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A factorial design for optimizing a flow injection analysis system

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Abstract The use of a factorial design for the response exploration of a flow injection (FI) system is described and illustrated by FI spectrophotometric determination of paraquat. Variable response (absorbance) is explored as a function of the factors flow rate and length of the reaction coil. The present study was found to be useful to detect and estimate any interaction among the factors that may affect the optimal conditions for the maximal response in the optimization of the FI system, which is not possible with a univariate design. In addition, this study showed that factorial experiments enable economy of experimentation and yield results of high precision due to the use of the whole data for calculating the effects.

Introduction

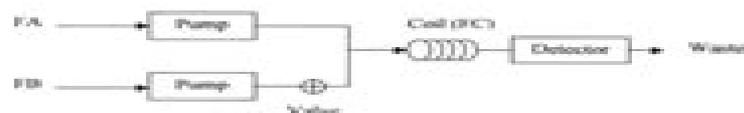
Flow injection analysis (FIA) represents an advancement of wet chemical methods because it offers great advantages such as flexibility, speed, high sampling rate and wide applicability in many fields [1–3]. However, the choice of the best set of operational conditions for assembling FIA systems is generally complex. The conventional method of optimization involves varying each parameter in turn, while keeping the others constant until an adequate response is obtained. This type of optimization procedure has some limitations, since it is not reasonable to assume that the variables may be independent from each other. On the other hand, in most cases it may be too demanding in terms of experimental work [4–6].

Today, as in chromatography, the optimization of FIA systems on basis of statistically designed experiments has been thoroughly developed and many strategies are available, such as the sequential simplex method, factorial design, central composite design, etc. [7–15]. However, a factorial design has not been employed in order to find the precise relationship between the large number of parameters involved and the optimal response for the measurement of paraquat by continuous-flow colorimetry. Therefore, in this work the development of a totally randomized factorial design has been used to detect and estimate any interaction among the factors (coil lengths and flow rates) that may affect the optimal conditions for the maximal response (absorbance) in the flow injection system.

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Department of Analysis and Control, Faculty of Pharmacy,
Universidad de los Andes, Mérida, 5101, Venezuela**Fig. 1** Representation of the flow-injection manifold employed**Experimental**

Reagents. All reagents used were of analytical grade. A stock solution of paraquat (0.1 µg/mL) was prepared by dissolving paraquat dichloride (Sigma, St. Louis, MO) in deionized, distilled water. A working standard solution was prepared by appropriate dilution of the stock solution. A sample solution of ascorbic acid reagent was prepared by dissolving 2.500 g of ascorbic acid (BDH), 0.100 g of potassium iodate (Merck) and 1.000 g of EDTA, disodium salt (BDH) in deionized, distilled water and the solution was diluted to the final volume of 1 L. This solution was stored in a refrigerator (5°C) during the experimental work. A 1 mol/L sodium hydroxide (Merck) solution was prepared from the solid base.

Apparatus. A SHIMADZU spectrophotometer model UV-1201 with a home-made glass flow cell was employed. A Cole-Palmer (model Masterflex L/S) with two peristaltic pumps was employed to propel the reagents. A Rhodyne (model 7000) injection valve was used for the sample injection. The Tygon tubing of 0.8 mm i.d. was used in all systems. All the connections were made as indicated in Fig. 1. Statgraphics statistical software package (version 5.1, Statistical Graphics) was used for data treatment.

Operation. The sample solution (a fixed volume, 60 µL) is injected into the carrier stream of 1 mol/L sodium hydroxide solution (Factor FB). After injection, the sample and the ascorbic acid reagent (Factor FA) emerge at a confluence point. Subsequently, sample and reagent are forwarded by both streams (FA and FB) to pass through a certain length of coiled tube (Factor FC) where reaction takes place. Paraquat is reduced by ascorbic acid reagent to yield a highly colored free radical absorbing at 600 nm. The variation of absorbance is thus continuously monitored in the flow cell [16, 17].

Procedures. For the present work the probable experimental region is unknown. Thus to start the experiment, a tentative experimental region was selected with the following minimum and maximum limits; 1 and 5 mL/min for both flow rates (FA and FB) and 50 and 100 cm for the reaction coil length (FC). Beginning with this tentative region various designs were studied and discussed to detect and estimate any interaction among the factors that may affect the maximal response at each step of optimization.

Results and discussion

Design 1. A 3^2 fractional factorial design was selected; specifically, a one-third fraction of a 3^2 design (or 3^{2-1}) which gives nine experimental points [18]. To the fractional factorial design five points were added in order to cover the whole experimen-

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