BOX-BEHNKEN EXPERIMENTAL DESIGN FOR OPTIMIZATION OF VOLTAMMETRIC NANOMATERIAL-MODIFIED ELECTRODES

Javier E. Vilasó-Cadre[1,*], Juan Hidalgo-Viteri[2], Iván A. Reyes-Domínguez[1,3,], Graziella Liana Turdean[2]**

[1]*Institute of Metallurgy, Autonomous University of San Luis Potosí, Mexico.*
[2]*"Babes-Bolyai" University, Faculty of Chemistry and Chemical Engineering, Department of Chemical Engineering, Research Centre of Electrochemistry and Non-Conventional Materials, Romania.*
[3]*National Council of Humanities, Science and Technologies of Mexico, Mexico City, Mexico.*
*e-mail: *a321418@alumnos.uaslp.mx, ** alejandro.reyes@uaslp.mx*

Abstract

In the field of analytical chemistry, experimental designs have been used in some of the most important areas such as separations, spectroscopic analytical methods, chromatography, and electroanalytical methods. In the latter, they have been used to optimize the electroanalytical response, taking into account factors such as sweep and pulse parameters in voltammetric methods, as well as physicochemical conditions. However, the amount of catalyst is the only electrode preparation variable generally considered as part of the experimental factors. This work presents the optimization of two voltammetric sensors using a surface response experimental design considering only factors related to the preparation of the electrode. The two types of sensors most commonly used in voltammetry were considered: a carbon paste electrode (CPE) and a glassy carbon electrode (GCE), both modified with electrocatalytic nanomaterial.

The Box-Behnken experimental design was used to optimize the sensors. In the case of the CPE, the experimental factors were: the amount of nanomaterial in the paste, the amount of mineral oil, and the resting time of the electrode before being used as a voltammetric sensor for an industrial analyte. The response variable for the CPE was the peak current intensity in cyclic voltammograms. In the case of the GCE, the experimental factors were: chitosan concentration, the amount of nanomaterial in the modifier suspension, and the amount of suspension drops deposited on the electrode. The response variable for the GCE was the peak current intensity of the voltammograms for the oxidation of an environmental analyte.

The analysis of variance in the optimization of both electrodes showed the factors with statistically significant influence on the peak current (p-value < 0.05). In addition, second-order effects of both individual factors and interactions were detected for both electrodes. From the mathematical model, the main effects plot was generated for each electrode, which allowed to study the effect of the factors, and it was verified in both cases that an excess of nanomaterial decreases the sensor response due to electron transfer resistance effects at the electrochemical interface. The response surfaces allowed the determination of the optimal response conditions of the sensors, which could be experimentally verified by observing a higher peak current with respect to the non-optimized electrode. This work demonstrated that Box-Behnken experimental designs are a powerful analytical tool to improve the electrochemical response of voltammetric sensors, taking into account factors related only to the electrode preparation, which shows the importance of these in the analytical response.