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Spectrophotometric determination of nitrite in soil and water using cefixime and central composite design

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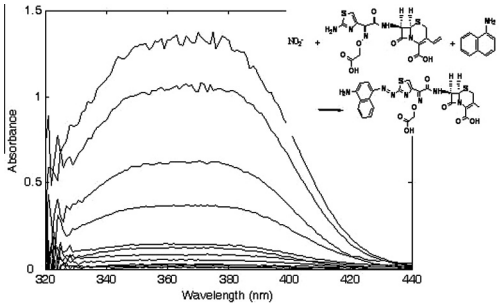
HIGHLIGHTS

- Cefixime and 1-naphtylamine were used as reagents for Griess reaction.
- Central composite design was used to explore the optimal reaction conditions.
- The method was successfully applied to determine nitrite in water and soil.

GRAPHICAL ABSTRACT



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ABSTRACT

The present paper seeks to develop a simple method for the spectrophotometric determination of nitrite in soil and water samples and also measure optimum reaction conditions along with other analytical parameters. The method is based on the diazotization-coupling reaction of nitrite with cefixime and 1-naphthylamine in an acidic solution (Griess reaction). The final product that is an azo dye has an orange color with maximum absorption at 360 nm which Beer's Law is obeyed over the concentration range 0.02–15.00 mg L^{-1} of nitrite. Optimal conditions of the variables affecting the reaction were obtained by central composite design (CCD). A detection limit of 4.3×10^{-3} mg L^{-1} was obtained for determination of nitrite by the proposed method. The proposed method was successfully applied to determine nitrite in soil and water samples. The molar absorptivity of the product of the reaction and RSD in determination of nitrite in real samples are 4.1×10^3 (L mol^{-1} cm^{-1}) and lower than 10%, respectively.

Introduction

Public health and the environment have faced serious threats due to the increasing concentration of nitrite in soil, groundwater, rivers and lakes. Nitrite is an excellent indicator of the extent of pollution in environmental samples and the continuous ingestion of this ion can have serious implications for animal and human health. Nitrite acts as a precursor in the formation of many N-nitrosamines at physiological pH, which are notorious as potent carcinogens in a variety of animals and suspected of causing cancer in humans as well. Moreover, high concentration of nitrite in blood can react with iron (III) of the hemoglobin, turning it into methemoglobin which has no oxygen-carrying ability. This fatal disease is called methemoglobinemia (oxygen deficiency).

According to United States Environmental Protection Agency (USEPA), the maximum contaminant level that is allowed for nitrite in drinking water is 1 mg L^{-1} [1]. Given that concentration of nitrite is widely recognized as a hazardous problem, there is a need to develop methods for monitoring the nitrite ion levels in environmental matrices, which is desirable from the view point of environmental analytical chemistry.

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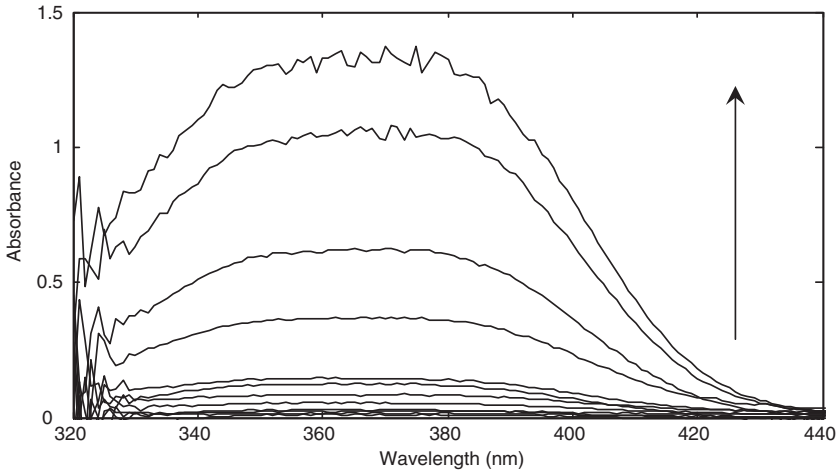

Fig. 1. Spectra of the calibration samples in conditions: 0.02–15.00 mg L^{-1} of nitrite, cefixime with concentration of 370 mg L^{-1}, 1-naphthylamine with concentration of 28 mg L^{-1} and pH 1, 30 min after mixing.

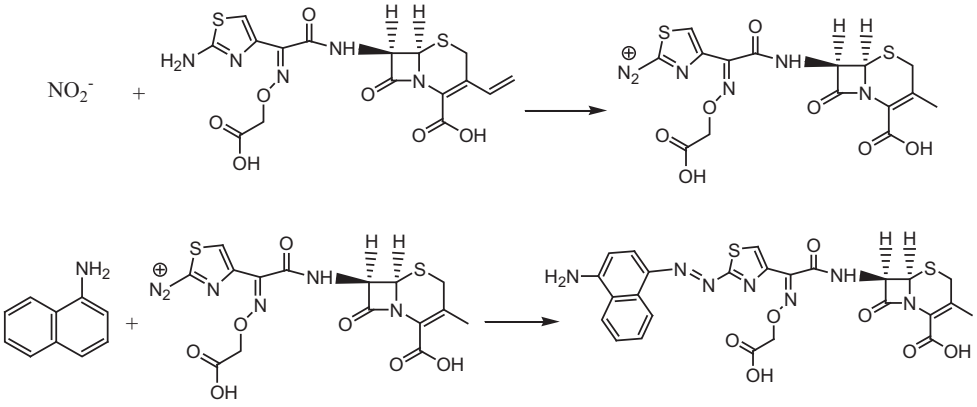

Fig. 2. The proposed mechanism for the reaction of nitrite with cefixime and 1-naphthylamine.

Table 1
Experimental factors and their investigated levels.

Factors	Levels		
	−1	0	1
X_1 (pH)	0.7	2.5	4.3
X_2 (cefixime concentration)	70	250	430
X_3 (1-naphthylamine concentration)	18	30	42

Various methods have been already put forward for facilitating the determination of nitrite [2–4]. However, some of these methods such as ion chromatography [5–8], electrophoresis [11–14], chemiluminescence [15], spectrofluorimetry [16–19], electrochemical [20–22], voltammetry [23], amperometry [24,25], potentiometry [26] are relatively expensive. Spectrophotometric methods are simple and cheaper methods for the analysis. Spectrophotometric methods for determination of nitrite [27–33] are usually based on Saltzman reaction [34], Jacobs–hochheiser reaction [35], Griess–Ilosvey reaction [36]. Kinetic spectrophotometric methods [9,10] have also been used for determination of nitrite.

Experimental design is a strategy to gather empirical knowledge based on the analysis of experimental data and not on theoretical models. The objectives of experimental design are the following: efficiency (get more information from fewer experiments), and focusing (collect only the information you really need) [37]. An effective experimental design technique commonly used for process analysis and modeling is central composite design (CCD).

Table 2
Experiments based on central composite design with three factors.

Run Order	1-Naphthylamine concentration	Cefixime concentration	pH	Response
1	40	100	4.0	0.1240
2	30	250	2.5	0.0517
3	20	400	1.0	0.0713
4	40	100	1.0	0.1510
5	30	250	2.5	0.1230
6	40	400	1.0	0.1050
7	42	250	2.5	0.0712
8	30	70	2.5	0.0289
9	30	250	2.5	0.0798
10	30	250	2.5	0.0517
11	18	250	2.5	0.0272
12	40	400	4.0	0.0936
13	20	100	4.0	0.1230
14	20	400	4.0	0.0959
15	30	250	2.5	0.0514
16	30	250	4.3	0.0588
17	20	100	1.0	0.0494
18	30	250	2.5	0.1110
19	30	430	2.5	0.0981
20	30	250	0.7	0.0599