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Assessment of the Dynamic Parameters of an Ion-Selective Microsensor †

Eugenia Eftimie Totu [1,*], Ibrahim Isildak [2], Daniel Costinel Petre [1], Selim Işildak [2], Mustafa Niğde [2], Özlem Tavukçuoğlu [2], Ridvan Yildirim [2], Tiberiu Totu [3] and Ismail Ağır[4]

[1] Faculty of Applied Chemistry and Materials Science, University Politehnica of Bucharest, 1-7 Polizu St., sector 1, 011061 Bucharest, Romania; danielpetre.empresa@hotmail.com
[2] Department of Bioengineering, Faculty of Chemical and Metallurgical Engineering, Yildiz Technical University, 34210 Esenler-Istanbul, Turkey; iisildak@gmail.com (I.I.); se.isildak@gmail.com (S.I.); mnigde@gmail.com (M.N.); ozlemoztolan@gmail.com (Ö.T.); ridvanyildirim@ymail.com (R.Y.)
[3] School of Life Sciences, Ecole Polytechnique Fédérale de Lausanne (EPFL), Route Cantonale, 1015 Lausanne, Switzerland; tiberiu.totu@yahoo.com
[4] Bioengineering Department, Istanbul Medeniyet University, Goztepe, 34700 Istanbul, Turkey; agr.isml@gmail.com
* Correspondence: eugenia.totu@upb.ro
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Abstract: We report the statistical parameters associated with the dynamic characteristics of a calcium microelectrode. The electrochemical behavior of the obtained microsensor was assessed against the main interferents.

Keywords: ion sensing device; clinical applications; dynamic characteristics; statistical parameters

1. Introduction

Selective ion microelectrodes with selective membranes as sensitive elements are used in many applications in industrial environments, environmental protection, or clinical studies [1–3].

The impressive development of new technologies for the realization of microstructures and the reduced manufacturing costs have recommended, in recent years, the use of microsensors, especially in situations where the use of minimal amounts of an analyte is required. At the same time, the tendency to achieve selective ion membranes that are subsequently compatible with microtechnology, due to the need to expand the use of miniaturized ion selective electrodes (ISE), should be noted [4–6].

Selective microsensors are characterized through a specific dynamic behavior in complex matrices when interfering ions are present. In analytical laboratories, the determination of the dynamic electrochemical characteristics of the sensing devices is essential. The current work introduces the statistical assessment of the dynamic features for a calcium microelectrode.

2. Experimental

The used electrochemical system consisted of a carbon paste solid-contact electrochemical calcium microsensor and a homemade solid-state Ag/AgCl as a micro-reference electrode [7]. The determinations were performed with the help of a multichannel MEDISEN type IEMS-4 potentiometric system, provided by Medisen Medikal Teknolojiler Araştırma Geliştirme San. Tic. Ltd. Şti., Turkey. The electrochemical behavior was studied for calcium solutions over a broad

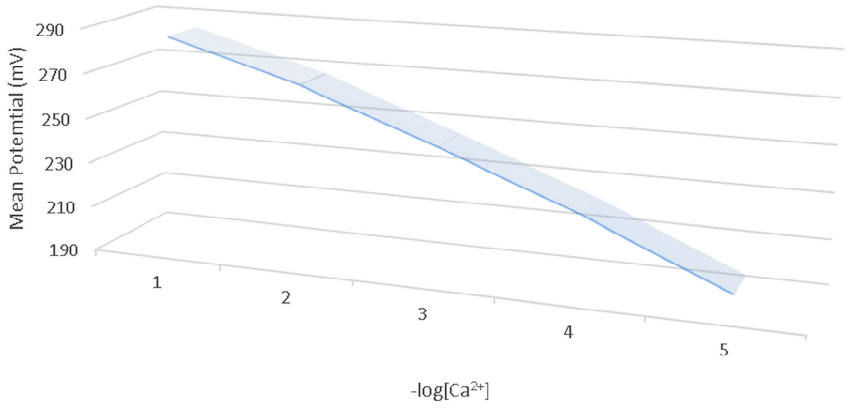
concentration range 10^{-1} to 10^{-6} mol/L and in the presence of the interfering ions: magnesium, potassium, lithium, sodium, ammonium, and barium (10^{-2} mol/L).

3. Results and Discussion

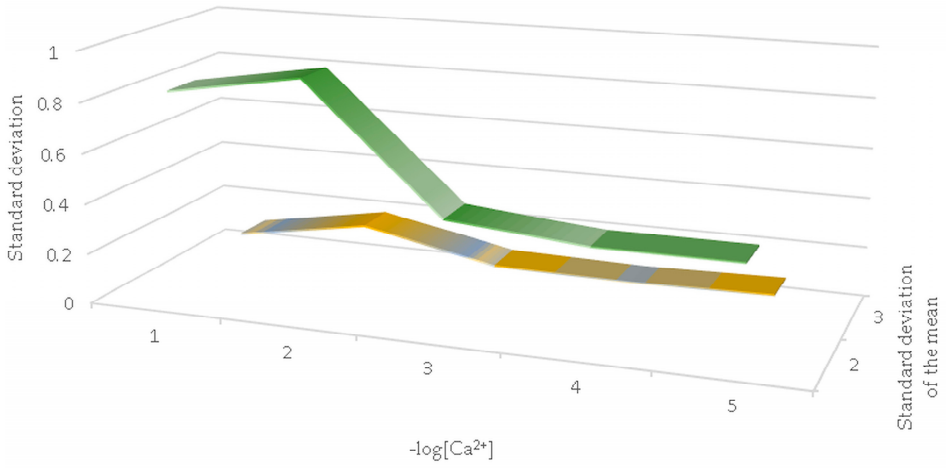
The tested calcium microelectrode presented a Nernstian electrochemical answer over the whole concentration range (26.8 ± 0.3 mV/decade). In the presence of the interfering ions, the microelectrode exhibited a reproducible and highly selective response. The selectivity coefficients ($\log K_{Ca,i}$) ranged from −3.62 for Mg^{2+} to −4.51 for NH_4^+, highlighting the satisfactory selectivity of the sensing device. Moreover, the fast response time (6.0 ± 0.2 s) qualifies the solid-state microsensor for applications where a fast and accurate electrochemical response is required.

The repeatability parameter completed the assessment of the main dynamic characteristics as electrochemical response, selectivity, detection limit (3.2×10^{-6} mol/L), and response time. The determined values supported the reliability of the calcium microsensor.

The statistical parameters calculated for the mean potentials recorded at different concentrations (10^{-2} mol/L, 10^{-3} mol/L, 10^{-4} mol/L) evidenced a low standard deviation (0.98, 0.43, 0.38), respectively, and a standard deviation of the mean (0.20, 0.09, 0.08)—see Figure 1. The low values for the statistical parameters signify the high reliability of the recorded experimental data.



(**a**)



(**b**)

Figure 1. (**a**) Mean potential; (**b**) statistical parameters.

The study of the electrochemical potential variation over a pH range between four and nine for the calcium microelectrode showed no significant variation (maximum 7 mV).

4. Conclusions

The electrochemical dynamic characteristics and the assessment of the statistical parameters for the calcium microelectrode indicate that the sensing device could be successfully used for the study of complex matrices (clinical or environmental) where a quick and reliable determination of the calcium ion levels is requested.

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Conflicts of Interest: The authors declare no conflicts of interest.

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