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Voltammetric and Quantum Investigation of Selected Succinimides

Bojan Božić[1], *Jelena Lović*[2] , *Nebojša Banjac*[3], *Željko Vitnik*[4], *Vesna Vitnik*[4], *Dušan Mijin*[5], *Gordana Ušćumlić*[5], *Milka Avramov Ivić*[2,*]

[1] Institute of Physiology and Biochemistry, Faculty of Biology, University of Belgrade, Belgrade, Serbia
[2] Institute of Electrochemistry, University of Belgrade, Belgrade, Serbia
[3] Faculty of Agriculture, Food Technology and Biochemistry, University of Belgrade, Zemun, Serbia
[4] Department of Chemistry, ICTM, University of Belgrade, Belgrade, Serbia
[5] Department of Organic Chemistry, Faculty of Technology and Metallurgy, University of Belgrade, Belgrade, Serbia
[*]E-mail: milka@tmf.bg.ac.rs

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A series of succinimide derivatives were studied using the cyclic and square wave voltammetry. Density function theory was used in order to determinate which of the structural parameters influence the electrochemical activity. The quantum chemical calculations of the investigated succinimides were linked with the experimental electrochemical data and used to propose the oxidation mechanism. The most active among studied succinimides is 1,3-diphenylsuccinimide. The results obtained from the cyclic and square wave voltammetry and quantum chemical calculations indicate that the investigated compounds undergo oxidation by irreversible, diffusion controlled process including transfer of 1e^{-} and 1 proton. The voltammetric and DFT results signify that the mechanism of electrochemical oxidation of all compounds involve the conversion of carbonyl-methyne-phenyl segment or methylene group in free radical. This conversion proceeds by the loss of one proton one electron process.

Keywords: Diphenylsuccinimide, Threephenylsuccinimide, Voltammetry, Quantum chemical calculations, Reaction mechanisms

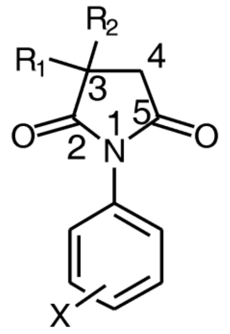
1. INTRODUCTION

Succinimides (cyclic imides) or molecules that contain succinimide ring exhibit a great variety of biological activities and pharmaceutical uses. They posses antimicrobial and antioxidant, analgesic, antifungal, antitumor, cytostatic, anorectic, antispasmodic, muscle relaxant, hypotensive, anticonvulsant and antiepileptic, and antitubercular properties [1-11]. Their synthesis and properties have been also reviewed [12, 13].

Recently, 1-aryl-3-phenyl-, 1-aryl-3-methyl- and 1-aryl-3,3-diphenylpyrrolidine-2,5-diones were investigated [14, 15]. The structural properties of the selected molecules were correlated with chromatographic data. These properties obtained by medicinal chemistry methods are related to the absorption, distribution, metabolism, elimination and toxicity. In view of these results, the investigated succinimides could be drug candidates and hence became the subject for the further investigations.

A number of nitrogen containing compounds have been electrochemicaly investigated and characterized [16, 17], but to the best of our knowledge, succinimides (heterocyclic "pyrrolidine-2,5-dione" derivatives) have not been subjected to such electrochemical studies.

So, in this work we investigated a series of succinimide derivatives (Fig. 1), using the cyclic voltammetry (CV) and square wave voltammetry (SWV). In order to determinate which of the structural parameters influence the electrochemical activity, density function theory (DFT) has been applied. The obtained DFT data were linked with the experimental electrochemical data and used to propose the oxidation mechanism of the examined succinimides.



No.	R_1	R_2	X
1	H	Ph	H
2	Ph	Ph	H
3	H	Ph	4-Cl
4	Ph	Ph	4-Cl
5	H	Ph	3-Cl
6	Ph	Ph	3-Cl

Figure 1. Structure of the investigated succinimides: **1** (1,3-diphenylsuccinimide) and **2 (**1,3,3-triphenylsuccinimide) as well as their chloro derivatives **3** (1-(4-chlorophenyl)-3-phenylsuccinimide), **4** (1-(4-chlorophenyl)-3,3-diphenylsuccinimide), **5** (1-(3-chlorophenyl)-3-phenylsuccinimide) and **6** (1-(3-chlorophenyl)-3,3-diphenylsuccinimide).

2. EXPERIMENTAL

2.1. Materials

All investigated compounds have been previously synthesized [18]. Sodium hydrogen carbonate and sulphuric acid were p.a. grade and obtained commercially from Sigma. 18 MΩ cm deionised water was obtained from a Milipore Waters Milli-Q purification unit.

2.2. Electrochemical experiments

Standard equipment PGZ 402 Volta Lab (Radiometer Analytical, Lyon, France) and the three electrode electrochemical cell were used as previously described [19,20]: polycrystalline gold working electrode (surface area 0.07 cm^2), a gold wire (counter electrode) and a saturated calomel electrode (SCE) as reference electrode. Gold electrode was prepared as previously [19] and cleaned between experiments. All experiments were done at room temperature.

2.3. Method of calculation

The DFT calculations were performed on the B3LYP [21] and M06-2X [22] level of theory using 6-311++G(d,p) basis set [23, 24] using Gaussian 09 molecular package [25]. Initial geometries are fully minimized with the default convergence criteria and without any constraint. The unrestricted approach was applied for a proper description of radicals and radical cations. The water solvent effects are introduced in calculations using conductor polarizable continuum model (CPCM) [26].

3. RESULTS AND DISCUSSION

3.1. Electrochemical characterization of the investigated succinimides

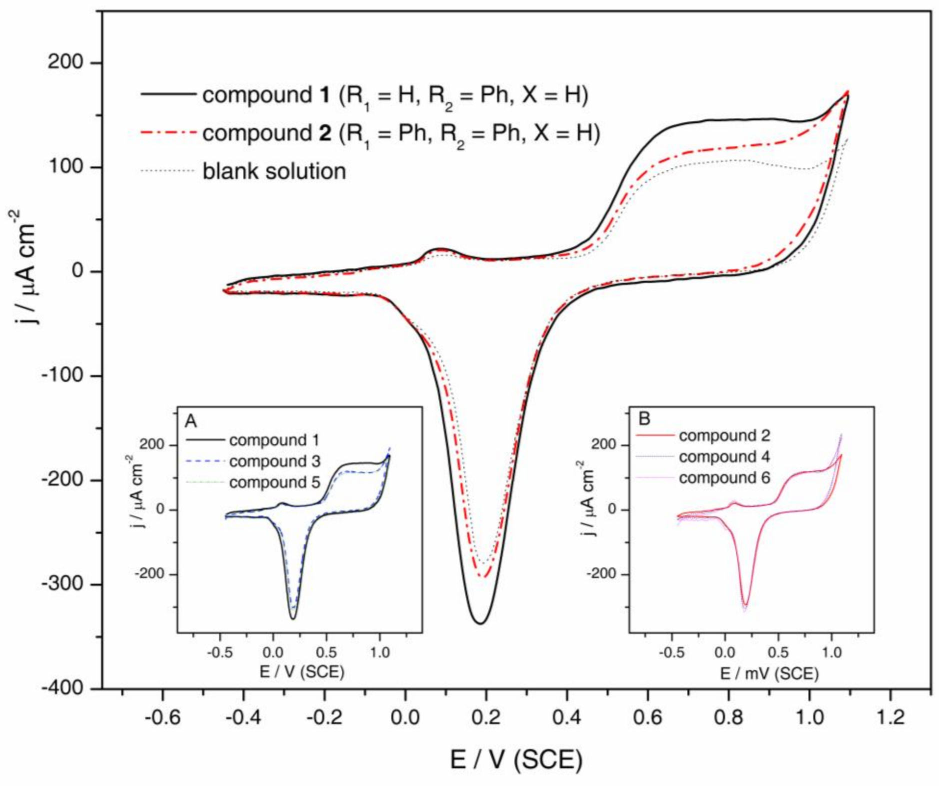


Figure 2. CVs of Au electrode (blank, dotted line) in 0.05 M $NaHCO_3$ and in the presence of compound **1** (full line) and compound **2** (dash-dotted line), c = 1 μM, v = 50 mV s^{-1}, inset A: compounds **1**, **3**, **5**. Inset B: compounds: **2**, **4**, **6**.

Electrochemical characterization of succinimides was realized using cyclic and square wave voltammetry. Figure 2 shows the recorded cyclic voltammograms of Au electrode in 0.05 M $NaHCO_3$

in the presence of succinimides **1** (1,3-diphenylsuccinimide) and **2** **(**1,3,3-triphenylsuccinimide). In the surface oxide formation/reduction region succinimides gave higher reaction currents compared to Au electrode in 0.05 M $NaHCO_3$ (dotted line in Fig. 2) indicating an electrochemical oxidation of succinimides on Au electrode. The most active among studied succinimides is compound **1**. It is clear from Fig. 2 that succinimide with two phenyl substituent attached to the heterocyclic system in position 3 (compound **2**) in comparison to compound **1** shows lower activity in the region of Au oxide formation and consequently, reduction. Its incipient potential appears at more positive value of the potential related to the compound **1**. Also it can be noticed (insets A,B in Fig. 2) that compounds with substituent on phenyl ring (compounds **3-6**) exhibit very similar electrochemical behaviour with almost overlapped oxidation peak indicating no influence of substituent on the phenyl ring on the electrochemical reaction.

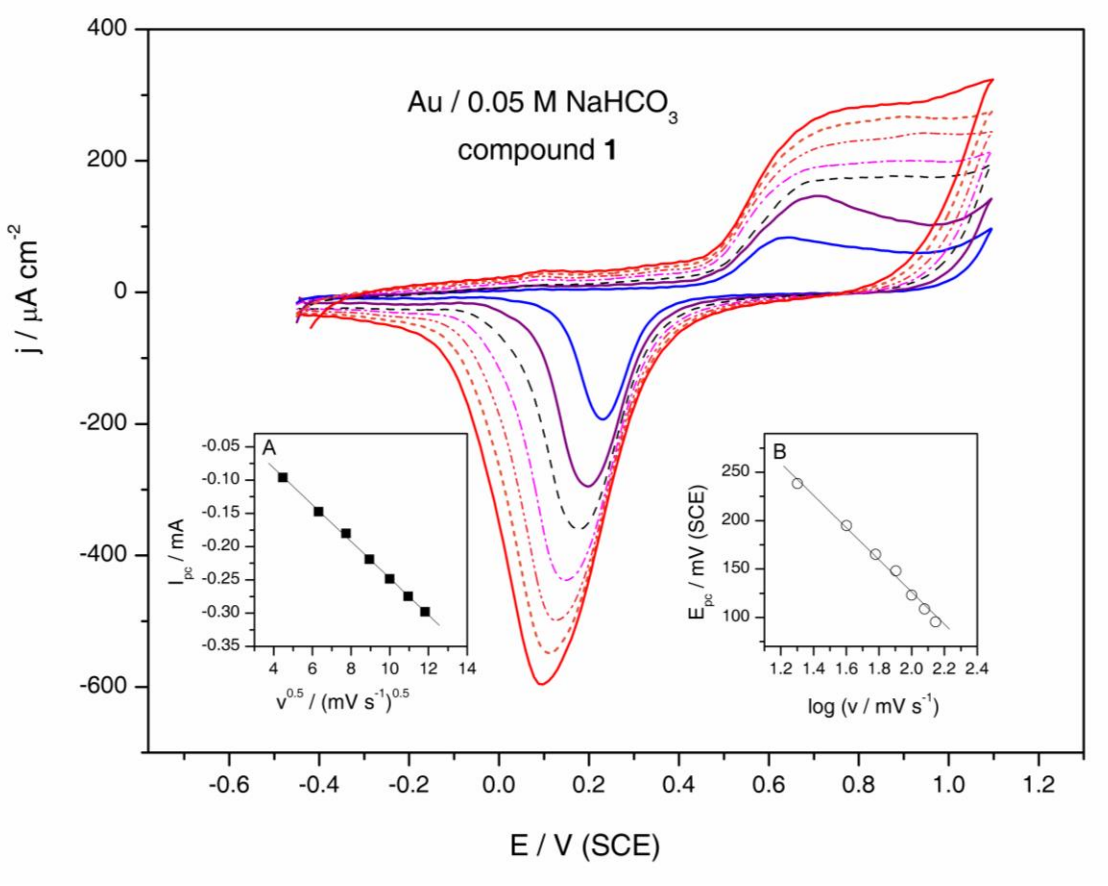


Figure 3. CVs of 1 μ M of compound **1** on Au electrode using 0.05 M $NaHCO_3$ for scan rates: 20, 40, 60, 80, 100, 120 and 140 mV s^{-1}. Insets: plots of peak current vs. $v^{0.5}$ (A) and peak potential shift vs. ln of scan rates (B).

The electrochemical behaviour of the all compounds was further tested by varying the scan rates and as representative and the most active one, the compound **1** was selected and presented in Fig. 3. The currents increase with the increased scan rate. In Fig. 3 the dependency of peak current on $v^{0.5}$ is presented (inset A) showing linearity signifying that the succinimide oxidation is diffusion controlled process [27]. The cathodic peak potential is shifted to the negative direction with the increasing scan rate, and a straight line correlation can be observed from the dependency of the catodic peak potentials on log of scan rates (inset B), suggesting that the succinimide oxidation is an irreversible electrode process [27]. The Laviron's theory for irreversible processes was employed in order to calculate the heterogeneous electron-transfer rate constant (k_s) as is presented in Table 1 [28]. It can be noticed that ks obtained on electrode in the presence of compound **1** is more than two times greater than with compound **6** (1-(3-chlorophenyl)-3,3-diphenylsuccinimide) indicating higher ability for promoting the transfer of electrons between succinimide and the electrode surface. Compounds **2**, **3** (1-(4-chlorophenyl)-3-phenylsuccinimide) and **5** (1-(3-chlorophenyl)-3-phenylsuccinimide) had similar and moderate value for k_s regardless to compound **1**, while the value for k_s of the compound **4** (1-(4-chlorophenyl)-3,3-diphenylsuccinimide) with para Cl substituent on phenyl ring is ~37% less than for compound **1**.

Table 1. Values of calculated electron transfer rate constant (k_s) for investigated succinimides

sample	1	2	3	4	5	6
k_s / s^{-1}	1.14	0.53	0.58	0.72	0.57	0.49

The same experiment, as was shown in Fig. 2 is performed in 0.1M H_2SO_4 at pH = 1 and it is presented in Fig. 4B. From Fig. 4B it is quite clear that acid environment inhibits in a greater extent the electrochemical activity of compound **1** suggesting the proton participation in electrooxidation process.

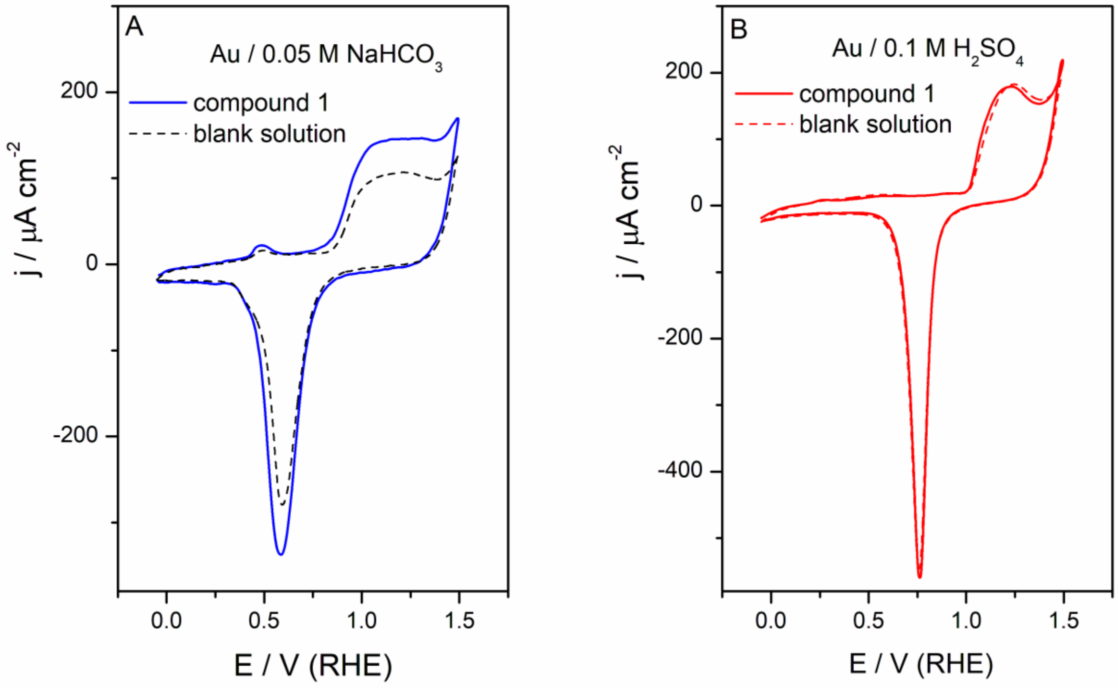


Figure 4. CVs of Au electrode in the presence of compound **1**, c = 1 μM, v = 50 mV s^{-1}, in 0.05 M $NaHCO_3$ (A), and in 0.1M H_2SO_4 (B).

In addition, the SWV signal of the gold electrode towards the electrochemical reaction of the all succinimides has been analyzed. The square-wave voltammograms of the succinimides **1** and **2** were selected and presented in Fig. 5. A voltammetric response for the oxidation of the succinimides **1** and **2** is established and as was shown by cyclic voltammetry, the compound **1** is most active. Also, the substituents on phenyl ring do not affect the electrochemical activity and the shape of their voltammetric signal is the same as is presented for compounds **1** and **2** but the activity is similar but lower.

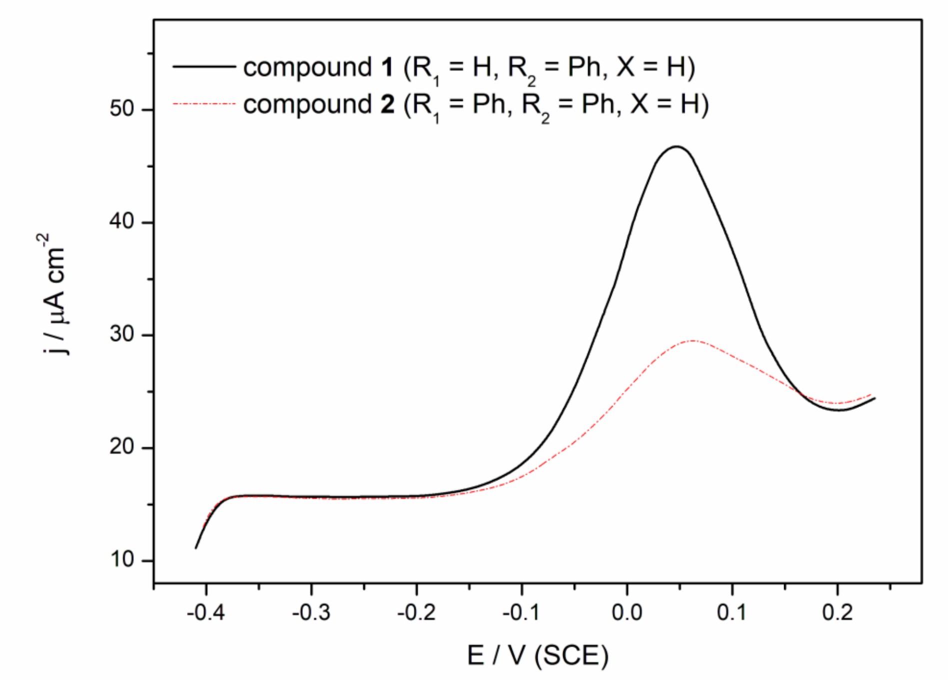


Figure 5. SWVs of compounds **1** and **2** (c =1 µ M) obtained with the Au electrode using 0.05 M $NaHCO_3$. Accumulation time: 0.2 s at E = - 0.45 V; step size 5 mV, pulse size 75 mV, frequency 10 Hz, scan rate 50 mV s^{-1}.

3.2. Computational study

The data and conclusions obtained by CV (Figs. 3 and 4) are used as one of the basis for the computational analysis. DFT B3LYP and M06-2X calculations with 6-311++G(d,p) basis set were performed in order to make comparison of the energy differences between systems which are involved in the oxidation mechanism. H_2O molecule was added to the energy of succinimide molecule (G) and to the corresponding radical cation ($R^{\bullet +}$) in order to keep systems on the same level of the energy. In addition, the energy of H_3O^+ ion was added to the energy of radical and anionic species.

From the Table 2 and Fig. 6, it is observed that monophenyl succinimide derivatives (**1**, **3**, **5**) can form two species of radical and anion after deprotonation at position C3 and C4. For these compounds (**1**, **3**, **5**) is characteristic that energetically the formation of radical and anion at position C3 is favored.

Table 2. Relative energies[a] [kcal/mol] of systems[b] involved in electrochemical oxidation mechanism for succinimides: radical cation ($R^{\bullet +}$), radical on C3 (RC3) and C4 (RC4) atoms, anion on C3

(AC3) and C4 (AC4) atoms and the energy differences for formation of RC3 and RC4 radicals from R^+, AC3 and AC4 species.

Compound **1**	B3LYP		M06-2X	
	Vacuum	Water	Vacuum	Water
$R^{\bullet+}$	189.88	155.47	201.19	166.35
RC4	240.97	169.22	241.29	169.37
RC3	225.58	154.55	228.10	156.66
AC4	188.39	75.97	186.56	72.56
AC3	173.19	63.86	172.37	61.50
$R^{\bullet+}$ – RC4	51.09	13.75	40.11	3.02
$R^{\bullet+}$ – RC3	35.69	–0.93	26.92	–9.69
AC4 – RC4	52.59	93.26	54.73	96.81
AC3 – RC3	52.39	90.69	55.73	95.16

Compound **2**	B3LYP		M06-2X	
	Vacuum	Water	Vacuum	Water
$R^{\bullet+}$	184.41	153.03	198.15	167.76
RC4	239.93	167.71	240.59	168.12
RC3	/	/	/	/
AC4	185.53	73.19	184.40	70.07
AC3	/	/	/	/
$R^{\bullet+}$ – RC4	55.53	14.68	42.44	0.36
$R^{\bullet+}$ – RC3	0.00	0.00	0.00	0.00
AC4 – RC4	54.40	94.53	56.20	98.05
AC3 – RC3	0.00	0.00	0.00	0.00

Compound **3**	B3LYP		M06-2X	
	Vacuum	Water	Vacuum	Water
$R^{\bullet+}$	189.94	155.77	201.55	166.89
RC4	240.76	169.17	241.42	168.95
RC3	225.46	154.45	228.06	156.48
AC4	183.97	74.66	182.35	71.30
AC3	169.30	62.80	168.94	60.33
$R^{\bullet+}$ – RC4	50.82	13.40	39.87	2.06
$R^{\bullet+}$ – RC3	35.52	–1.32	26.51	–10.41
AC4 – RC4	56.79	94.52	59.07	97.65
AC3 – RC3	56.16	91.66	59.12	96.15

Compound **4**	B3LYP		M06-2X	
	Vacuum	Water	Vacuum	Water
$R^{\bullet+}$	185.11	152.93	198.40	168.07
RC4	239.84	167.57	240.96	168.38
RC3	/	/	/	/
AC4	181.43	71.86	180.79	68.89
AC3	/	/	/	/
$R^{\bullet+}$ – RC4	54.73	14.64	42.55	0.31
$R^{\bullet+}$ – RC3	/	/	/	/
AC4 – RC4	58.41	95.71	60.17	99.49
AC3 – RC3	/	/	/	/

Compound **5**	B3LYP		M06-2X	
	Vacuum	Water	Vacuum	Water
$R^{\bullet+}$	191.74	156.67	203.71	173.08

RC4	240.83	169.17	241.48	169.25
RC3	225.48	154.43	228.43	156.85
AC4	183.99	74.14	182.42	70.82
AC3	169.26	62.52	168.81	60.37
$R^{\bullet+}$ – RC4	49.09	12.50	37.77	–3.84
$R^{\bullet+}$ – RC3	33.74	–2.25	–13.05	–16.23
AC4 – RC4	56.84	95.03	59.06	98.43
AC3 – RC3	56.22	91.91	59.62	96.49
Compound **6**	B3LYP		M06-2X	
	Vacuum	Water	Vacuum	Water
$R^{\bullet+}$	185.78	153.46	200.69	168.06
RC4	239.88	167.57	240.96	168.43
RC3	/	/	/	/
AC4	181.45	71.48	180.63	68.85
AC3	/	/	/	/
$R^{\bullet+}$ – RC4	54.10	14.12	40.27	0.37
$R^{\bullet+}$ – RC3	/	/	/	/
AC4 – RC4	58.42	96.10	60.32	99.58
AC3 – RC3	/	/	/	/

[a] $\Delta E^0 = E^0(S) - E^0(G)$; $E^0(G) = E(G) + ZPE$

[b] H_2O molecule was added to the energy of the optimized succinimide molecule and to the corresponding radical cation in order to keep systems on the same level the energy. In addition, the energy of H_3O^+ ion was added to the energy of radical and anionic species.

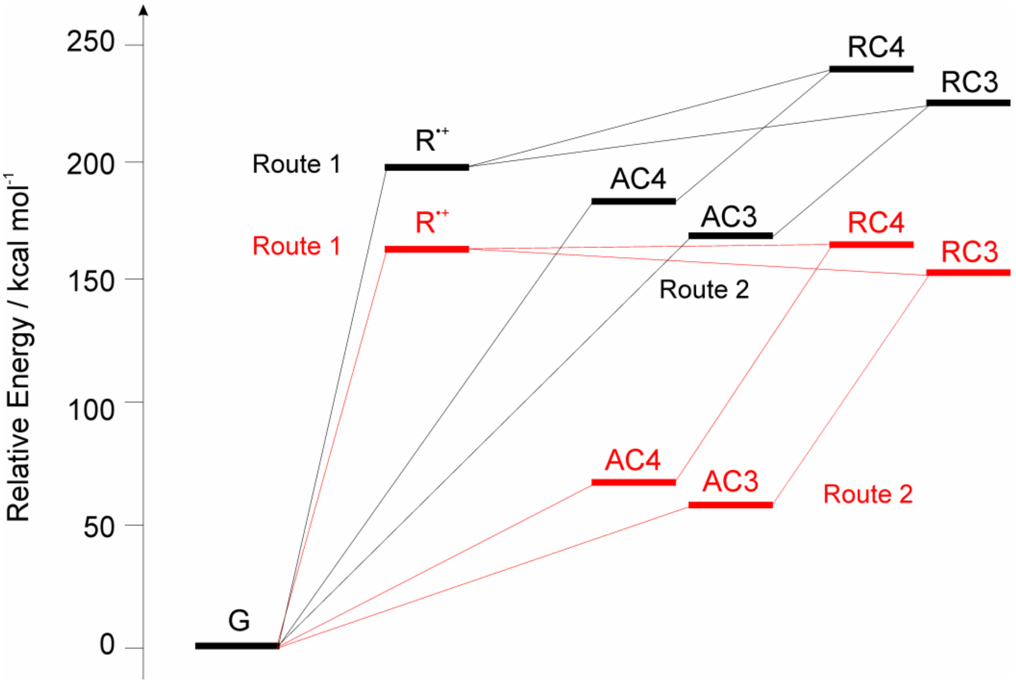


Figure 6. Schematic energy diagram for mechanism of electrochemical oxidation of compound **1** based on M06-2X/6311G(d,p) calculations in vacuum (black) and water (red) environment.

Relative energies difference between radicals RC4 and RC3 (or anions AC4 and AC3) is 10-15 kcal/mol so thermodynamically only the formation of RC3 or AC3 is possible. Spin density of the obtained radical (RC3) is increased on the carbon C3, but the huge part of spin density is also delocalized on the neighboring phenyl ring as well as on the oxygen from carbonyl C2 group. Exactly these delocalizations can be the reason of the increased RC3 stability (Fig. 7). On the other hand, 3,3-diphenyl succinimide derivatives (**2**, **4**, **6**) can form only one species of radical and anion as consequences of deprotonation at position C4. Spin density of the obtained radical is mostly localized on the cabon C4 as well as on the oxygen from the neighboring carbonyl group (Fig. 8). Spin density of $R^{\bullet+}$ for all molecules are very similar and the huge delocalization is observed over all phenyl rings (Fig. 9).

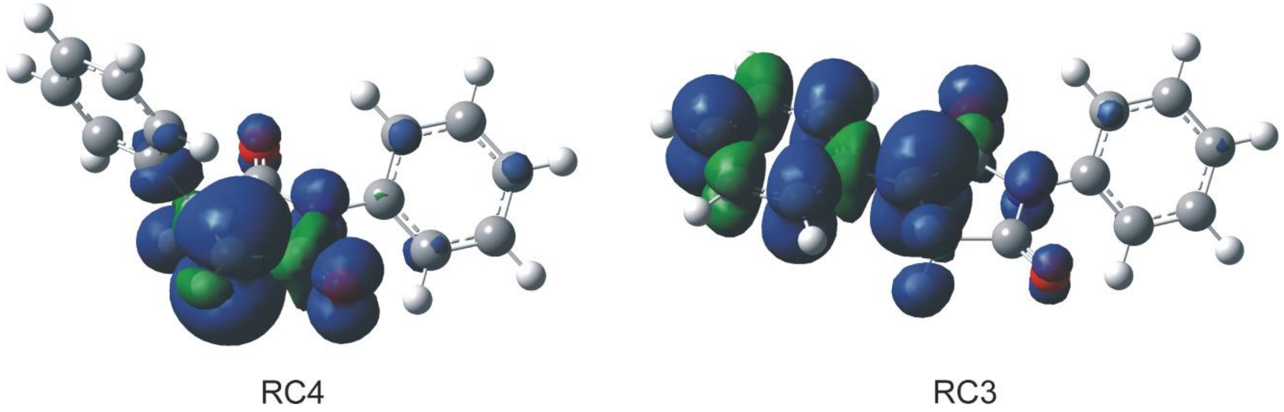

RC4 RC3

Figure 7. Spin densities of the obtained radicals for compound **1**.

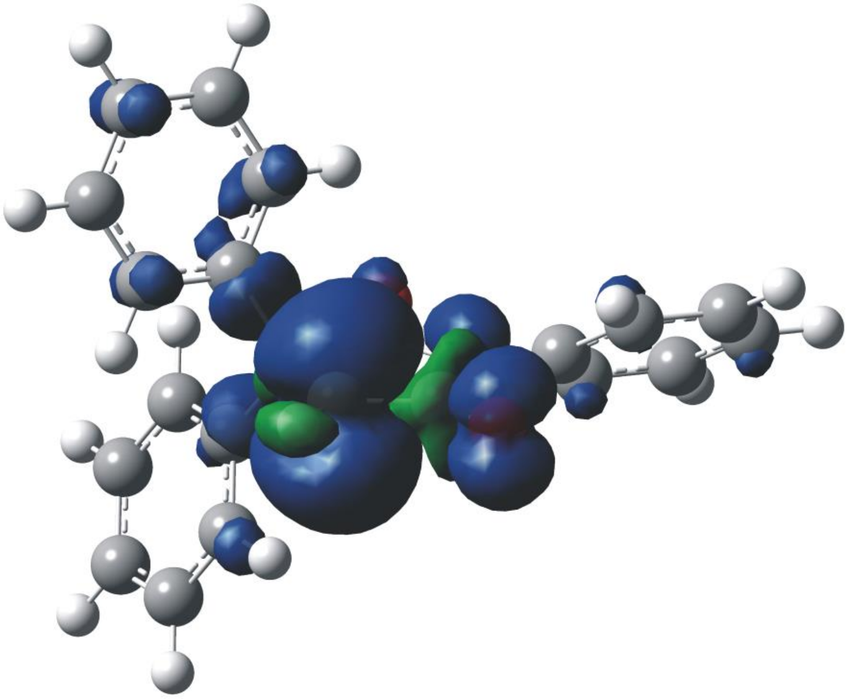


Figure 8. Spin density of RC4 radical for compound **2**.

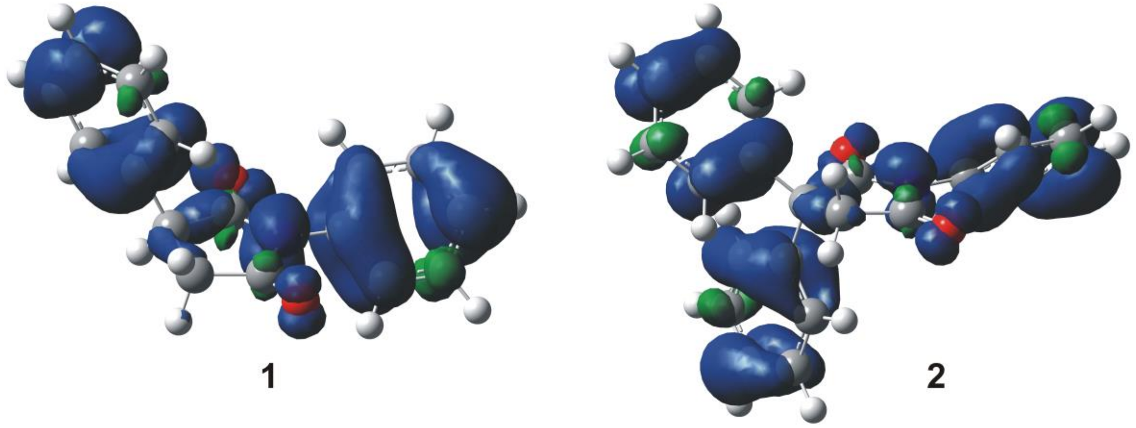
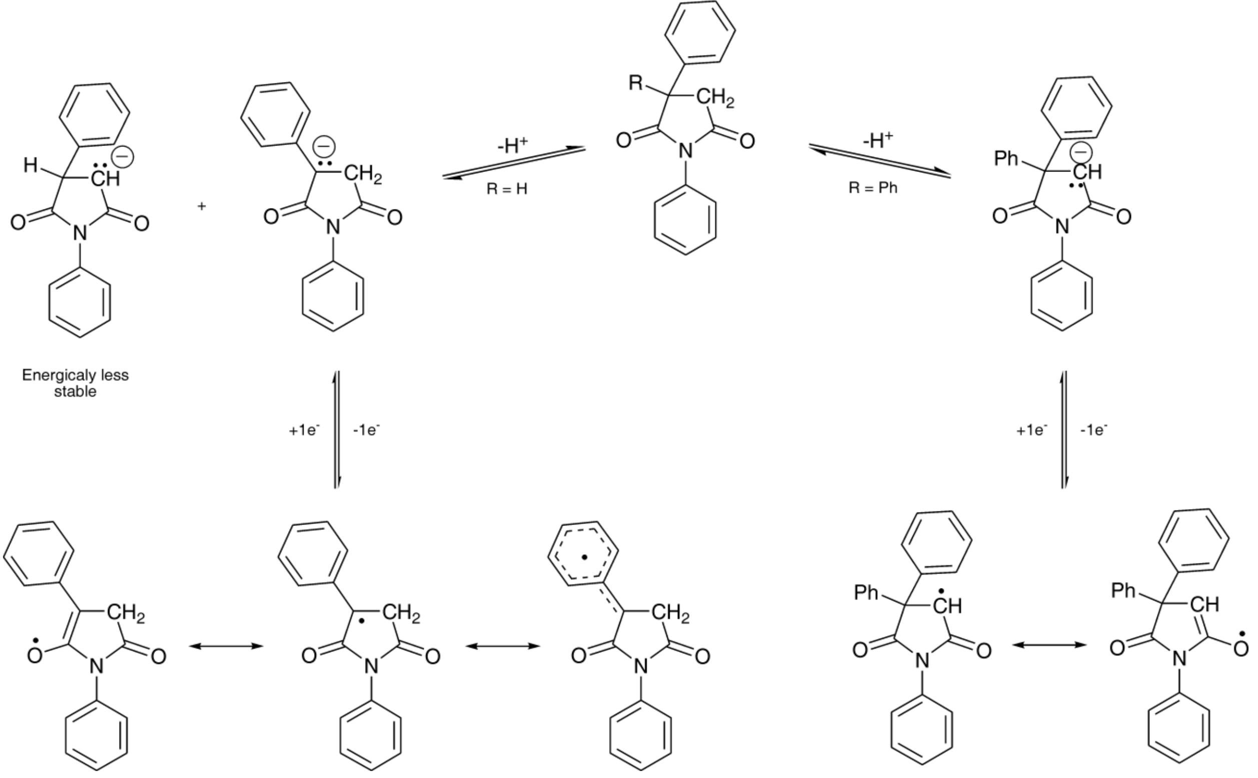


Figure 9. Spin densities of radical cations ($R^{\bullet+}$) for compounds **1** and **2**.

3.3. Possible reaction mechanism

On the basis of DFT calculations and electrochemical experiments (Figs. 3,4), the mechanism of electrooxidation of the studied succinimides can be proposed (Scheme 1). The results obtained from the CV and quantum chemical calculations indicate that the investigated compounds undergo oxidation by irreversible, diffusion controlled process including transfer of $1e^-$ and 1 proton. The voltammetric results depicted that all compounds are electrochemically oxidized by the proposed mechanism (Scheme 1) involving the conversion of carbonyl-methyne-phenyl (**1**, **3**, **5**) segment or methylene (**2**, **4**, **6**) group in free radical by the loss of one proton one electron process which exists in equilibrium with its resonating structures.



Scheme 1. Suggested electrochemical oxidation mechanism of the examined compounds.

Electrochemical characterization of the investigated succinimides was performed using cyclic and square wave voltammetry. The most active among studied succinimides is compound **1** (1,3-diphenylsuccinimide). The value of the electron transfer rate constant (k_s) obtained on electrode in the presence of compound **1** is more than 2 times greater than with compound **6** indicating higher ability for promoting the transfer of electrons between succinimide and the electrode surface. In addition, the square-wave voltammetric shown that the compound **1** is the most active one. The substituents on the phenyl ring do not affect the electrochemical activity and the shape of their voltammetric signal.

The manuscripts treating the electrochemistry of succinimides that could be find in the literature appear as [29, 30]. Unfortunately they treat only the electroreduction of succinimides with the structures which differ from selected succinimides in our study. There is lack of manuscripts containing electrooxidation of succinimides and therefore, we can not compare the obtained results with the published one.

DFT calculations showed that monophenyl succinimide derivatives (**1**, **3**, **5**) can form two species of radical and anion after deprotonation at position C3 and C4. For these compounds is characteristic that energetically the formation of radical and anion at position C3 is favored. Spin density of the obtained radical is mostly localized on the cabon C4 as well as on the oxygen from the neighboring carbonyl group. Spin density of $R^{\bullet+}$, for all molecules, is very similar and the huge delocalization is observed over all phenyl rings.

4. CONCLUSION

Electrochemical characterization of the investigated succinimides was realized using cyclic and square wave voltammetry. The most active among studied succinimides is 1,3-diphenylsuccinimide denoted as compound **1**. The value of the electron transfer rate constant (k_s) obtained on electrode in the presence of compound **1** is more than 2 times greater than with compound **6** indicating higher ability for promoting the transfer of electrons between succinimide and the electrode surface. In addition, the square-wave voltammetric shown that the compound **1** is the most active one. The substituents on the phenyl ring do not affect the electrochemical activity and the shape of their voltammetric signal.

DFT calculations showed that monophenyl succinimide derivatives (**1**, **3**, **5**) can form two species of radical and anion after deprotonation at position C3 and C4. For these compounds is characteristic that energetically the formation of radical and anion at position C3 is favored. Spin density of the obtained radical is mostly localized on the cabon C4 as well as on the oxygen from the neighboring carbonyl group. Spin density of $R^{\bullet+}$ for all molecules are very similar and the huge delocalization is observed over all phenyl rings.

The results obtained from the cyclic voltammetry and quantum chemical calculations indicate that the investigated compounds undergo oxidation by irreversible, diffusion controlled process including transfer of $1e^-$ and 1 proton. The voltammetric results depicted that all compounds are electrochemically oxidized by the proposed mechanism involving the conversion of carbonyl-methyne-phenyl segment or methylene group in free radical by the loss of one proton one electron process. According to the proposed mechanism free radical exists in equilibrium with its resonating structures.

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