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The Theoretical Description for the Use of Poly(7-hydroxyphenoxazone) as Electrode Modifier for pOH Monitoring

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Abstract: The potential for electroanalytical variation of a well-known litmus test for pOH measurement has been evaluated theoretically. In this system, pOH is determined anodically through a series of electrochemical reactions involving electropolymerized 7-hydroxyphenoxazone—the most pH-sensitive litmus dye. Mathematical modeling confirms that a cathode modified with poly(7-hydroxyphenoxazone) is suitable for pOH measurements. However, oscillatory and monotonic instabilities are more likely to occur in this system compared to similar systems.

Keywords: litmus; alkalinity; 7-hydroxyphenoxazone; electropolymerization; electrochemical sensor; electrochemical oscillations; stable steady-state.

1. Introduction

The litmus test is one of the earliest methods for assessing solution acidity and basicity [1–4]. Various litmus types are found in lichens, notably *Rocella*, *Lecanora*, *Ochrolechia*, *Parmotrema*, *Dendrographa*, and *Palmotrema*. Their pH and pOH sensitivity arises from different litmus dyes, including litmines, which comprise both carbo- and heterocyclic structures. The main chromophore, 7-hydroxyphenoxazone (Figure 1) [5 - 7], is violet in neutral solutions, red in acidic media, and blue in basic media.

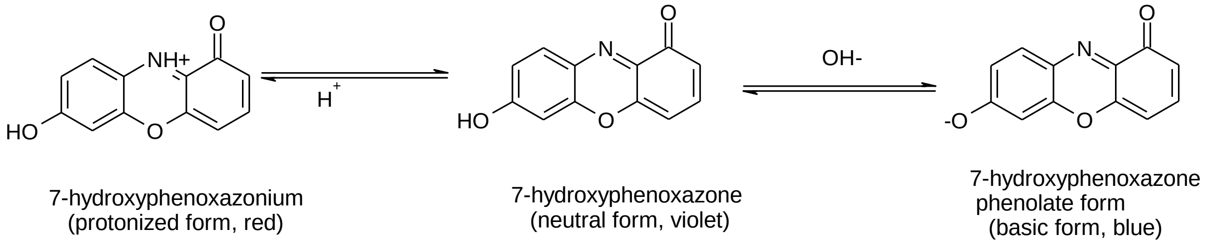

Figure 1. 7-hydroxyphenoxazone and litmus colors.

Nowadays, dye electropolymerization has become a widely used and efficient tool in electroanalytical processes [7–14]. In such systems, dye polymers act as active substances and mediators for the electroanalysis of various analytes. In the case of poly(7-hydroxyphenoxazone)-assisted pOH sensing, this approach represents a modernized version of the classical method. Because OH^- participates directly in the electroanalytical process, direct monitoring of pOH becomes feasible, whereas pH measurement in alkaline media remains indirect.

However, electrochemical instabilities—which can complicate the interpretation of analytical signals—may arise from various dynamic effects inherent to these processes [15–17]. Such instabilities are typical and can limit both analytical and remediation applications of the electrochemical system. To predict their occurrence and potential impact, it is necessary to investigate the process mechanistically and analyze its behavior theoretically.

The aim of this work is a mechanistic evaluation of poly(7-hydroxyphenoxazone)-assisted oxidative pOH measurement, in which hydroxyl ions react with the dye polymer, followed by oxidation of the phenolized ortho-carbon and formation of the quinonic moiety. A corresponding mathematical model is developed and analyzed using linear stability theory and bifurcation analysis. Theoretical results are further compared with the behavior of similar systems [18–24].

2. Materials and Methods

Dye electropolymerization is generally performed on a carbon material, which is more akin to the conjugated monomer. In the case of anodic electropolymerization, two or more peaks on the CV are observed. The first corresponds to electrode activation, the second (if any) to low-molecular-weight dye oxidation, and the last to electropolymerization.

The first stage of the electroanalytical process involves the phenolate (blue litmus form), which thereby becomes more electrophilic. Reacting once more with the hydroxyl yields the ortho-semiquinone intermediate, which is then oxidized to quinone, thereby providing the appearance of the analytical signal (Figure 2).

The final quinonic form will become more bright due to the appearance of the quinonic moiety.

If additional OH doping occurs, three electrochemical stages have to be mentioned and described. All of them are pOH-dependent and may be used for pOH direct measurement.

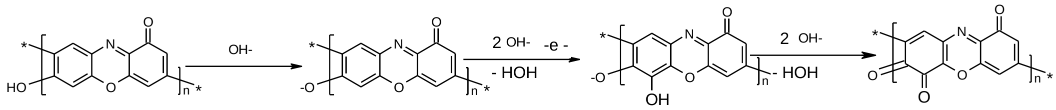

Figure 2. The scheme for the electroanalytical process.

Accepting certain assumptions [18–24], we describe the behavior of this system by the trivariate equation set, exposed as (1):

$$\begin{cases} \frac{da}{dt} = \frac{2}{\delta}\left(\frac{A}{\delta}(a_0 - a) - r_1 - r_2 - r_{31} - r_{32}\right) \\ \frac{dp}{dt} = \frac{1}{P}(r_1 - r_2) \\ \frac{ds}{dt} = \frac{1}{S}(r_2 - r_{31} - r_{32}) \end{cases} \quad (1)$$

Herein, a is the hydroxyl pre-surface layer concentration, A is its diffusion coefficient, δ stands for the diffusion layer thickness, a_0 for hydroxyl bulk concentration, p is the phenolate (blue) polymer form surface coverage degree, P is its maximal surface concentration, s is the semiquinone polymer form surface coverage degree, S is its maximal surface concentration, and the parameter r stands for the corresponding reaction rates, including the additional OH-polymer doping, calculated as:

$$r_1 = k_1 a(1 - p - s)\exp(-\lambda a) \quad (2)$$

$$r_2 = k_2 p a^n \exp\left(\frac{xF\varphi_0}{RT}\right) \quad (3)$$

$$r_{31} = k_{31} s a^m \exp\left(\frac{yF\varphi_0}{RT}\right) \quad (4)$$

$$r_{32} = k_{32} s a^l \exp\left(\frac{yF\varphi_0}{RT}\right) \quad (5)$$

Here, the parameters k represent the corresponding reaction rate constants, n is the number of monomer units, λ is the parameter relating pOH to the DEL ionic force, l, m, and n are the hydroxyl reaction orders in the electrochemical stages, and x, y, and z are the numbers of transferred electrons. F is the Faraday constant, φ_0 is the zero-charge-related potential slope, R is the universal gas constant, and T is the absolute temperature.

The ionic species are heavily involved in all chemical and electrochemical stages, which increases the likelihood of DEL-related oscillatory behavior compared to simpler systems. Nevertheless, anodic pOH monitoring assisted by poly(7-hydroxyphenoxazone) remains efficient, as demonstrated below.

3. Results and Discussion

In order to investigate the electrochemical behavior of the system with the poly(7-hydroxyphenoxazone)-assisted pOH monitoring, we analyze the equation set (1) alongside the algebraic relations (2–5) by means of linear stability theory. The steady-state Jacobian matrix members may be described 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:

$$a_{11} = \frac{2}{\delta}\left(-\frac{A}{\delta} - k_1(1-p-s)\exp(-\lambda a) + \lambda k_1 a(1-p-s)\exp(-\lambda a) - nk_2pa^{n-1}\exp\left(\frac{xF\varphi_0}{RT}\right) - mk_{31}sa^{m-1}\exp\left(\frac{yF\varphi_0}{RT}\right) - lk_{32}sa^{l-1}\exp\left(\frac{yF\varphi_0}{RT}\right)\right) \quad (7)$$

$$a_{12} = \frac{2}{\delta}\left(k_1 a\exp(-\lambda a) - k_2a^n\exp\left(\frac{xF\varphi_0}{RT}\right) + jk_2pa^n\exp\left(\frac{xF\varphi_0}{RT}\right) + jk_{31}sa^m\exp\left(\frac{yF\varphi_0}{RT}\right) + jk_{32}sa^l\exp\left(\frac{yF\varphi_0}{RT}\right)\right) \quad (8)$$

$$a_{13} = \frac{2}{\delta}\left(-k_1 a\exp(-\lambda a) + fk_2pa^n\exp\left(\frac{xF\varphi_0}{RT}\right) - k_{31}a^m\exp\left(\frac{yF\varphi_0}{RT}\right) + fk_{31}sa^m\exp\left(\frac{yF\varphi_0}{RT}\right) - k_{32}a^l\exp\left(\frac{yF\varphi_0}{RT}\right) + fk_{32}sa^l\exp\left(\frac{yF\varphi_0}{RT}\right)\right) \quad (9)$$

$$a_{21} = \frac{1}{P}\left(k_1(1-p-s)\exp(-\lambda a) + \lambda k_1 a(1-p-s)\exp(-\lambda a) - nk_2pa^{n-1}\exp\left(\frac{xF\varphi_0}{RT}\right)\right) \quad (10)$$

$$a_{22} = \frac{1}{P}\left(-k_1 a\exp(-\lambda a) - k_2a^n\exp\left(\frac{xF\varphi_0}{RT}\right) + jk_2pa^n\exp\left(\frac{xF\varphi_0}{RT}\right)\right) \quad (11)$$

$$a_{23} = \frac{1}{P}\left(k_1 a\exp(-\lambda a) + fk_2pa^n\exp\left(\frac{xF\varphi_0}{RT}\right)\right) \quad (12)$$

$$a_{31} = \frac{1}{S}\left(nk_2pa^{n-1}\exp\left(\frac{xF\varphi_0}{RT}\right) - mk_{31}sa^{m-1}\exp\left(\frac{yF\varphi_0}{RT}\right) - lk_{32}sa^{l-1}\exp\left(\frac{yF\varphi_0}{RT}\right)\right) \quad (13)$$

$$a_{32} = \frac{1}{S}\left(k_2a^n\exp\left(\frac{xF\varphi_0}{RT}\right) - jk_2pa^n\exp\left(\frac{xF\varphi_0}{RT}\right) + jk_{31}sa^m\exp\left(\frac{yF\varphi_0}{RT}\right) + jk_{32}sa^l\exp\left(\frac{yF\varphi_0}{RT}\right)\right) \quad (14)$$

$$a_{33} = \frac{1}{S}\left(-fk_2pa^n\exp\left(\frac{xF\varphi_0}{RT}\right) - k_{31}a^m\exp\left(\frac{yF\varphi_0}{RT}\right) + fk_{31}sa^m\exp\left(\frac{yF\varphi_0}{RT}\right) - k_{32}a^l\exp\left(\frac{yF\varphi_0}{RT}\right) + fk_{32}sa^l\exp\left(\frac{yF\varphi_0}{RT}\right)\right) \quad (15)$$

Avoiding the cumbersome expression during the determinant analysis, we introduce new variables and rewrite the determinant as (16):

$$\text{Det J} = \frac{4}{\delta PS}\begin{vmatrix} -\kappa - \Xi - T - \Sigma & B + \Lambda + P & -A - \Gamma - \Omega \\ \Xi - T & B - \Lambda & A - \Gamma \\ T - \Sigma & \Lambda - P & \Gamma - \Omega \end{vmatrix} \quad (16)$$

Which, considering the determinant properties and Gaussian transformations, may be rewritten as (17):

$$\frac{4}{\delta PS}\begin{vmatrix} -\kappa - \Xi - 2\Sigma & B + 2\Lambda & -A \\ -\frac{1}{3}\kappa - T - 5\Sigma & -\frac{2}{3}B - \frac{1}{3}\Lambda - \frac{2}{3}P & -\Gamma \\ -\kappa - 3\Sigma & 2B + 2\Lambda - P & -3\Omega \end{vmatrix} \quad (17)$$

Considering that:

$$-Det\ J \begin{cases} > 0, for\ steady-state\ stability \\ = 0\ monotonic\ instability \end{cases} \quad (18)$$

Opening the brackets, applying the Det J<0 requisite, salient from the criterion, and changing the signs to the opposite, we rewrite the condition set as (19):

$$A\left(10\Sigma B - \frac{2B}{3} - 2TB + 10\Sigma\Lambda - \frac{2\Lambda}{3} - 2T\Lambda + 5\Sigma P - \frac{B}{3} - TP\right) + \Gamma(\kappa B + 2\kappa P + 2\Xi B + 2\Xi\Lambda + 2\Xi P + \Sigma B - 2\Sigma\Lambda + 4\Sigma P) + \Omega(2\kappa B + \kappa\Lambda + 2\kappa P + 2\Xi B + \Xi\Lambda + 2\Xi P + 4\Sigma B + 2\Sigma\Lambda + 4\Sigma P + \kappa B + 3TB + 15\Sigma B + 2\kappa\Lambda + 6T\Lambda + 30\Sigma\Lambda)\begin{cases} > 0, curve\ linearity \\ = 0, detection\ limit \end{cases} \quad (19)$$

If −Det J>0, the Routh-Hurwitz stability criterion is satisfied, and the steady state is thereby stable, allowing efficient steady-state drug determination. Furthermore, the wide stability region enables the system to be applied across a variety of conditions.

This criterion is readily met when the kinetic parameters Ξ, Σ, Λ, Ρ, and Ω are positive. In most cases, these parameters have positive values, and since the other variables in the determinant are also positive, this indicates a large steady-state stability region. The electroanalytical process is therefore predominantly kinetically controlled.

In the absence of side reactions or other factors that could compromise the stability of the analyte or modifier—excluding the reactions predicted by the mechanism—the relationship between the electrochemical parameter and concentration remains linear, ensuring efficient interpretation of the analytical signal, which is critical for pOH monitoring.

The condition Det J=0 corresponds to the detection limit, manifested as monotonic instability. This appears as an N-shaped segment in the steady-state voltammogram, marking the boundary between stable and unstable states and reflecting steady-state multiplicity. In other words, multiple unstable steady states coexist at this point.

Oscillatory behavior arises beyond the detection limit through a Hopf bifurcation. Its occurrence requires positive feedback-related terms in the main diagonal. Observing the main diagonal elements (7), (11), and (15), oscillatory behavior becomes possible when the kinetic parameters aaa and jjj are positive, reflecting DEL effects from both chemical and electrochemical stages. This feature is typical of similar systems [27,28] and can be described by the positivity of these elements $a\lambda k_1 a(1-p-s)\exp(-\lambda a)$, if λ>0, like also $jk_2 pa^n \exp\left(\frac{xF\varphi_0}{RT}\right) > 0$, if j>0 and $fk_{31}sa^m \exp\left(\frac{yF\varphi_0}{RT}\right) + fk_{32}sa^l \exp\left(\frac{yF\varphi_0}{RT}\right) > 0$, if f>0, describing the positive callback, dependent on the system´s characteristics. For example, the oscillation frequency and amplitude will depend on the background electrolyte composition, which has been proven experimentally and theoretically [18–24].

The behavior of this system becomes more dynamic than that of similar ones [18–24]. Nevertheless, if the polymer is completely doped, the reaction rate r_{32} will be equal to nil, and the behavior will be simplified. Moreover, in a neutral medium (pOH=pH=7) or close to neutral, the participation of OH^- in the electrochemical stages may be neglected, and the model will be even more simplified.

4. Conclusions

From the investigation of system behavior during pOH electrochemical monitoring using a litmus chromophore polymer as the electrode modifier, it can be concluded that pOH can be effectively tracked through a sequence of chemical and electrochemical processes involving phenolization, semiquinone formation, and quinone formation. The process dynamics are further influenced by transformations of ionic species, which increase the likelihood of oscillatory behavior. Nevertheless, the overall process remains efficient and is predominantly controlled by the kinetics of the chemical and electrochemical reactions involved.

Author Contributions

Conceptualization, V.V.T., T.V.M., J.I.F.P.M., Y.G.I., P.I.Y., M.V.K. and M.P.K., methodology, V.V.T.; T.V.M.; A.O.H.; M.V.K.; S.C.O.; I.V.K.; I.I.K.; V.V.K.; I.G.B.; T.B.S.; Y.G.I.; M.G.B.; P.I.Y.; A.O.S.;J.R.G.; J.I.F.P.M.; G.F.T.; O.P.M.; M.V.M.; O.O.M.; M.J.M.;

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Data supporting the findings of this study are available upon reasonable request from the corresponding author.

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Conflicts of Interest

The authors declare no conflict of interest.

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