly, and a wide range of applications for its polymers from corrosion protection via Li-ion batteries to ion sensors and electrochromic devices have also been suggested.[3–37] However, there are still controversial issues reported in the literature concerning the structure of the formed polymers. In the case of polyindole, theoretical considerations about the polymer structure support the C2-C3, C2-C7 connections.[18,28] Spectroscopic data obtained for indole derivatives suggest the C2-C3 polymerization as well.[30] The formation of cyclic forms is

not excluded, either.[13,28] The electropolymerization of indole derivatives such as 5-carboxyindole,[6,10,20,30] 5-cyanoindole,[11,16,19] 4-fluoroindole,[31] 5-chloroindole,[26] 5-aminoindole,[12,26,34,35] 5-nitroindole[17] have been studied. Jennings et al.[12] claimed, that amino or hydroxy substituted indole derivatives (5-aminoindole and 5-hydroxyindole) adsorbing on the surface of a platinum electrode via the substituents inhibit the redox active film formation. In order to clarify the contradictions regarding the electropolymerization and redox behavior of indole and indole derivatives, we have started a systematic investigation by using electrochemical quartz crystal nanobalance (EQCN) technique. It was hoped that by the help of piezoelectric nanogravimetry a deeper understanding could be achieved on the role of adsorption of the monomer, on the film formation during electropolymerization and on the redox mechanism by monitoring the ionic exchange processes accompanying the redox transformations of the polymers. We have studied the electropolymerization of indole as well as 4- and 6-aminoindoles, recently.[36,37] According to our findings, electrochemically active polymers could be produced from these monomers by electropolymerization, however, their properties strongly depend on the conditions, especially on the potential range applied. The ad-

sorption of amino-substituted indoles really exists on platinum; however, it does not prevent the reaction. It has been found that the amino group remains intact during the electropolymerization.[34,36,37]

In this work we present the results of the electropolymerization of 5-aminoindole (Fig. 1.) in acid media, and the nanogravimetric characterization of poly(5-aminoindole) formed.
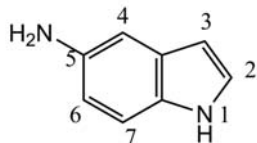
Figure 1. The formula of 5-aminoindole and the numbering of atoms on the indole ring.

Based on the simultaneously measured charge and mass responses, reaction schemes are proposed concerning the reversible redox transformations of poly(5-aminoindole) and the overoxidation process.

2. Experimental

Analytical grade chemicals such as H_2SO_4 (Merck), Na_2SO_4 (Merck), $K_3[Fe(CN)_6]$ (Reanal, Budapest) and 5-aminoindole (Sigma-Aldrich) were used as received. Doubly distilled water was used (Millipore water). The solutions from 5-aminoindole were always freshly prepared. The solutions were deaerated before the monomer was dissolved. All solutions were purged with oxygen-free argon (purity: 5.0, Linde Gas Hungary Co. Cltd.) and an inert gas blanket was maintained throughout the experiments.

Five megahertz AT-cut crystals of one inch diameter coated with platinum with titanium underlayer (Stanford Research Systems, SRS, U.S.A.) were used in the EQCN measurements. The electrochemically and the piezoelectrically active areas were equal to 1.37 cm² and 0.4 cm², respectively. The Sauerbrey equation was used to relate the surface mass change (Δm) to the frequency shift (Δf):

$$\Delta f = - C_f \Delta m/A \tag{1}$$

The integral sensitivity of the crystals (C_f) was found to be 56.6×10^6 Hz g⁻¹ cm², i.e., 1 Hz corresponds to 17.7 ng cm⁻². The apparent molar mass of the deposited or the exchanged species (M) was calculated from the slope of the Δf vs. Q curve using the following formula:

$$M = (nFA/C_f) \, \mathrm{d}\Delta f/\mathrm{d}Q, \tag{2}$$

where n is the number of electrons involved in the electrochemical reaction, F is the Faraday constant, Δf is the frequency change, Q is the charge consumed and A is the electrode surface area.

Platinum wire was used as a counter electrode. The reference electrode was a sodium chloride saturated calomel electrode (SCE) separated with a double frit from the main compartment. An Electroflex 450 potentiostat (Szeged, Hungary) was used for the measurements.

Electropolymerization in acid media was carried out by potentiodynamic cycling at 50 mV · s⁻¹ or potentiostatically at different potentials keeping the positive potential limit below 0.62 V. The concentration of the monomer solutions was 5×10^{-3} mol dm⁻³. The deposited films were also investigated by Focused Ion Beam Scanning Electron Microscope (FIB-SEM) type FEI Quanta 3D, the Netherlands.

# 3. Results and Discussion

3. 1. Electropolymerization of 5-aminoindole at Platinum in 0.5 mol dm⁻³ Sulfuric Acid

According to our preliminary measurements, the formation of the polymer film strongly depends on the positive potential limit applied during potential cycling or the potential used in the course of potentiostatic deposition. At potentials higher than ca. 0.62 V (under conditions of 0.5 mol dm⁻³ H_2SO_4 and 5 mmol·dm⁻³ 5-aminoindole and at a scan rate, $v = 50$ mV·s⁻¹) an overoxidation of the polymer starts which eventually leads to an electrochemically non-active film. On the other hand, when the potential is kept under this limit the polymer formed shows a reversible redox behavior. Therefore, we avoided the potential range higher than 0.62 V, except in the studies of the overoxidation. Albeit the adsorption of 5-aminoindole on Pt occurs, indeed, we did not observe its inhibiting effect.[12] Figure 2 shows the cyclic voltammogram and the respective EQCN frequency response obtained at a platinum electrode in contact with 0.5 mol dm⁻³ H_2SO_4 and with a solution containing 0.5 mol dm⁻³ H_2SO_4 solution and 5 mmol·dm⁻³ 5-aminoindole, respectively. In the case of the latter solution the very first cycle is presented in Figure 2. It is evident that an adsorption of the organic compound takes place since the hydrogen waves in the hydrogen underpotential deposition (UPD) region decreases (Figure 2a) as well as the initial frequency value is smaller by ca. 40 Hz than in pure sulphuric acid solution (Figure 2b). From this value – taking into account of the surface roughness of the platinum – a surface concentration of $2–6 \times 10^{-9}$ mol cm⁻² can be calculated, i.e., at least a monolayer of the adsorbed compound was formed on the Pt surface. It is also possible that not only monomeric species participate in the adsorption but also dimeric or trimer species that can be formed during the spontaneous oxidation of 5-aminoindole by the traces of oxygen present in the solution.[38] Furthermore, the frequency decrea-
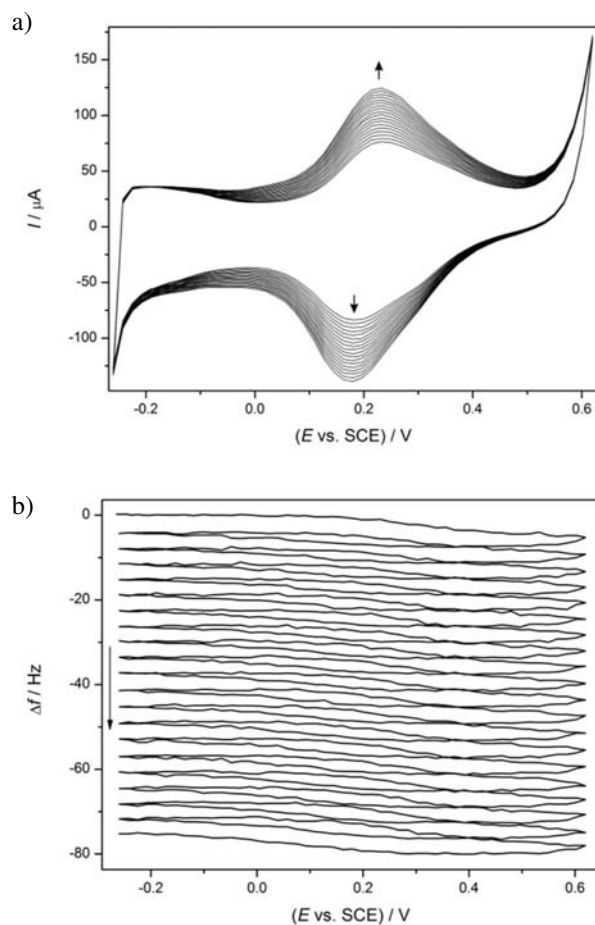
se is higher than that detected in the double layer region of clean platinum.

It is also seen that the electrooxidation of 5-aminoindole starts about 0.5 V.

a)

b)
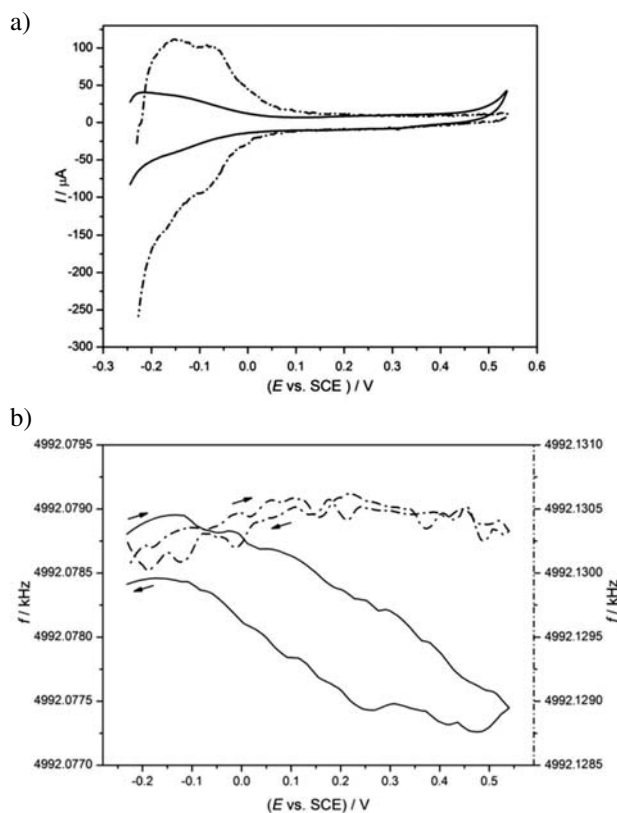
Figure 2. Comparison of the cyclic voltammetric curves (a) and the corresponding frequency changes (b) obtained for a clean platinum electrode in 0.5 mol dm^{-3} H$_2$SO$_4$ solution (dotted dashed line) and in contact with a solution containing 0.5 mol dm^{-3} H$_2$SO$_4$ solution and 5 mmol dm^{-3} 5-aminoindole (continuous line). Note that the left y-axis and the right y-axis correspond to the frequency change of the full line curve and the dotted dashed curve, respectively. $v = 50$ mV · s^{-1}.

a)

b)

Figure 3. The cyclic voltammetric curves (a) and the corresponding frequency changes (b) obtained during the electropolymerization of 5-aminoindole at Pt in contact with a solution containing 0.5 mol dm^{-3} H$_2$SO$_4$ solution and 5 mmol dm^{-3} 5-aminoindole. $v = 50$ mV · s^{-1}. Seven consecutive runs involving 20 cycles each were applied. For the better visibility only 20 cycles of the 2nd run are displayed in Figures 3a and 3b.

Under these conditions the polymer film gradually grows during potential cycling (Figure 3). A slight increase of the positive limiting potential causes no changes in the redox response of the electrochemically active polymer. The rate of the polymer formation increases when higher positive switching potential is applied, however, as the film becomes thicker the rate of the polymer formation somewhat slows down. It is partially due to the fast consumption of the monomer in the vicinity of the substrate which also manifests itself in the difference of the first and the subsequent cycles in each run involving 20 cycles, during the waiting time at the initial potential, the original monomer concentration is restored at the surface. However, a closer inspection reveals that other changes also occur: the anodic wave slightly shifts to the direction of more positive potentials, while the cathodic peak considerably shifts to the more negative potentials. A pair of prewaves also appears at ca. 0 V on the expense of the main wave which shows a slight decrease of the peak current intensity.

The molar mass values of the deposited species – which were calculated by Equation (2) using $n = 2.2$, i.e., considering both the charge related to the electropolymerization and the oxidation of the polymer formed – shows a decreasing trend. During the first 20 cycles $M = 95 \pm 10$ g mol^{-1} was found, while after 120 cycles this value gradually decreased to $M = 40 \pm 10$ g mol^{-1}. Taking into account of the molar mass of 5-aminoindole, $M = 132.16$ g mol^{-1} and also the elimination of two protons, it follows that in the first phase of the deposition the film contains more anions and water molecules. If we consider the full protonation of the amino group and consequently one HSO$_4^-$ charge compensating ion $M = 227$ g mol^{-1} could be expected. Therefore, the amino groups of the poly(5-aminoindole) is only partially protonated.

a)

b)
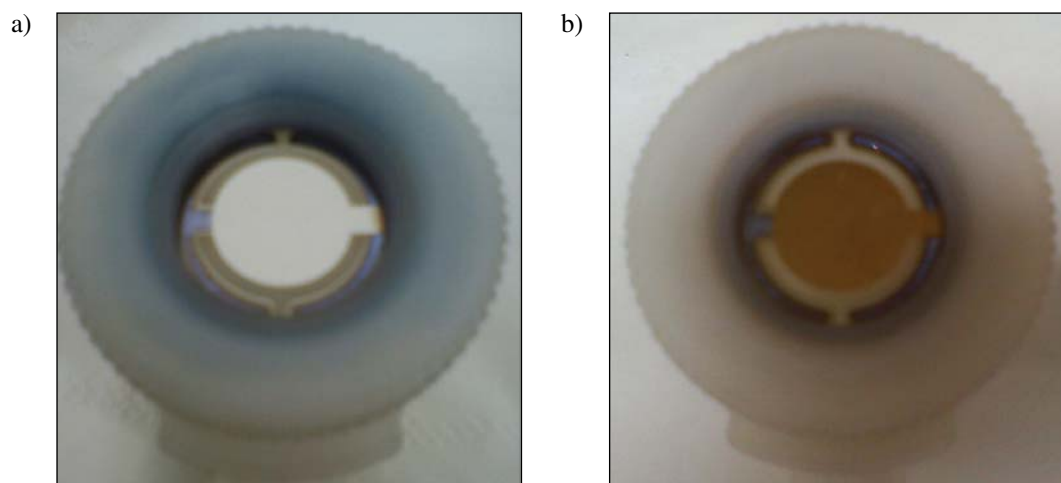
Figure 4. The picture of the platinum electrode before (a) and after the deposition of poly(5-aminoindole) film (b).
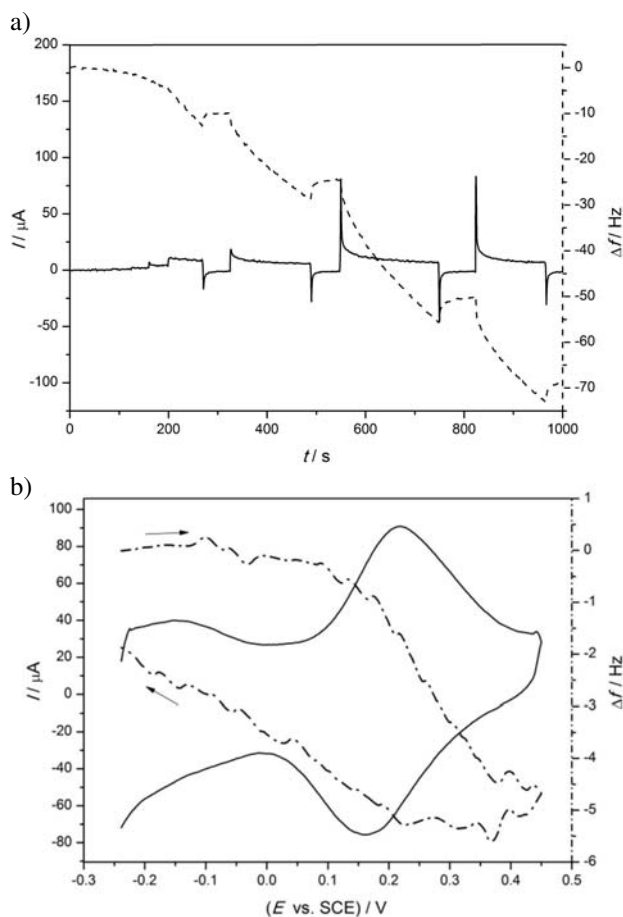
a)

b)

c)

d)
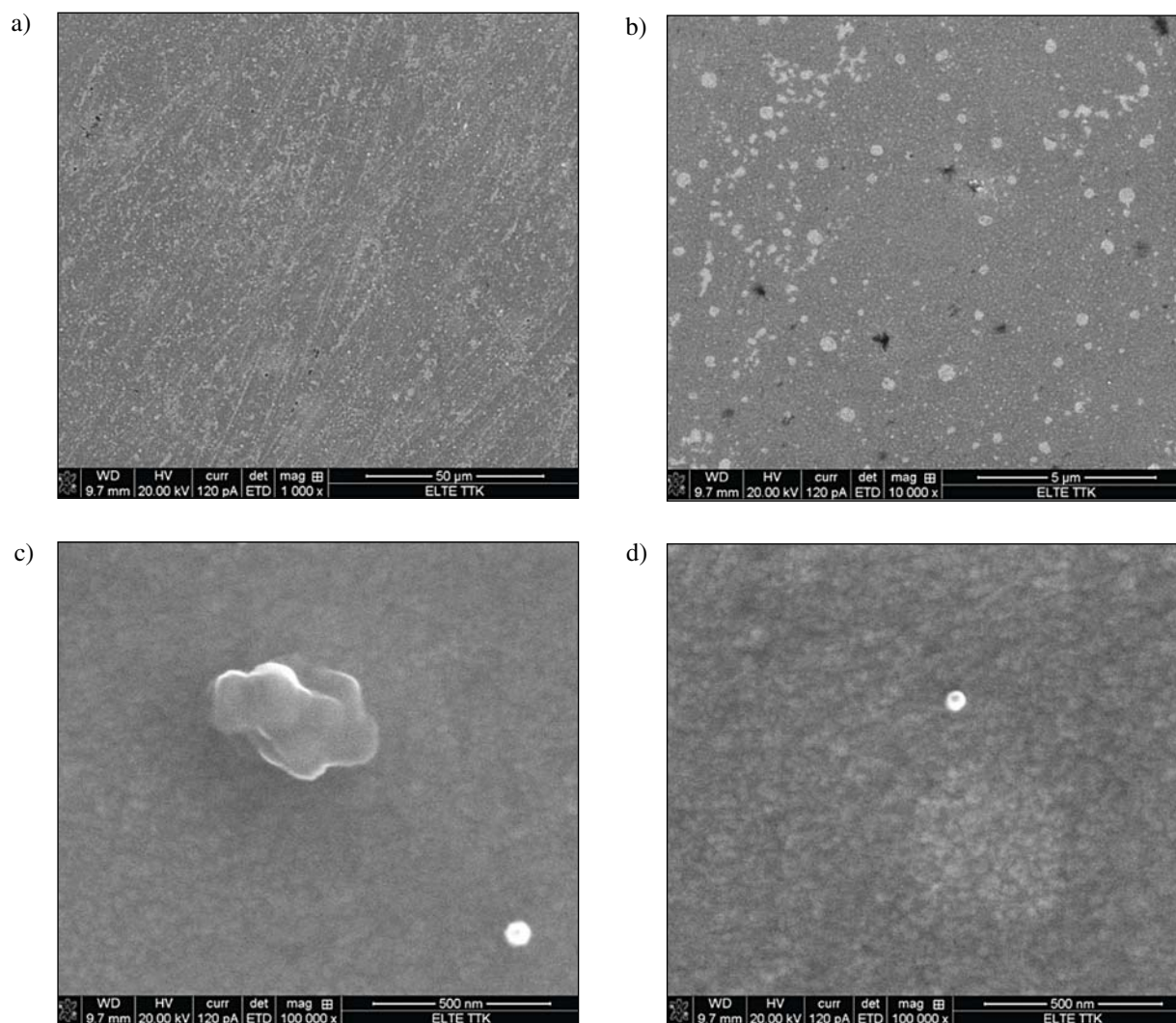
Figure 5. SEM/FIB images of a poly(5-aminoindole) film. Magnifications are: 1 000 (a), 10 000 (b), and 100 000 (c) and (d), respectively. Acceleration voltage: 20 kV, current: 120 pA.

The deposited poly(5-aminoindole) film is yellow and macroscopically uniform as seen in Figure 4. The SEM/FIB images also attest that the film is uniform (Fig.5), albeit granules of 100–200 nm can also be seen in the surface (Figure 5c). Figures 5c and 5d were taken from different spots of the same film.

Similar polymer film can be obtained by using potentiostatic deposition (Figure 6a). It is possible to check the cyclic voltammetric and EQCN response of the polymer formed after each step providing that the positive potential limit is below the oxidation potential of 5-aminoindole (Figure 6b).

It can be seen in Figure 6 that the rate of the polymer formation and deposition increases with increasing potential as expected. A step back to 0 V, i.e., reducing the polymer after an oxidation 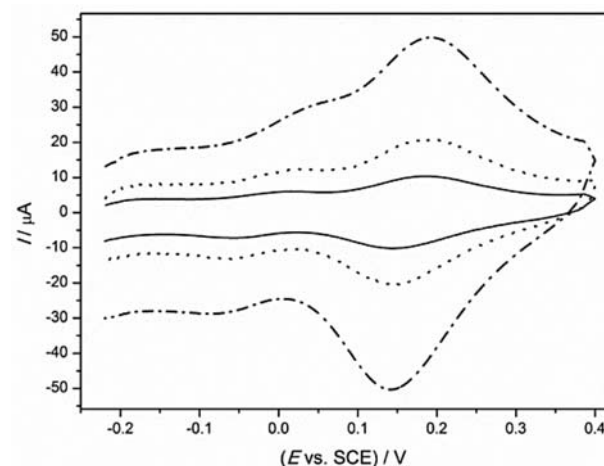period, results in the increase of frequency which is due to the redox response of the polymer. The magnitude of this frequency increase is proportional to the growing film thickness. However, the frequency response in the case of both the potential step and the cycling experiments is smaller than expected for an anion desorption during reduction. The same result can be obtained in the case of mass increase during the polymer oxidation. It follows that the anionic charge transport is not dominating in the charge-compensating process, and it is reasonable to assume that the hydrogen ions also participate, i.e., deprotonation and protonation accompanying the redox transformations.

## 3. 2. The Investigation of Poly(5-aminoindole) Films in Supporting Electrolytes

The polymer films shows a surface cyclic voltammetric response as a function of the scan rate (*v*) (Figure 7), since the peak currents linearly depend on *v*.

a)

b)

Figure 6. Formation and deposition of poly (5-aminoindole) on Pt at different potentials. (a) The current (continuous line) and the EQCN frequency (dashed line) responses following potential steps, (b) The cyclic voltammetric (continuous line) and EQCN (dotted dashed line) responses of the film formed after 9th stepping (due to the positive potential limit of 0.45 V further polymerization is prevented). Electrolyte: 5 mmol dm^{-3} 5-aminoindole in 0.5 mol dm^{-3} H$_2$SO$_4$. The potential steps were as follows: 0.48 V (160 s), 0.5 V (200 s), 0.0 V (270 s), 0.5 V (325 s), 0.0 V (490 s), 0.52 V (550 s), 0.0 V (750 s), 0.52 V (825 s) and 0.0 V (968 s). Scan rate, *v* = 50 mV · s^{-1}.

Figure 7. The cyclic voltammetric curves obtained for a Pt|poly(5-aminoindole) film in contact with a solution containing 0.5 mol dm^{-3} H$_2$SO$_4$ solution at different scan rates: *v* = 25 (dotted dashed line), 10 (dotted line), 5 (continuous line) mV · s^{-1}.

A cyclic voltammogram and the simultaneously obtained EQCN frequency curve at a slow scan rate, i.e., close to the equilibrium situation, is shown in Figure 8. It can be seen that during the oxidation a frequency decrease (mass increase) can be observed, while during the reduction the respective mass decrease occurs in an almost reversible manner. However, the complete return to the initial frequency indicating the total re-reduction occurs after holding the potential at −0.2 V for several minutes. By using Equation (2), it becomes evident that the apparent molar mass value calculated for the exchange species with *n* = 1, *M* = 6.5 ± 1 g mol^{-1} is far from that of the molar mass of the anion.

It follows that excess positive charges in the film due to oxidation are compensated by the incorporation of
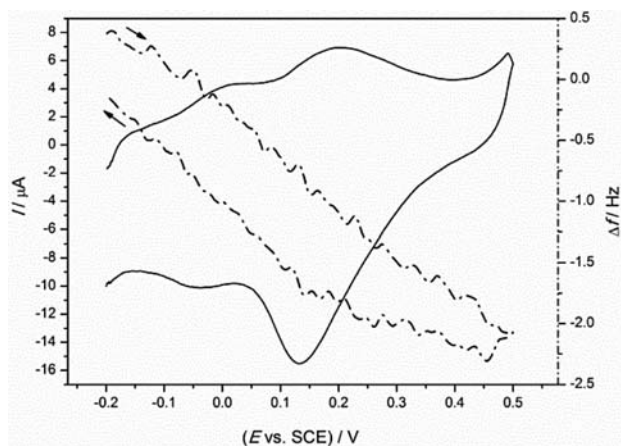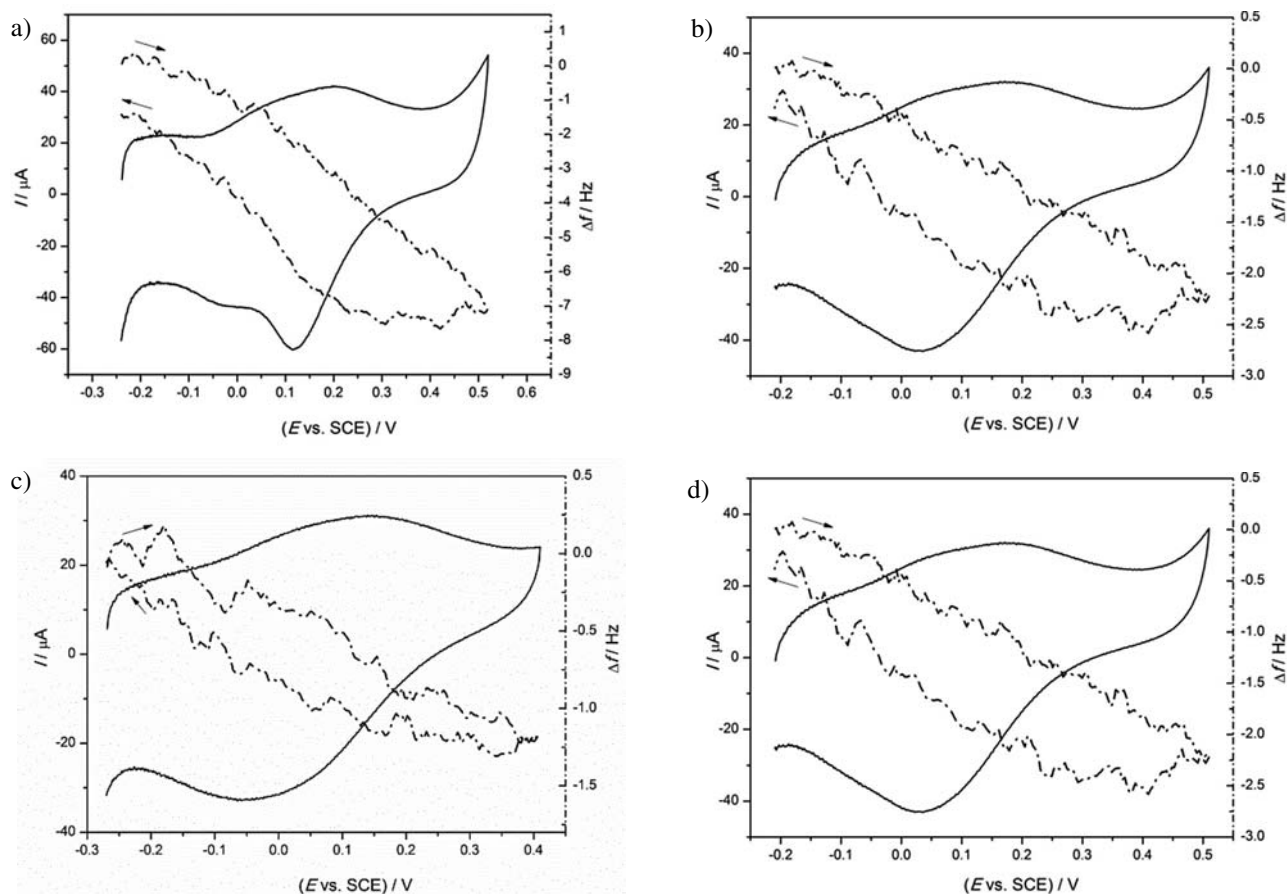
Figure 8. The cyclic voltammetric (continuous line) and the simultaneously detected EQCN (dotted dashed line) responses of a poly(5-aminoindole) film formed. Electrolyte: 0.5 mol dm^{-3} H$_2$SO$_4$. $v = 2$ mV·s^{-1}.

mass increase observed. Similar findings and explanations have been reported by Skompska et al.[39] in the case of poly(1,8-diaminocarbazole) in contact with aqueous solutions of HClO$_4$ assuming that counterions are already in the surface layer due to the protonation of the amino groups. This view is supported by the fact that in the case of unsubstituted indole, a mass change can be observed during the redox processes the magnitude of which is proportional to the molar mass of the counterions, i.e., an expected sorption / desorption occurs in the course of the oxidation and reduction, respectively.[40]

Because the deprotonation – protonation most likely accompanies the redox transformations of the polymer, the investigation of the pH dependence of the cyclic EQCN response is a logical step to prove the role of the H$^+$ ions in the electrode reaction. The cyclic voltammograms and the simultaneously obtained EQCN frequency curves are shown in Figure 9.

The participation of the protons (hydronium ions) is evidenced by the shift of the cyclic voltammograms as a function of pH. The 60 mV / pH shift indicates a H$^+$ / e$^-$ reaction.

the counterions only to a minor extent, the oxidation is accompanied by partial deprotonation, and the balance of these two charge transport processes causes the minor
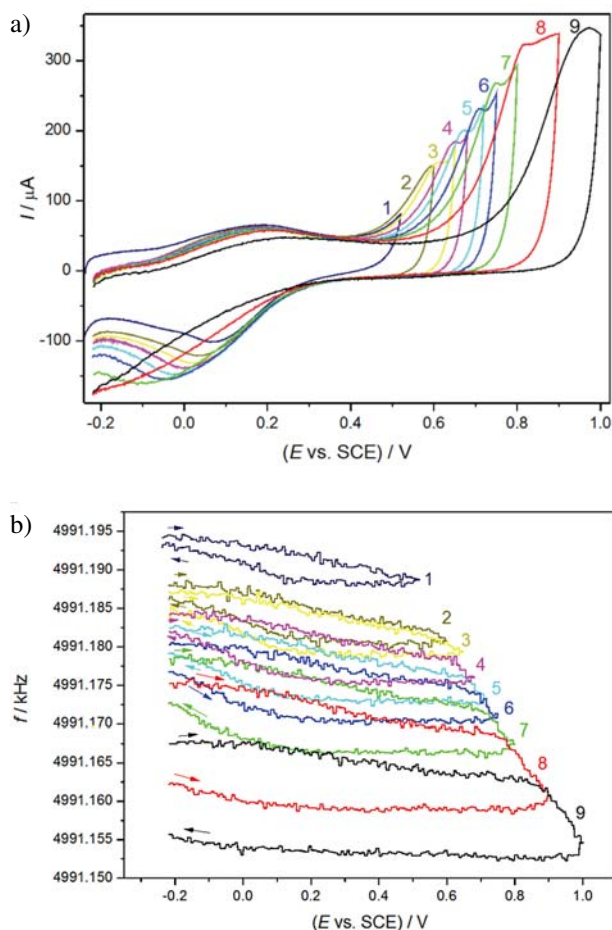

Figure 9. Cyclic voltammograms (continuous line) and the simultaneously detected EQCN frequency responses (dotted dashed line) obtained for a poly(5-aminoindole) film in contact with solutions of different pH values: Scan rate: 20 mV · s^{-1}. pH values: (a) –0.7 (5 M H$_2$SO$_4$), (b) 0 (1 M H$_2$SO$_4$), (c) 2 (H$_2$SO$_4$ – Na$_2$SO$_4$ buffer), (d) 7 (0.5 M Na$_2$SO$_4$).

It is of interest that the molar mass values calculated for the exchanged species by using Equation (2) show a systematic variation as a function of the pH of the solutions. The highest M value, $M = 5.5 \pm 3$ g mol^{-1} was obtained in the case of the very acidic solution, while it decreased to $M = 2.3 \pm 1$ g mol^{-1} at pH 7. It follows that at low pH values even the oxidized form of the poly(5-aminoindole) remains protonated, most likely the –NH of the indole ring, at least to a higher extent than at higher pH values.

3. 3. Effect of Overoxidation

It is known from the literature of heterocyclic compounds that indole is easily oxidized in presence of oxygen or other oxidizing agents in acid media.[38] Different compounds can be formed, among others hydroxyl and oxo derivatives or indigo and indirubin. Acid-catalysed

polymerization resulting in the formation of dimer and trimer products is also described in the organic chemistry literature.[38] Some of the differences in the electrochemical literature concerning the electropolymerization may be due to the presence of oxygen or to the too positive potential limit during cycling. Figure 10 shows a series of cyclic voltammograms (Figure 10a) and the respective EQCN frequency curves (Figure 10b) obtained for the same poly(5-aminoindole) film when the positive potential limit was gradually increased.

The reversible redox pair of wave gradually diminishes as the potential is cycled to more positive potentials. At higher potentials an irreversible voltammetric peak develops, however, this oxidation process also becomes increasingly hindered, i.e., it starts at more positive potentials. The peak current of the irreversible wave becomes higher and higher and is accompanied with a higher mass change. During continuous cycling until 1,0 V this peak current and the mass change decrease, and eventually the polymer loses its electrochemical activity. This effect is also seen when the 5-aminoindole was polymerized in the presence of $K_3[Fe(CN)_6]$ using a switching potential of 0.8 V (Figure 11). While at clean Pt or at a not overoxidized film the $[Fe(CN)_6]^{3-/4-}$ redox reaction occurs unhindered, as the overoxidized film grows on the Pt surface, this reaction gradually diminishes. It is also seen that the mass increase due to the formation of the surface film gradually decreases, too, and eventually stops.

a)

b)
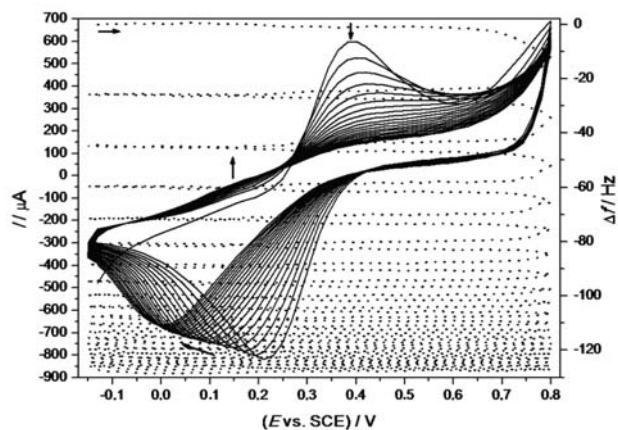
Figure 11. Cyclic voltammetric curves (continuous line) and the simultaneously obtained EQCN responses (dotted line) during electropolymerization of 5-aminoindole in the presence of $K_3[Fe(CN)_6]$. Solution composition: 50 mmol dm^{-3} $K_3[Fe(CN)_6]$ in pH=2 H_2SO_4 – Na_2SO_4 buffer. $v = 50$ mV·s^{-1}.

Figure 10. The effect of the positive potential limit of the cyclic voltammetric (a) and EQCN frequency (b) responses a Pt | poly(5-aminoindole) film in contact with a solution containing 5 mol dm^{-3} H_2SO_4 solution. $v = 50$ mV·s^{-1}. Switching potentials and the order of the experiments were as follows: 0.52 (1), 0.60 (2), 0.64 (3), 0.68 (4), 0.72 (5), 0.76 (6), 0.80 (7), 0.90 (8), and 1.00 V (9).
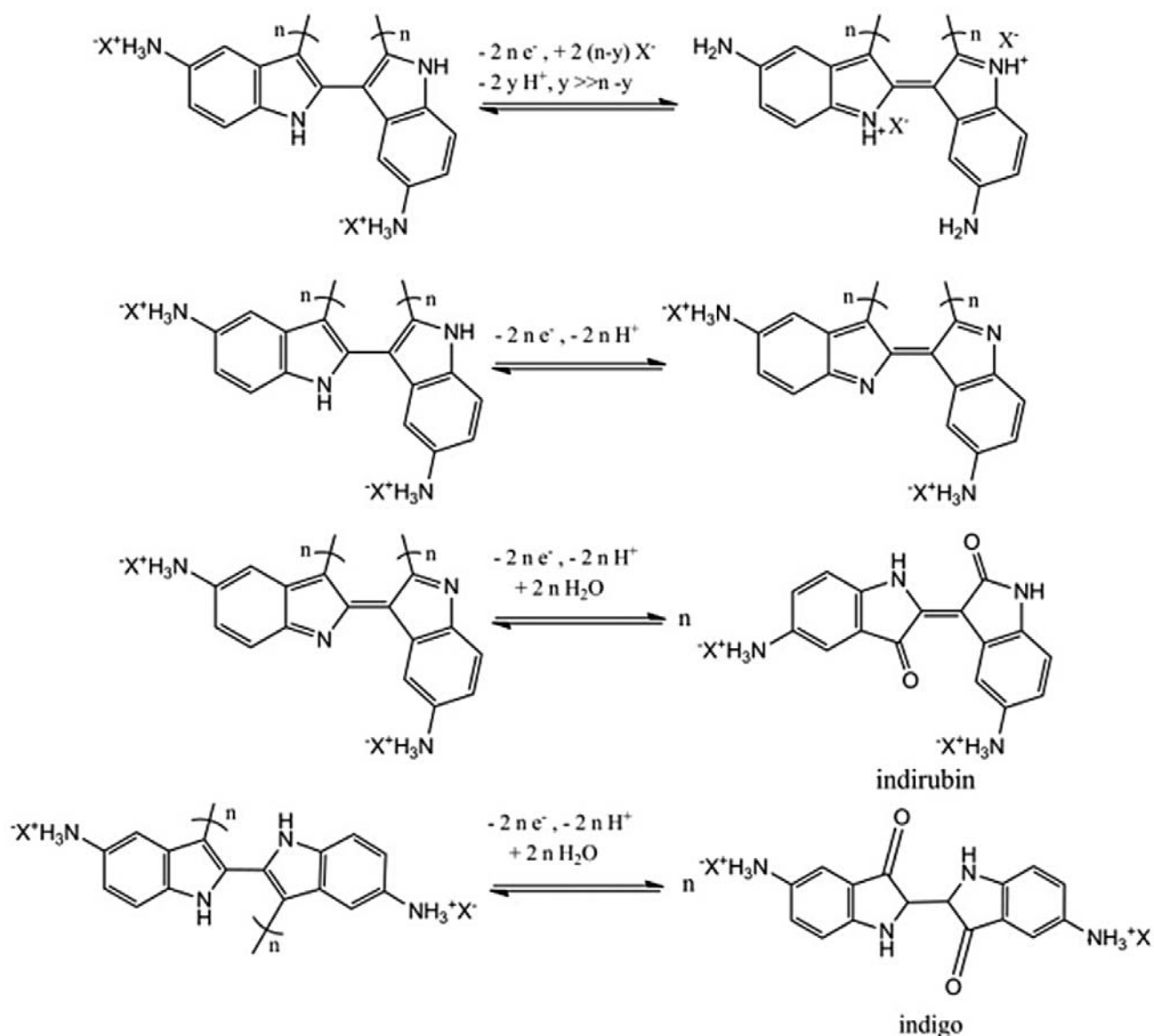
Nevertheless, a bluish-purple layer, which was formed, remained in the surface. Because of the loss of electrochemical activity, it can be concluded that the conjugated polymer bonds break down, and an indigo or indirubin-like insoluble, colored layer is formed.

Figure 12. Schemes of the redox transformations of poly(5-aminoindole) and the further oxidation reactions (overoxidation) at potentials higher than 0.62 V.

In summary, for the redox processes and the overoxidation reactions the following scheme (Figure 12) can be proposed:

4. Conclusions

Electrooxidation of 5-aminoindole at platinum in acid media below 0.62 V vs. SCE leads to the formation of a colored surface film of poly(5-aminoindole) which shows reversible redox activity. The redox transformations are accompanied with the exchange of hydronium ions, the participation of the anions in the charge-compensating process is minor and pH-dependent. It is a consequence of the protonation and salt formation of the amino group present in the compound. During electrooxidation deprotonation and expulsion of protons from the film maintain the electroneutrality conditions, while the counterions (anions) which are already present in the surface film compensate the excess charge due to the removal of electrons from the polymer by oxidation. At higher positive potentials a blue, non-electroactive film is formed. The overoxidation causes bond-ruptures and results in indigo-type, insoluble compounds that remain on the electrode surface.

5. Acknowledgement

6. Reference

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Povzetek

Z elektrokemijsko kvarčno tehtnico smo raziskali elektropolimerizacijo 5-aminoindola na platinski elektrodi v kisli raztopini. Z elektrooksidacijo 5-aminoindola, bodisi s potenciostatskim načinom ali s cikliranjem potenciala, se na platinski elektrodi tvori enakomerno, rumena polimerna plast, ki je elektrokemijsko aktivna. Predlagali smo shematski potek redoks transformacije poli(5-aminoindola), ki vključuje protonacijo-deprotonacijo ob prenosu elektrona. Pri visokih pozitivnih potencialih prihaja do nadaljnje oksidacije, pri čemer se tvori modro-vijočasta, indigu-podobna spojina, ki je sicer pritrjena na površino kovine ampak izkazuje majhno ali nobeno redoks aktivnost.