

The Theoretical Description for Sucralose and Alitam Food Sweeteners Electrochemical Determination by CoO(OH)/CoO₂ Redox Pair

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Abstract: For the first time, the electrochemical determination of sucralose and alitam on the anode, modified by cobalt (III) oxyhydroxide, paired with tetravalent cobalt oxide, has been described theoretically. Sucralose electrooxidation by tetravalent cobalt derivative is caused by hydroxymethyl group oxidation, whereas alitam is oxidized by both primary amine and thioether sulfur. The correspondent mathematical model analysis has shown that the steady-state stability range is wider than in most systems of electrochemical determination due to the fragile ionization of both analytes in a neutral medium. For this reason, the CoO(OH)/CoO₂ redox pair may be efficiently used for the electrochemical determination of both sucralose and alitam.

Keywords: sucralose; alitam; cobalt(III) oxyhydroxide; electrochemical sensors; stable steady-state.

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1. Introduction

The sweetener [1–4] is a substance generally added to food and drinks to give them a sweet taste. Common sugar or sucrose is the most used sweetener in the world. Nevertheless, the abusive consumption of sugar may lead to complexities like diabetes Mellitus type I and II, which is the reason why sugar substitutes are widely used as dietetic sweeteners.

Sucralose (Figure 1) is one of the most used dietetic sweeteners in Portugal and throughout the European Union in the alimentary and pharmaceutical industries as a flavor corrector [5]. In the USA, it is also known by its registered trademark, Splenda®. In Codex Alimentarius, it is registered as E955. It is a trichloro-substituted derivative of galactosucrose, which has twice the sweetness of saccharin, triple the sweetness of aspartame, and is up to a thousand times as sweet as the common sugar. When it comes to physical properties, free sucralose is a white, shiny, odorless substance soluble in water.

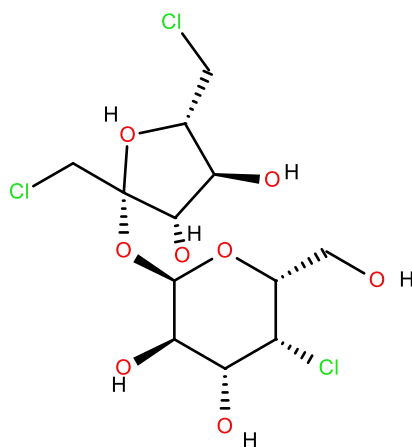


Figure 1. Sucralose.

Although sucralose is a derivative of natural compounds, it itself is not a natural compound. They synthesize it from sucrose in several steps (Figure 2).

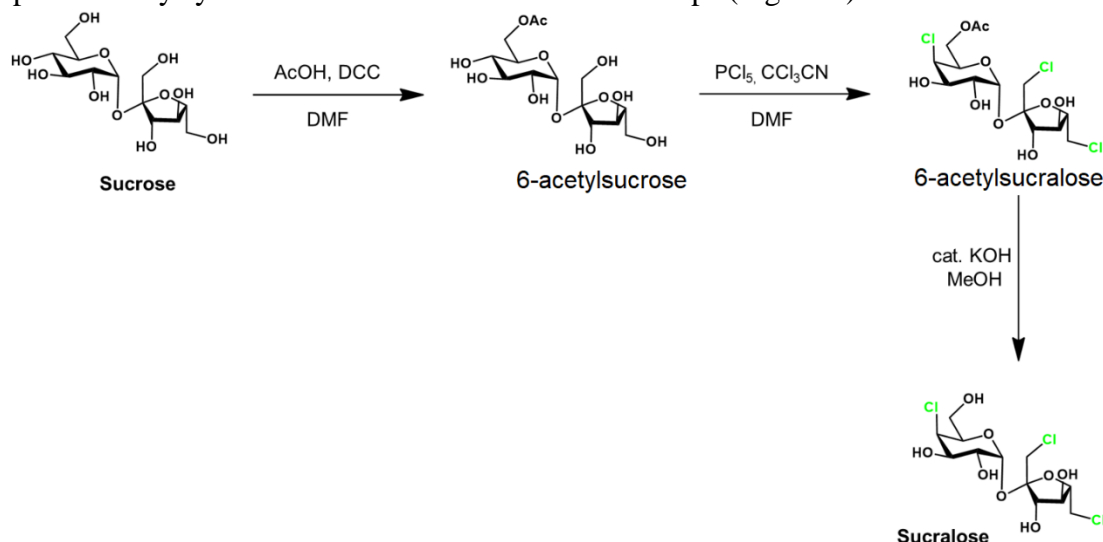


Figure 2. Sucralose synthesis from sucrose. Adapted from [6] with the correspondent's permission.

The acylation in the first stage may also be made by diethylazodicarboxylate. The C4 epimerization (mutarotation) is realized in the second stage. This synthesis involves either the toxic reagents (DCC , DAAD , and PCl_5) or the intermediate (6-acetyl sucralose), as mentioned below.

Despite being considered safe for use by diabetics and athletes, its harmful effects on human health and the environment are still unknown, and some of its negative effects have only begun to be studied now. A recent study involving pregnant and breastfeeding women [7–8] confirms that sucralose enters breast milk, causing irreparable damage to the development of the gut microbiota of the human fetus in the last months of pregnancy, as well as in neonates and babies, the reason why its safety for use during pregnancy and breastfeeding is still questioned.

Moreover, as sucralose is nearly not biodegradable, it accumulates in the environment [9,10]. Furthermore, when sucralose decomposes thermally or by some bacteria, it transforms into toxic compounds such as dioxins and tetrachlorodibenzofurans. It should not be forgotten that sucralose is also part of the group of halogen organic compounds (Figure 3). Therefore, developing a method for eliminating sucralose from the environment, mainly from sewage and groundwater, is truly current [11,12].

Alitam (Figure 3) is an aspartic acid derivative, twice as sweet as sucralose. It was developed in 1980 by Pfizer. Its half-life is lower than that for acesulfame K and aspartame. It is permitted for use in Canada, Mexico, Australia, and some European countries. Nevertheless, it is still not in use in the US and most European countries, and EU authorities have not approved it, although its E-code is already attributed to E956. Moreover, its use may have long-term effects, which still have not been manifested [13–16]. For this reason, developing a technique capable of detecting alitam is actual [17, 18].

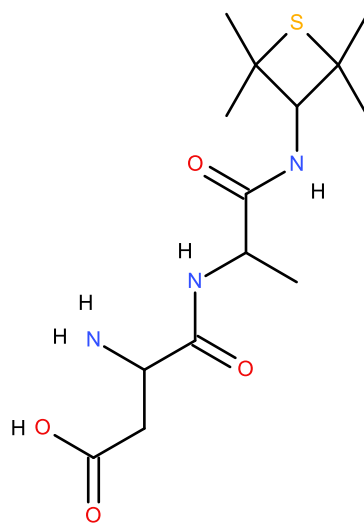


Figure 3. Alitam.

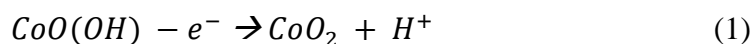
The possibility of the electrochemical sensing of sucralose by anodic oxidation of the remaining hydroxyl groups has already been foreseen by us theoretically [19–21], being thereby confirmed experimentally in [22]. Aspartame derivatives have also been electrochemically detected [23], and alitam won't be an exception. In this case, cobalt (III) oxyhydroxide, popular in electroanalytics [24–26], could be efficiently used as an electrode modifier for determining both sucralose and alitam. Moreover, considering the presence of accepting groups of alitam and its high oxidation potential, the CoO(OH)/CoO_2 redox pair should be used.

Nevertheless, the analogous processes tend to be accompanied by electrochemical instabilities, negatively impacting the sensor stability and analytical signal interpretation [27,28]. Taking this into account, it's necessary to evaluate theoretically the behavior of this

electroanalytical system in terms of stability and most common electrochemical instabilities. So, this investigation, which also includes the comparison of the behavior of this system with that of similar ones, is the aim of the present study.

2. Materials and Methods

Cobalt (III) oxyhydroxide, possessing trivalent cobalt, may act as either oxidant or reductant. Moreover, $\text{CoO}(\text{OH})$ exists in different modifications, and their interchange may also be a redox process. In this case, it is oxidized, yielding the more active tetravalent cobalt derivative:



Cobalt dioxide will thereby oxidize both sucralose and alitum, as shown in Figure 4.

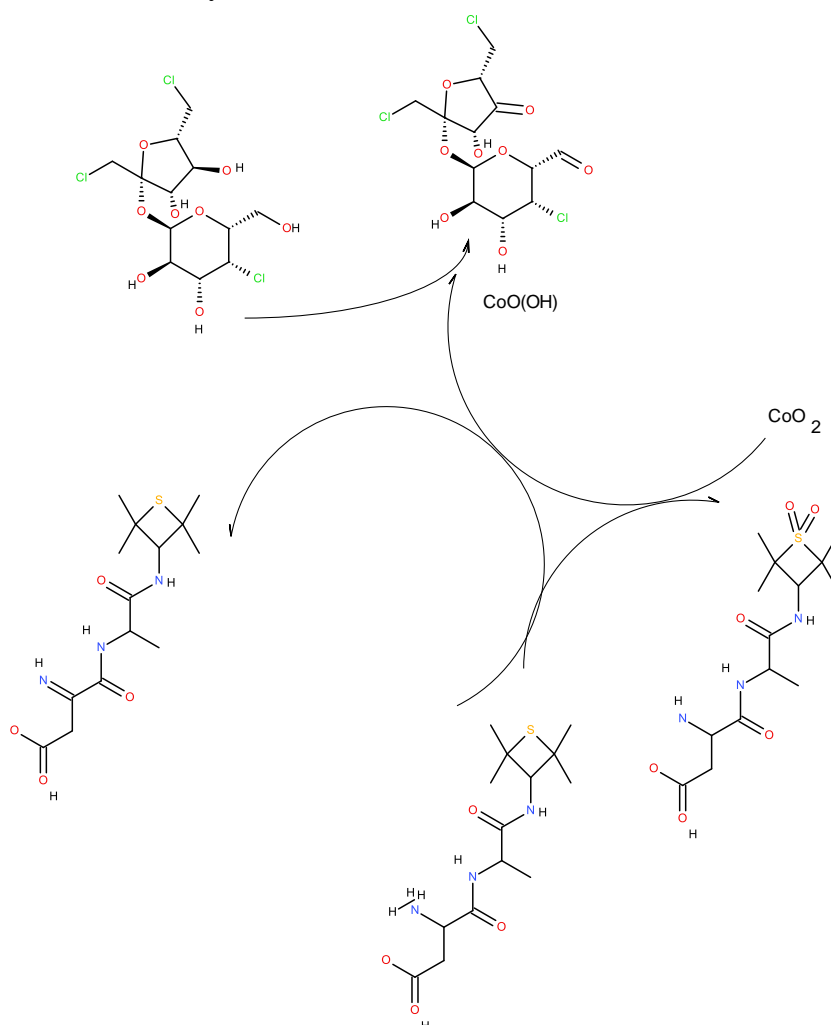


Figure 4. The scheme of the electroanalytical process.

Taking this into account and taking some assumptions [27, 28], we describe the behavior of this system by a trivariate balance differential equation-set (2):

$$\begin{cases} \frac{da}{dt} = \frac{2}{\delta} \left(\frac{\Delta}{\delta} (a_0 - a) - r_{11} - r_{12} \right) \\ \frac{ds}{dt} = \frac{2}{\delta} \left(\frac{S}{\delta} (s_0 - s) - r_2 \right) \\ \frac{dc}{dt} = \frac{1}{c} (r_{11} + r_{12} + r_2 - r_o) \end{cases} \quad (2)$$

Herein, a and s are acetyl sucralose and sucralose pre-surface concentrations, a_0 and s_0 are their correspondent bulk concentrations, Δ and S stand for diffusion coefficients, δ is the

pre-surface layer thickness, c is the cobalt (III) oxyhydroxide surface coverage degree, C is its maximal concentration, and the parameters r are the correspondent reaction rates, calculated as (3 – 6):

$$r_{11} = k_1 a (1 - c)^2 \quad (3)$$

$$r_{12} = k_1 a (1 - c)^2 \quad (4)$$

$$r_2 = k_2 s (1 - c)^2 \quad (5)$$

$$r_o = k_o c \exp\left(-\frac{F\varphi_0}{RT}\right) \quad (6)$$

Herein, the parameters k stand for the correspondent reactions rate constants, $F=N_A \cdot e$ is the Faraday number, φ_0 is the zero-charge related potential slope, R is the universal gas constant, and T is the absolute reaction temperature.

Taking into account that in a neutral medium, neither carboxyl nor the amino group is ionized and neither acidic nor alkaline hydrolysis of both of the analytes is realized, it is the most suitable for the most efficient electroanalytical process, as either steady-state stability is easier to obtain and maintain, or oscillatory and monotonic instability is realized, as shown below.

3. Results and Discussion

We investigate the behavior of the system with the electroanalytical detection of sucralose and its 6-acetyl derivative using the linear stability theory. The stationary elements of the Jacobi functional matrix can be calculated as (7):

$$\begin{pmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{pmatrix} \quad (7)$$

Sendo:

$$a_{11} = \frac{2}{\delta} \left(-\frac{A}{\delta} - k_{11}(1 - c)^2 - k_{11}(1 - c)^2 \right) \quad (8)$$

$$a_{12} = 0 \quad (9)$$

$$a_{13} = \frac{2}{\delta} (2k_{12}a(1 - c) + 2k_{12}a(1 - c)) \quad (10)$$

$$a_{21} = 0 \quad (11)$$

$$a_{22} = \frac{2}{\delta} \left(-\frac{S}{\delta} - k_2 s (1 - c)^2 \right) \quad (12)$$

$$a_{23} = \frac{2}{\delta} (2k_2 s (1 - c)) \quad (13)$$

$$a_{31} = \frac{1}{P} (k_{11}(1 - c)^2 + k_{12}(1 - c)^2) \quad (14)$$

$$a_{32} = \frac{1}{P} (k_2 s (1 - c)^2) \quad (15)$$

$$a_{33} = \frac{1}{P} \left(-2k_{12}a(1 - c) - 2k_{12}a(1 - c) - 2k_2 s (1 - c) - k_o \exp\left(-\frac{F\varphi_0}{RT}\right) + jk_o c \exp\left(-\frac{F\varphi_0}{RT}\right) \right) \quad (16)$$

Avoiding the cumbersome expressions during the determinant analysis, we rewrite the Jacobian determinant as (17):

$$\frac{4}{\delta^2 P} \begin{vmatrix} -\kappa - \Xi & 0 & \Lambda \\ 0 & -\xi - \Sigma & P \\ \Xi & \Sigma & -\Lambda - P - \Omega \end{vmatrix} \quad (17)$$

Opening the straight brackets, applying both the Routh-Hurwitz stability criterion and monotonic instability condition, and changing the signs to the opposite, we obtain the requirement set, expressed as (18):

$$\kappa(\xi\Lambda + \xi P + \xi\Omega + \Sigma\Lambda + \Sigma\Omega) + \Xi(\xi\Phi + \xi\Omega + \Sigma\Omega) \begin{cases} > 0, \text{linear dependence} \\ = 0, \text{detection limit} \end{cases} \quad (18)$$

If $-\text{Det } J > 0$, the Routh-Hurwitz stability criterion is valid, and the steady-state is thereby stable, providing an efficient steady-state determination of both sucralose and alitام. Moreover, the wide stability region permits its use in pharmaceutical formulations, food, beverages, and biological liquids, as well as in the environment. In some cases, the neutralization, achieved by the pH adjusting to 7 by alkaline or acidic background electrolyte and/or polymer organic phase, is needed.

This criterion is readily satisfied if the kinetic parameter Ω obtain positive values, which occurs in the vast majority of the cases, indicating the vast steady-state stability topological region. The electroanalytical process is kinetically controlled.

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 important for drug concentration monitoring.

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, which depicts the margin between the stable and unstable states and corresponds to the steady-state multiplicity. In other words, multiple steady-states, each one unstable, coexist at this point.

As for the oscillatory behavior, it is realized beyond the detection limit in the case of the Hopf bifurcation realization. Its realization requires the presence of the positive-callback related positive addendums in main diagonal elements.

Observing the main diagonal elements (8), (12), and (16), we may observe that the only cause for the oscillatory behavior is the DEL influences of all the electrochemical stages, typical for similar systems [27,28]. It may be described by the positivity of the element $jk_0c \exp\left(-\frac{F\phi_0}{RT}\right) > 0$ if $j > 0$, describing the similar cyclic phenomena on both DEL and surface during the electrochemical stages. These phenomena are why the oscillation frequency and amplitude depend on the background electrolyte composition, which has been proven experimentally and theoretically [27,28].

As for the cathodic process, it is also possible. Nevertheless, it will be realized on different electrode modifiers at low pH, leading to sucralose dehalogenation, alitام acidic hydrolysis, and salt formation, all affecting the DEL and augmenting the probability of the oscillatory behavior. This case will be analyzed in our next publications

4. Conclusions

From the analysis of the process with the cathodic detection of sucralose and alitام, assisted by trivalent cobalt oxyhydroxide, paired with tetravalent cobalt derivative, it is possible to conclude that in the present process, the polymer facilitates the obtaining and maintenance of the stable, steady state in this system, due to the analytes fragile ionization in neutral medium. The electrochemical process is mostly kinetically controlled. The oscillatory behavior is possible in this system, being caused by periodic effects on the structure of the double electric layer. These effects are observed at the electrochemical stage. The electroanalytical process may be used for the environment, food, beverages, and biological liquids. Nevertheless, it is recommended that the sample pH be neutralized before use.

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Conflicts of Interest

The authors declare no conflict of interest.

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