Electrochemical Characterization of Some Pyrrolo[1,2-c]Pyrimidine Derivatives

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The electrochemical characterization of two pyrrolo[1,2-c]pyrimidine derivatives (I1 and I2) was realized by cyclic, differential pulse, and rotating disk electrode voltammetric methods. The character of redox processes was established by scanning on different domains and with different scan rates. The diffusion coefficients were determined. Modified electrodes have been obtained by cycling the potential or by controlled potential electrolysis at anodic potentials. The electrodes obtained with polyI1 and polyI2 films were characterized by cyclic voltammetry in ferrocene solution.

Keywords: pyrrolo[1,2-c]pyrimidines, cyclic voltammetry, differential pulse voltammetry, rotating disk electrode voltammetry, modified electrodes

Pyrrolo[1,2-c]pyrimidine is a nitrogen containing heterocycle which is found in natural products and synthesized bioactive compounds. This class of compounds has been intensively studied [1-8] for its bioactivity (antimicrobial, antitubercular, antifungal, anticancer, antihistamine) and for the fluorescence applications [9-14]: solar cell dyes, organic light-emitting diode (OLED). Some pyrrolo-pyrimidine derivatives are used for their antiproliferative [15], anti-inflammatory and analgesic [16] activity, as well.

For the synthesis of pyrrolo[1,2-c]pyrimidine derivatives different procedures are reported in literature, starting from pyrrole [17, 18] or pyrimidine [19-22]. The 1,3-dipolar cycloaddition of the pyrimidinium N-ylides with acetylenic or olefinic dipolarophiles is the most used method. The multistep procedure which leads to pyrrolo[1,2-c]pyrimidines starts with the preparation of pyrimidinium salts. The second step of this procedure is the reaction between these salts and electronic-deficient alkenes or alkynes in the presence of a base, leading directly to pyrrolo[1,2-c]pyrimidines. The disadvantage of this procedure is the formation of inactivated products from the pyrimidinium-N-ylides [23-26]. In order to prevent their formation, a new one-pot three components procedure of synthesis using pyrimidine derivatives, substituted 2-bromo-acetophenones, and electron deficient alkynes has been reported. It takes place in the presence of an epoxide, which acts as reaction medium and acid scavenger [27, 28].

The investigation of the electrochemical properties of several pyrrolo[1,2-c]pyrimidines is of interest [29] due to the opportunity of using such compounds to obtain electrochemical sensors [29, 30]. This class of compounds also has remarkable fluorescent properties.

Experimental part

The new pyrrolo[1,2-c]pyrimidines were obtained by one-pot three components reaction, which involved mixing the reactants (substituted pyrimidine, 2-bromo-acetophenone and diethyl acetylenedicarboxylate or ethyl propiolate, respectively) in 1,2-epoxybutane as reaction medium [24-28]. The structure of the investigated compounds is presented in figure 1.

The electrochemical experiments were performed using a PGSTAT 12 AUTOLAB potentiostat connected to a three-electrode cell (containing acetonitrile and tetrabutylammonium perchlorate (TBAP) from Fluka as solvent and supporting electrolyte salt, respectively). A glassy carbon electrode disk (3 mm diameter) from Metrohm was used as working electrode. Its active surface was polished before each experiment with diamond paste (2 µm) and cleaned with acetonitrile. Ag/10 mM AgNO3 in 0.1 M TBAP, CH3CN was used as reference electrode, and a platinum wire served as an auxiliary electrode. All the potentials were finally referred to the potential of ferrocene/ferricinium redox couple (Fc/Fc+), which was +0.07 V in our experimental conditions. All electrochemical experiments were performed at room temperature (25ºC) under argon atmosphere. The cyclic voltammetry (CV) curves were recorded at various scan rates (0.1 – 1Vs-1), and the different pulse voltammetry (DPV) curves were recorded at 0.01 Vs-1 with a step time of 0.2s. Rotating disk electrode voltammetry (RDE) experiments were performed at 0.01 Vs-1.

![Fig. 1. Structure of investigated compounds](image_url)

<table>
<thead>
<tr>
<th>Compound</th>
<th>T1</th>
<th>T2</th>
</tr>
</thead>
<tbody>
<tr>
<td>R</td>
<td><img src="image_url" alt="Structure T1" /></td>
<td><img src="image_url" alt="Structure T2" /></td>
</tr>
</tbody>
</table>

*email: em_ungureanu2000@yahoo.com*
Results and discussions

The electrochemical characterization of the pyrrolo[1,2-c]pyrimidines $I_1$ and $I_2$ was performed by CV, DPV, and RDE at different concentrations in acetonitrile containing 0.1 M TBAP.

Electrochemical characterization of $I_1$

Figure 2 summarizes the CV and DPV curves at different concentrations of $I_1$ in 0.1 M TBAP, CH$_3$CN. They were obtained by dilution from the initial 0.75 mM solution which is the highest $I_1$ concentration in this system. The anodic and cathodic curves were recorded starting from the stationary potential.

DPV curves obtained at different concentrations show five oxidation peaks (denoted a$_1$ - a$_5$ in fig. 2) and four reduction peaks (denoted c$_1$ - c$_4$ in fig. 2), in order of their appearance in the anodic and cathodic scans, respectively. On CV curves only one anodic peak (a$_1$) is obvious, the others appear as shoulders. The three cathodic peaks c$_1$ - c$_3$ in DPV are seen as shoulders in CV. The notation of the peaks from the DPV curves was kept for all processes taking place at the corresponding potentials in the other methods. The CV and DPV currents are directly proportional with $I_1$ concentration. The linear dependences of CV and DPV peak currents on $I_1$ concentration [$I_1$] are shown in figure 2 insets for all peaks in DPV and for a$_1$, c$_1$, c$_2$, c$_3$ in CV. Their equations and their correlation coefficients are given in table 1 which contains the peaks with the best correlation coefficients for CV and DPV.

It can be seen that the first anodic peak (a$_1$) and all the cathodic peaks (c$_1$-c$_4$) have good correlation coefficients in DPV. The other peaks are less correlated. This behavior could be explained by occurrence of parallel irreversible electrochemical and chemical processes. a$_1$ slope is the highest, both in CV and DPV, this process being attributed to the formation of radical cation.

Table 1

<table>
<thead>
<tr>
<th>Method</th>
<th>Equation</th>
<th>Correlation coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>DPV</strong></td>
<td>$i_{peak \ a_1} = -0.82 + 22.67 \cdot [I_1]$</td>
<td>0.956</td>
</tr>
<tr>
<td></td>
<td>$i_{peak \ a_2} = 2.87 + 14.33 \cdot [I_1]$</td>
<td>0.848</td>
</tr>
<tr>
<td></td>
<td>$i_{peak \ a_3} = 5.50 + 13.98 \cdot [I_1]$</td>
<td>0.873</td>
</tr>
<tr>
<td></td>
<td>$i_{peak \ a_4} = -4.89 + 9.57 \cdot [I_1]$</td>
<td>0.539</td>
</tr>
<tr>
<td></td>
<td>$i_{peak \ a_5} = -0.08 + 10.40 \cdot [I_1]$</td>
<td>0.874</td>
</tr>
<tr>
<td></td>
<td>$i_{peak \ a_6} = -7.39 + 10.52 \cdot [I_1]$</td>
<td>0.596</td>
</tr>
<tr>
<td><strong>CV</strong></td>
<td>$i_{peak \ c_1} = 1.99 + 65.16 \cdot [I_1]$</td>
<td>0.952</td>
</tr>
<tr>
<td></td>
<td>$i_{peak \ c_2} = -14.11 + 16.39 \cdot [I_1]$</td>
<td>0.599</td>
</tr>
<tr>
<td></td>
<td>$i_{peak \ c_3} = -18.32 + 25.64 \cdot [I_1]$</td>
<td>0.859</td>
</tr>
</tbody>
</table>

*peak is expressed in µA, and $[I_1]$ in mM.

Figure 3 shows the anodic and cathodic RDE curves at different rotation rates. In order to establish a correspondence between the processes seen in RDE curves and the peak processes seen in DPV, anodic and cathodic RDE curves were put together with the DPV curve (for the same concentration). RDE cathodic curves obtained for $I_1$ in 0.1 M TBAP, CH$_3$CN at different rotation rates are regular. Globally, two waves are seen in the anodic domain, then the currents suddenly drop after reaching the potential of the peak a$_4$. The shape of RDE curves confirms the coverage of the electrode with an insulating layer, in the domain of anodic potentials (at potentials more positive than a$_4$).

Currents are increasing with the rotation rates of RDE. One isosbestic point can be seen for RDE curves at the potential of 1.547 V, which corresponds to an inversion of the current values obtained at different rotation rates.
Starting from this potential (which corresponds to the peak a4 in DPV), the current gets lower to very small values and remains constant over a potential range of about 1 V.

In order to study the processes reversibility, CV curves were recorded at different scan rates (0.1-1 Vs⁻¹) for the first anodic peak a1 and for the first cathodic peak c1 (fig. 4). The cathodic peaks became more evident with the increase of the scan rate. All the current values increase with the scan rate. Linear dependences of the peak currents on the square root of the scan rate are obtained for a1, c1 and c2 with slopes given on the figure 4b.

Figure 5 shows the CV curves (0.1 Vs⁻¹) obtained on different potential domains. The anodic processes are irreversible, while the cathodic ones are mainly quasi-reversible.

Table 2 shows the anodic and cathodic potentials for CV and DPV peaks at and their characteristics issues from the influence of the scan rate and scan domain.

Table 2

<table>
<thead>
<tr>
<th>Peak</th>
<th>Method</th>
<th>Process characteristics</th>
</tr>
</thead>
<tbody>
<tr>
<td>a1</td>
<td>DPV</td>
<td>0.857 0.931 irreversible</td>
</tr>
<tr>
<td>a2</td>
<td>CV</td>
<td>- 1.044 irreversible</td>
</tr>
<tr>
<td>a3</td>
<td>-</td>
<td>1.226 1.293 irreversible</td>
</tr>
<tr>
<td>a4</td>
<td>-</td>
<td>1.418 1.387 -</td>
</tr>
<tr>
<td>a5</td>
<td>-</td>
<td>1.329 1.943 -</td>
</tr>
<tr>
<td>c1</td>
<td>-</td>
<td>-1.947 -1.992 irreversible</td>
</tr>
<tr>
<td>c2</td>
<td>-</td>
<td>-2.144 -2.207 reversible</td>
</tr>
<tr>
<td>c3</td>
<td>-</td>
<td>-2.436 -2.518 reversible</td>
</tr>
<tr>
<td>c4</td>
<td>-</td>
<td>-2.794 - -</td>
</tr>
</tbody>
</table>

Polymerization conditions for compound I1

PolyI1 modified electrodes have been prepared from 0.5 mM solution of I1 in 0.1 M TBAP, CH3CN by successive potential scans between -0.3V and different anodic limits (fig. 6). The modified electrodes were transferred in the ferrocene solution (1mM) in 0.1 M TBAP/CH3CN and their CV curves were recorded and compared to the ferrocene signal on bare electrode (fig. 6f). The signal of ferrocene for the modified electrode prepared by scanning up to 2.02 V is much more diminished than those for other limits. This shows the electrode coverage is much more effective as a result of the process which occurs at this potential. The resulting film by covers much better the electrode.

Modified electrodes have been prepared also by controlled potential electrolysis (CPE). Figure 7a presents the comparison between the CV curves for the modified electrodes prepared by CPE at different potentials and keeping a constant charge. In figure 7b it can be seen that for films of 2 mC the ferrocene current are constant both for the anodic (ipa) and cathodic (ipc) peaks (fig. 7b). However, at higher applied potentials (2.35 V) there is an
important decrease of ferrocene anodic and cathodic peak potentials (Epa and Epc, respectively), and also on the formal (Ef) potential (fig. 7c).

Higher charges 4 mC and 6 mC have been used for CPE in order to check the increase of the film. Figures. 7d, 7e (fig. 7 f). The ferrocene signal for the modified electrodes obtained at 2 mC for all anodic CPE potentials is close to that to the bare electrode (fig. 7b); this indicates the formation of a thin conducting film. For bigger charges of 4 and 6 mC, the ferrocene signal is more diminished, indicating the fact that thicker films are formed. As the charge is bigger (6 mC), the decrease is more evident. The pulse duration is also important in the film formation (table 3). It can be seen that for the CPEs of 6 mC the pulse at 2.02 V lasts longer (371 s) than the pulse at 2.35 V (66 s). That is why the best coverage of the electrode has been obtained at 2.02 V for a charge of 6 mC, when the time for film formation is bigger.

**Electrochemical characterization of I2**

The CV and the DPV curves obtained at different concentrations of I2 (0-0.75 mM) are shown in figure 8. The DPV curves at different concentrations present six anodic peaks denoted with a1-a6 and three cathodic peaks (c1-c3), in order of their appearance in the voltammograms. The CV curves show five anodic processes denoted in connection to the DPV peaks (a1 - a5) and four cathodic processes denoted in connection to DPV cathodic peaks (c1-c4).

The currents increase with concentration. The linear dependences of the main peak currents on I2 concentration for the CV and DPV curves are presented. The equations of the main peak currents on I2 concentration and their correlation coefficients are given in table 4. The slopes obtained for I2 (table 4) dependencies are smaller than those for I1 (table 1). It can be explained by a steric hindrance of the two methoxy groups in I2, and it is not correlated with their molar masses (M I1 < M I2).

RDE curves at different rotation rates (500-2000 rpm) for I2 are presented in figure 9 in comparison with the DPV anodic and cathodic curves. They show two waves in the anodic domain of the peaks a1 and a5 in DPV. The currents increase with the rotation rate. After the peak a5 the current drops suddenly. This behavior is characteristic for the electrode coverage with insulating films. The isosbestic point (at about 2 V) is less marked for I2. Also the passive domain is much shorter (0.2 V) than for I1 (1 V). In the cathodic domain the waves are difficult to be separated. These facts show that the film formed from I2 after a5 potential is more porous than the corresponding one in case of I1.
Table 4

EQUATIONS AND CORRELATION COEFFICIENTS FOR THE DEPENDENCES ON $I_2$ CONCENTRATION OF CV AND DPV PEAK CURRENTS

<table>
<thead>
<tr>
<th>Method</th>
<th>Equation</th>
<th>Correlation coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>DPV</td>
<td>$i_{\text{peak c1}} = -1.38 - 15.69 \cdot [I_2]$</td>
<td>0.928</td>
</tr>
<tr>
<td></td>
<td>$i_{\text{peak c2}} = 0.82 + 4.23 \cdot [I_2]$</td>
<td>0.903</td>
</tr>
<tr>
<td></td>
<td>$i_{\text{peak c3}} = 0.98 + 13.13 \cdot [I_2]$</td>
<td>0.930</td>
</tr>
<tr>
<td></td>
<td>$i_{\text{peak d1}} = 0.98 - 12.80 \cdot [I_2]$</td>
<td>0.994</td>
</tr>
<tr>
<td></td>
<td>$i_{\text{peak c2}} = 0.03 - 8.26 \cdot [I_2]$</td>
<td>0.999</td>
</tr>
<tr>
<td>CV</td>
<td>$i_{\text{peak c1}} = -0.03 - 41.03 \cdot [I_2]$</td>
<td>0.961</td>
</tr>
<tr>
<td></td>
<td>$i_{\text{peak c2}} = 9.01 + 59.28 \cdot [I_2]$</td>
<td>0.925</td>
</tr>
<tr>
<td></td>
<td>$i_{\text{peak c3}} = 150.47 + 85.58 \cdot [I_2]$</td>
<td>0.994</td>
</tr>
<tr>
<td></td>
<td>$i_{\text{peak c1}} = -6.39 - 24.57 \cdot [I_2]$</td>
<td>0.909</td>
</tr>
<tr>
<td></td>
<td>$i_{\text{peak c2}} = -6.39 - 29.33 \cdot [I_2]$</td>
<td>0.999</td>
</tr>
<tr>
<td></td>
<td>$i_{\text{peak c3}} = -5.6 - 35.05 \cdot [I_2]$</td>
<td>0.999</td>
</tr>
<tr>
<td></td>
<td>$i_{\text{peak c4}} = -9.01 + 51.22 \cdot [I_2]$</td>
<td>0.953</td>
</tr>
</tbody>
</table>

Figure 11. CV curves (0.1 V s$^{-1}$) on different potential domains on glassy carbon electrode (3 mm diameter) for $I_2$ (0.5 mM) in 0.1M TBAP, CH$_3$CN

Table 5

EQUATIONS AND CORRELATION COEFFICIENTS FOR CV CURVES AT DIFFERENT SCAN RATES

<table>
<thead>
<tr>
<th>Peak</th>
<th>Equation</th>
<th>Correlation coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>d</td>
<td>$i_{\text{peak a}} = -2.56 + 21.99 \cdot [I_2]$</td>
<td>0.997</td>
</tr>
<tr>
<td>a1</td>
<td>$i_{\text{peak c1}} = -7.65 + 98.69 \cdot [I_2]$</td>
<td>0.949</td>
</tr>
<tr>
<td>d'</td>
<td>$i_{\text{peak c1}} = -2.42 - 11.29 \cdot [I_2]$</td>
<td>0.941</td>
</tr>
<tr>
<td>c1</td>
<td>$i_{\text{peak c1}} = -0.38 - 57.68 \cdot [I_2]$</td>
<td>0.998</td>
</tr>
</tbody>
</table>

*: $i_{\text{peak}}$ is expressed in $\mu$A and $v$ is the scan rate (V s$^{-1}$)

Table 6

PEAK POTENTIAL VALUES (V) FROM CV AND DPV CURVES vs FC/FC$^+$ FOR $[I_2] = 0.5$ mM

<table>
<thead>
<tr>
<th>Peak</th>
<th>Method</th>
<th>Process characteristics</th>
</tr>
</thead>
<tbody>
<tr>
<td>a1</td>
<td>DPV</td>
<td>0.871</td>
</tr>
<tr>
<td>a2</td>
<td>DPV</td>
<td>1.044</td>
</tr>
<tr>
<td>a3</td>
<td>DPV</td>
<td>1.209</td>
</tr>
<tr>
<td>a4</td>
<td>DPV</td>
<td>1.522</td>
</tr>
<tr>
<td>a5</td>
<td>DPV</td>
<td>1.830</td>
</tr>
<tr>
<td>a6</td>
<td>DPV</td>
<td>2.258</td>
</tr>
<tr>
<td>c1</td>
<td>CV</td>
<td>-2.182</td>
</tr>
<tr>
<td>c2</td>
<td>CV</td>
<td>-2.449</td>
</tr>
<tr>
<td>c3</td>
<td>CV</td>
<td>-2.516</td>
</tr>
<tr>
<td>c4</td>
<td>CV</td>
<td>-2.914</td>
</tr>
</tbody>
</table>

CV curves for $I_2$ on different domains (0.1 V s$^{-1}$) are presented in figure 11. The processes associated with the curves and their characteristics are shown in table 6. The cathodic peaks are quasi-reversible.

Polymerization conditions for compound $I_2$

The preparation of modified electrodes based on $I_2$ was done in a similar way as for $I_1$ by successive scans (fig. 12), or by CPE at different anodic potentials or charges (fig. 12).
13). The ferrocene signals for the modified electrode obtained at 6 mC and 8 mC are diminished. The decrease is more significant at 2.14 V because the pulse time is bigger at this potential and there is more time for the polymer formation (table 7), as in the case of I1. The electrodes modified with charges of 0.3 mC, 0.8 mC, 1 mC and 1.1 mC are practically not covered by films.

Comparison between I1 and I2

From figure 14 and table 8 it can be observed that both compounds are oxidized at about 0.87 V, and I1 is reduced at less negative potentials than I2, as expected taking into account their structures (for I2 two methoxy groups have a electron donating effect and I2 is more difficult to be reduced).

From the dependences of the a1 peak currents on the square root of the scan rate (fig. 5 (b) and table 5) the diffusion coefficients have been calculated for the CV curves recorded at room temperature (298 K), using Randles–Sevcik equation (1), where \( i_p \) = peak current in A, \( n \) = number of electrons transferred in the redox process (is considered 1), \( A \) = electrode area in cm², \( D \) = diffusion coefficient in cm²/s, \( C \) = concentration in mole/cm³, and \( v \) = scan rate in V/s. The results are summarized in table 9.

\[
i_p = \frac{2.686 \times 10^{5} \times n^{3/2} \times A \times C \times D^{1/2} \times \sqrt{v}}{R \times T}
\]  

Several differences occur in the preparation of modified electrodes prepared from I1 and I2 (fig. 15) either by CPE or scanning. It can be seen that the transfer of the modified electrodes modified with charges of 0.3 mC, 0.8 mC, 1 mC and 1.1 mC are practically not covered by films.

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<table>
<thead>
<tr>
<th>Compound</th>
<th>E a1 (V)</th>
<th>E c1 (V)</th>
</tr>
</thead>
<tbody>
<tr>
<td>I1</td>
<td>0.867</td>
<td>-1.947</td>
</tr>
<tr>
<td>I2</td>
<td>0.871</td>
<td>-2.182</td>
</tr>
</tbody>
</table>
electrodes (in 1 mM ferrocene solution) obtained by CPE at the same charge (6 mC) and at close potentials (2.02 V for compound I1, and 2.14 V for compound I2), the signal for ferrocene is more flat for I1, indicating a less conducting film (fig. 15a). The electrode seems to be better covered by I1 than I2. However, when the films are prepared by cycling the differences are less significant (fig. 15b). This behavior could be explained by the fact that the preparation of the modified electrodes takes longer by scanning (than by CPE) for both I1 and I2, and the polymerization step can occur better.

Pyrrolo[1,2-c]pyrimidine is a complicated redox system, due to its relatively low aromatic character, which confers it a high reactivity in comparison to the common aryls. The difficulty of the peak assessment is increased by the presence of stabilizing functional groups, which have redox potentials close to the aromatic moiety (table 10).

An important description of the electrochemical processes have been already given by our group in 2012. [29]. However, the change of substituents leads to important changes of their electrochemical behaviour. For example, an extra OMe group attached to R = alkoxycphenyl reduces the oxidation potentials with about 0.1 V, but does not affect the reduction potentials, which remain at about 1.94 V. When two dimethoxybenzene units are present, as in I2, they oxidize to ortho-quinones at different potentials, as it can be seen by the increase of the number of peaks in DPV curves for this compound. When the extra methoxy group is placed on the benzoyl substituent, the reduction potential is increased by 0.25 V to -2.18 V. These data prove that the keto group is firstly involved into system reduction to alcohol, followed by the heterocycle moiety reduction. The reducing steps of the pyrrolo[1,2-c]pyrimidines are quasi-reversible due to the partial opening of the pyrrole ring.

Conclusions
The new pyrrolo[1,2-c]pyrimidines have been characterized by electrochemical methods. By cycling the potential in different anodic domains or by controlled potential electrolysis at anodic potentials poly I1 and poly I2 films were deposited on to the electrodes. The modified electrodes were characterized in ferrocene solution in 0.1 M TBAP, CH3CN. A decrease of Fc/Fc+ current has been seen, confirming the electrode coverage by films.

Further studies will be directed to investigations of poly I1 and poly I2 modified electrodes in view of their direct relevance in practical applications.

Acknowledgements: The authors are grateful for the financial support from: Executive Unit for Financing Education Higher, Research Development and Innovation (UEFISCDI) project ID PN-II-RU-TE-2014-4-0594 contract no. 10/2015, PN-II-PT-PCCA-2013-4-2151 contract no. 236/2014, and Romania–China bilateral project 6B8M/2016 (CH 41.16.04).

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