

Dibenzoxazepine Anodic Determination and Removal on CuS Nanoparticles: A Theoretical Insight

Volodymyr V. Tkach ^{1,*}, Marta V. Kushnir ², Sílvia C. de Oliveira ³, Igor G. Biryuk ⁴, Tetyana B. Sykyrytska ⁴, Yana G. Ivanushko ⁴, Petro I. Yagodynets ^{2,*}, Adriano O. da Silva ⁵, Nataliia P. Derevianko ⁶, Mykhailo P. Zavhorodnii ⁶, Vira M. Odyntsova ⁷, Mykola P. Krasko ⁷, Inna M. Shevchenko ⁷, Viktoriia M. Lavrinenko ⁸, Oleksii T. Yermak ⁸, Laziz N. Niyazov ⁹, Dilfuza M. Musayeva ⁹, Xayriddin Kosimov ⁹, Oysha Jabborova ⁹, Gulandom Sagdullayeva ⁹, Elvina Sharipova ⁹, Tetiana V. Morozova ¹⁰, Oleksandr V. Mudrak ¹¹, Jarem R. Garcia ¹², José Inácio Ferrão da Paiva Martins ¹³, Zholt O. Kormosh ^{14,*}, Alla V. Grekova ¹⁵, Ianina F. Burdina ¹⁵, Ruslan V. Lavrik ¹⁶

¹ University of Trás-os-Montes and Alto Douro, 5000-020, Quinta de Prados, Folhadela, Vila Real, Portugal

² Chernivtsi National University, 58001, Kotsyubynsky Str. 2, Chernivtsi, Ukraine

³ Institute of Chemistry. Federal University of Mato Grosso do Sul, 79074 – 460, Av. Sen. Felinto Müller, 1555, Vila Ipiranga, Campo Grande, MS, Brazil

⁴ Bukovinian State Medical University, 58001, Teatralna Sq, 9, Chernivtsi, Ukraine

⁵ Federal University of the West of Pará, Juruti Campus, 68170 – 000, Rua Veríssimo de Souza Andrade, s/n, Juruti, PA, Brazil

⁶ Khortytska Natsionalna Navchalno-Reabilitatsiyna Akademiya, 69000, Naukove Mistechko, 59, Khortytsia Island, Zaporizhzhia, Ukraine

⁷ Zaporizhzhia State Medical University, 69600, Mayakovsky Ave. 24, Zaporizhzhia, Ukraine

⁸ National Pedagogical University of Ukraine, 02000, Pyrohov Str.,9, Kyiv, Ukraine

⁹ Abu Ali Ibn Sino Bukhara State Medical Institute, 705018, Navoi Str., 1, Bukhara, Uzbekistan

¹⁰ State Scientific Institution "Institute of Ecological Restoration and Development of Ukraine", Environment Remediation Section, Mytropolite Vasyl Lypkivsky Str. 35, 01001 Kyiv, Ukraine

¹¹ Vinnytsya Regional Institute of Postgraduate Education, 21000, Hrushevskogo Str. 13, Vinnytsia, Ukraine

¹² State University of Ponta Grossa, Uvaranas Campus, Av. Gal. Carlos Cavalcanti, 4748, 84030-900, Ponta Grossa, PR, Brazil

¹³ Engineering Faculty of the University of Porto, 4200-465, Rua Dr. Roberto Frias, s/n, Porto, Portugal

¹⁴ Volyn National University, 43000, Voli Ave., 13, Lutsk, Ukraine

¹⁵ Odesa National Medical University, 65000, Valikhovsky Ln. 2, Odesa, Ukraine

¹⁶ National University of Life and Environmental Science of Ukraine, 03041, Heroiv Oborony Str, 15, Kyiv, Ukraine

* Correspondence: al2025173708@alunos.utad.pt (V.V.T.); ved1988mid@rambler.ru (P. I.Y.); zholt-1971@ukr.net (Z.O.K.);

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Abstract: For the first time, the electroanalytical system for the electrochemical determination and removal of dibenzoxazepine chemical warfare agent on CuS-modified anode has been given. Two oxidation scenarios, including the electropolymerization and N-oxidation, are possible for dibenzoxazepine. Either way, both dibenzoxazepine and its N-oxide will be removed from the environment towards the polymer phase. The analysis of the electroanalytical process using linear stability theory and bifurcation analysis confirms that the steady-state stability allows us to use this process for both electroanalytical and electrocatalytic purposes.

Keywords: chemical warfare agents; dibenzoxazepine; electrochemical sensing; electrochemical determination; electrochemical removal; copper sulfide; electropolymerization; stable steady-state.

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1. Introduction

Dibenzoxazepine (Figure 1 – code name CR) – i.e., according to the Hanch-Widman nomenclature, a heterocyclic compound with a seven-membered ring and heteroatoms of Nitrogen and Oxygen, condensed with two benzene rings – is a combat poison obtained in 1962 by the Swiss chemists Higginbot and Sushitsky. It belongs to the chemical warfare class of complex action stimulators [1–4].

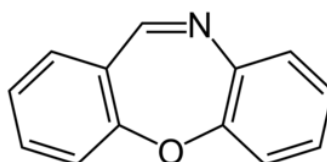


Figure 1. Dibenzoxazepine.

By its effect, dibenzoxazepine is similar to BZ-gas, but twice as strong [5–7]. Skin contact with as little as 2 mg of dibenzoxazepine within 10 minutes will cause redness. 5 mg causes erythema, and 20 mg causes excruciating pain, which increases when in contact with water. Therefore, the development of effective and rapid methods for the determination of dibenzoxazepine is a truly urgent task, and electrochemical methods offer advantages due to their low cost and the ability to detect and remove trace amounts of CWA quickly.

Considering that dibenzoxazepine is an aromatic heterocyclic compound with two aromatic fragments, we may suppose that dibenzoxazepine may be used as a monomer for a conducting polymer [8–12], suitable for use in electroanalytics and energy conversion. Nevertheless, the presence of an accepting pyridinic nitrogen atom augments the polymerization potential. Moreover, it adds a new center for the electrooxidation reaction.

In this case, strong oxidants, such as peroxides, cobalt(III) oxyhydroxide in combination with cobalt dioxide, and bivalent copper compounds, which are oxidized in an alkaline medium, yielding in situ trivalent copper compounds, are generally used in an anodic process. Copper sulfide is very popular among bivalent copper compounds used as electrode modifiers because it can generate trivalent copper, which is highly active. These oxidants are generally stabilized by a conjugated organic oligo- or macromolecular compound, such as a squaraine dye, a conducting polymer, or a carbon material [13–18]. The polymeric modifier also favors the polymerization scenario for dibenzoxazepine.

Polymeric hybrid materials have been extensively used over the last two years in both anodic and cathodic processes [19–21]. Moreover, these processes are often accompanied by electrochemical instabilities that can affect the electrochemical equipment in general and the sensing properties of the concrete system. The analysis of theoretical and experimental data [22–28] confirms that these instabilities are generally caused by ionic forces, surface phenomena, and autocatalysis, as well as by electrode material conductivity, and are characteristic of different CMEs.

Therefore, in this work, we theoretically describe the possibility of electrochemically determining and removing dibenzoxazepine using a composite containing a squaraine dye and copper sulfide nanoparticles. This is realized through the mechanism suggestion, its mathematical description via the development and analysis of the mathematical model from a stability perspective, and the comparison with similar systems [27–32].

2. Materials and Methods

Copper sulfide nanoparticles are easily oxidized at moderately high potential, yielding *in situ* an aggressive trivalent copper sulfohydroxide:



In its turn, copper sulfohydroxide oxidizes dibenzoxazepine by two parallel scenarios: N-oxidation and electropolymerization.

N-oxide also integrates the resulting polymer, as it participates in chain propagation.

Therefore, taking some assumptions [27,28], we describe the system's behavior by a trivariant equation set (3):

$$\begin{cases} \frac{db}{dt} = \frac{2}{\delta} \left(\frac{B}{\delta} (b_0 - b) - r_1 - r_p \right) \\ \frac{dn}{dt} = \frac{2}{\delta} (r_1 - r_p) \\ \frac{dc}{dt} = \frac{1}{c} (r_1 + r_p - r_0) \end{cases} \quad (2)$$

Herein, *b* and *n* stand for dibenzoxazepine and its N-oxide pre-surface concentrations, *b*₀ stands for dibenzoxazepine bulk concentration, *B* stands for dibenzoxazepine diffusion coefficient, *C* is the copper sulfide maximal matrix concentration, *c* is its surface coverage degree, and the parameters *r* stand for the corresponding reaction rates, calculated as:

$$r_1 = k_1 b (1 - c)^2 \quad (3)$$

$$r_p = k_p b^m h^n (1 - c)^x \exp(-abn) \quad (4)$$

$$r_0 = k_0 c \exp\left(\frac{F\varphi_0}{RT}\right) \quad (5)$$

Herein, the parameters *k* stand for the corresponding reaction rate constants, *m*, *n*, and *x* are the polymerization reaction orders, *a* is a variable describing the polymerization DEL effect, *F* stands for the Faraday number, φ_0 is the zero-charge related DEL potential slope, *R* is the universal gas constant, and *T* is the solution absolute temperature.

As the pyridinic nitrogen atom is not ionized in alkaline media, the ionic form transformation occurs only during the CuS(OH)-assisted electropolymerization (as radical-cation transformations). Therefore, although DEL is less affected than in the acidic medium, it is more affected than in the case of non-polymerizable analytes. Nonetheless, the stability of the topological area is enhanced, which facilitates easy analytical signal interpretation, as shown below.

3. Results and Discussion

We analyze the system with dibenzoxazepine electrochemical determination on CuS nanoparticles, deposited over a conducting polymer matrix by analyzing the equation set (2), as also the algebraic relations (3 -5) by means of the linear stability theory and, for this purpose, expose the Jacobian matrix members as (6):

$$\begin{pmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{pmatrix} \quad (6)$$

In which:

$$a_{11} = \frac{2}{\delta} \left(-\frac{B}{\delta} - k_1 (1 - c)^2 - m k_p b^{m-1} h^n (1 - c)^x \exp(-abn) + a n k_p b^m h^n (1 - c)^x \exp(-abn) \right) \quad (7)$$

$$a_{12} = \frac{2}{\delta} \left(-n k_p b^m h^{n-1} (1 - c)^x \exp(-abn) + a b k_p b^m h^n (1 - c)^x \exp(-abn) \right) \quad (8)$$

$$a_{13} = \frac{2}{\delta} (2k_1b(1-c) + mk_p b^{m-1} h^n (1-c)^x \exp(-abn)) \quad (9)$$

$$a_{21} = \frac{2}{\delta} (k_1(1-c)^2 - mk_p b^{m-1} h^n (1-c)^x \exp(-abn) + ank_p b^m h^n (1-c)^x \exp(-abn)) \quad (10)$$

$$a_{22} = \frac{2}{\delta} (-nk_p b^m h^{n-1} (1-c)^x \exp(-abn) + abk_p b^m h^n (1-c)^x \exp(-abn)) \quad (11)$$

$$a_{23} = \frac{2}{\delta} (-2k_1b(1-c) + mk_p b^{m-1} h^n (1-c)^x \exp(-abn)) \quad (12)$$

$$a_{31} = \frac{1}{c} (k_1(1-c)^2 + mk_p b^{m-1} h^n (1-c)^x \exp(-abn) - ank_p b^m h^n (1-c)^x \exp(-abn)) \quad (13)$$

$$a_{32} = \frac{1}{c} (nk_p b^m h^{n-1} (1-c)^x \exp(-abn) - abk_p b^m h^n (1-c)^x \exp(-abn)) \quad (14)$$

$$a_{33} = \frac{1}{c} \left(-2k_1b(1-c) - mk_p b^{m-1} h^n (1-c)^x \exp(-abn) - k_o \exp\left(\frac{F\phi_o}{RT}\right) + jk_o c \exp\left(\frac{F\phi_o}{RT}\right) \right) \quad (15)$$

From the Jacobian main diagonal elements (7), (11), and (15), it is possible to conclude that the positive callback, necessary for the Hopf bifurcation, describing the oscillatory behavior, is possible, as the mentioned elements possess positive addenda.

In this system, the oscillatory behavior is more probable than for the simplest case, but, either way, it is less probable than in the acidic medium, as the Hopf bifurcation conditions in this system are less likely to be satisfied. This type of behavior is caused by two factors, one against the other in the simplest case, but the DEL ionic force change factor is only manifested in the assisted electropolymerization reaction, which involves the appearance, transformation, and recombination of cation radicals, according to the standard Díaz or Kim mechanism. Also, the oscillatory behavior will be caused by changes in DEL and surface ionic force and conductivity during the electrochemical stage, leading to cyclic changes in DEL's electrochemical and electrophysical properties, which are thereby manifested as oscillations in electrochemical parameter values. Mathematically, they are manifested by the positivity of the elements $ank_p b^m h^n (1-c)^x \exp(-abn) > 0$, $abk_p b^m h^n (1-c)^x \exp(-abn) > 0$ if $a > 0$, like also $-jk_3 c \exp\left(\frac{F\phi_o}{RT}\right) > 0$ if $j > 0$. As for the oscillations frequency and amplitude, it will be dependent on the background electrolyte composition, as already shown both experimentally and theoretically [27,28].

Avoiding the cumbersome expressions during the steady-state stability investigation using the Routh-Hurwitz criterion, we rewrite the Jacobian determinant as (16):

$$\frac{4}{\delta^2 c} \begin{vmatrix} -\kappa - \mathcal{E} - \Lambda & -\Sigma & \Psi + P \\ \mathcal{E} - \Lambda & -\Sigma & P - \Psi \\ \mathcal{E} + \Lambda & \Sigma & -P - \Psi - \Omega \end{vmatrix} \quad (16)$$

Which may be transformed, by using the determinant properties, into (17):

$$\frac{4}{\delta^2 c} \begin{vmatrix} -\kappa & 0 & -\Omega \\ 2\mathcal{E} & 0 & -2\Psi - \Omega \\ \mathcal{E} + \Lambda & \Sigma & -P - \Psi - \Omega \end{vmatrix} \quad (17)$$

Opening the brackets, applying the Det J < 0 requisite, salient from the stability criterion, like also Det J = 0 monotonic stability criterion, and changing the signs to the opposite, we obtain the steady-state stability and monotonic instability (corresponding to the detection limit) requirement exposed as (18):

$$\Sigma(2\Psi\kappa + 2\Omega\kappa + 2\mathcal{E}\Omega) \begin{cases} > 0, \text{ steady - state stability} \\ = 0, \text{ monotonic instability} \end{cases} \quad (18)$$

defining a typical diffusion- and kinetically controlled electropolymerization system [27,28], efficient from both electroanalytical and electrosynthetic points of view. The condition (18) is warranted to be realized, in the case of the positivity of the kinetic parameters \mathcal{E} and Ω (as the rest of them are always positive). As this positivity is observed across the majority of parameter values, the system is electroanalytically and electrosynthetically efficient.

In the absence of the side reactions or other factors capable of compromising the analyte and (or) modifier stability, excluding the reactions foreseen by the mechanism, the linearity between the electrochemical parameter and concentration is observed, providing an efficient analytical signal interpretation, which is really important for CWA concentration monitoring. Again, in a neutral or lightly alkaline medium, the analytical signal interpretation will be more efficient.

The condition $\text{Det } J=0$ corresponds to the detection limit, manifested by the monotonic instability. It may be seen as an N-shaped part of the steady-state voltammogram, depicting the boundary between stable and unstable steady states and corresponding to the steady-state multiplicity. In other words, multiple steady states, each unstable, coexist at this point, thereby defining the stability margin.

Poly(dibenzoxazepine) may have multiple applications, including electroanalytics, energy storage and conversion, and corrosion protection. It is possible that electroanalytical applications will be analyzed in one of our next works.

4. Conclusions

Based on the analysis of the system, the electrochemical determination and removal of dibenzoxazepine via assisted N-oxidation and electropolymerization over the conducting polymer/CuS composite, it is possible to conclude that the electroanalytical process is efficient. Moreover, in neutral and lightly alkaline media, it is more efficient than in acidic and strongly alkaline solutions. Although oscillatory behavior is possible, it is even more probable than in the simplest case, it will be less probable than in acidic solutions. As for the electroanalytical-electrosynthetic process, it is either diffusion or kinetically controlled, and the electroanalytical signal is easy to interpret.

Author Contributions

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Conflicts of Interest

The authors declare no conflict of interest.

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