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Simultaneous spectrophotometric determination of synthetic dyes in food samples after cloud point extraction using multiple response optimizations

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ABSTRACT

A sensitive cloud point extraction method for simultaneous determination of trace amounts of sunset yellow (SY), allura red (AR) and brilliant blue (BB) by spectrophotometry was developed. Experimental parameters such as Triton X-100 concentration, KCl concentration and initial pH on extraction efficiency of dyes were optimized using response surface methodology (RSM) with a Doehlert design. Experimental data were evaluated by applying RSM integrating a desirability function approach. The optimum condition for extraction efficiency of SY, AR and BB simultaneously were: Triton X-100 concentration 0.0635 mol L^{-1}, KCl concentration 0.11 mol L^{-1} and pH 4 with maximum overall desirability D of 0.95. Correspondingly, the maximum extraction efficiency of SY, AR and BB were 100%, 92.23% and 95.69%, respectively. At optimal conditions, extraction efficiencies were 99.8%, 92.48% and 95.96% for SY, AR and BB, respectively. These values were only 0.2%, 0.25% and 0.27% different from the predicted values, suggesting that the desirability function approach with RSM was a useful technique for simultaneously dye extraction. Linear calibration curves were obtained in the range of 0.02–4 for SY, 0.025–2.5 for AR and 0.02–4 μg mL^{-1} for BB under optimum condition. Detection limit based on three times the standard deviation of the blank ($3S_b$) was 0.009, 0.01 and 0.007 μg mL^{-1} ($n=10$) for SY, AR and BB, respectively. The method was successfully used for the simultaneous determination of the dyes in different food samples.

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1. Introduction

Synthetic dyes are usually added to food stuffs and soft drinks. They improve appearance, color and texture. This class of compounds has been added legally into foods since the 1880s to make food more attractive for customers. However, some synthetic colorants can be pathogenic, particularly if they are excessively consumed [1,2].

Sunset Yellow (FD and C Yellowd, Y6), Allura Red (FD and C Red-40, R40) and Brilliant Blue FCF (FD and C Blue 1, Bl) are commonly used in many food products such as gelatins, puddings, confections and beverages. In Europe, Allura red is not recommended for consumption by children. It is used in some tattoo inks and many products. Generally, synthetic dyes contain azo (N=N) functional groups and aromatic ring structures that are harmful to human health [2,3]. For this reason, simple, rapid, accurate and selective analytical methods for the determination of synthetic dyes are required. There are various methods for the determination of synthetic dyes in food samples such as capillary electrophoresis [4], high performance liquid chromatography (HPLC) [5], ion pair HPLC [6], spectrophotometry [7] and fluorimetry [8].

Cloud point extraction (CPE) is a green alternative for liquid–liquid extraction, analyte preconcentration and sample clean-up. CPE follows the principles of "green chemistry" because it uses small amounts of mildly toxic surfactants compared with toxic organic solvents. Surfactants are also non-flammable and not particularly volatile. This analytical technique utilizes the clouding behavior observed when a solution containing a polyoxyethylene-type nonionic surfactant is stirred and/or heated before being allowed to settle. Because the surfactant is dehydrated during the settling process, the liquid separates into aqueous and surfactant-rich phases [9,10]. The volume of surfactant-rich phase is very small, thus a high enrichment factor can be obtained. This leads to an enhanced sensitivity of the analysis without further sample clean-up or evaporation steps [11]. Cloud point extraction methodology was used to spectrophotometric determination of sunset

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Table 1
Molecular structure and other properties of SY, AR and BB.

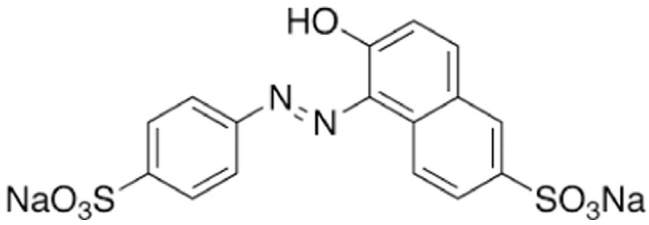
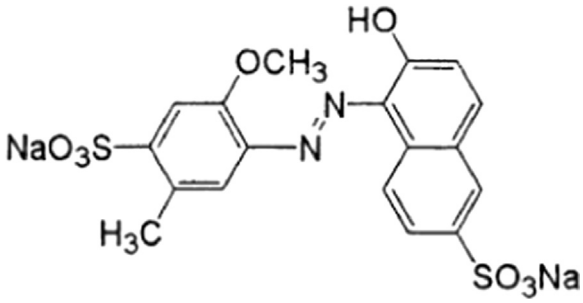
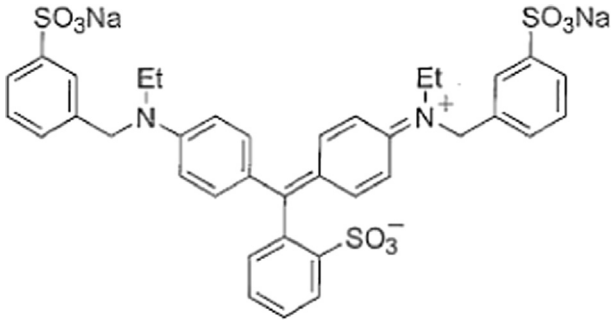
Dye	Molecular structure	Chemical formula	λ_{max} (nm)
SY		$C_{16}H_{10}N_2Na_2O_7S_2$	420
AR		$C_{18}H_{14}N_2Na_2O_8S_2$	520
BB		$C_{37}H_{34}N_2Na_2O_9S_3$	635

Table 2
Doehlert design matrix and experimental values

No. Exp.	**variables**						**Responses (extraction efficiency, %)**		
	X_1		X_2		X_3		SY	AR	BB
1	(1)	0.10	(0)	0.08	(0)	4.0	70.05 ± 0.94	53.96 ± 0.88	63.6 ± 0.8
2	(0.5)	0.08	(−0.5)	0.06	(0.707)	4.5	66.28 ± 0.86	57.80 ± 0.78	65.7 ± 1.5
3	(0.5)	0.08	(−0.5)	0.06	(−0.707)	3.5	61.3 ± 1.0	62.9 ± 1.5	70.3 ± 1.0
4	(0.5)	0.08	(0.5)	0.10	(0.707)	4.5	89.5 ± 1.7	75.9 ± 1.4	72.8 ± 1.1
5	(0.5)	0.08	(0.5)	0.10	(−0.707)	3.5	87.4 ± 1.8	76.4 ± 1.8	77.1 ± 1.2
6	(0)	0.06	(−1)	0.04	(0)	4.0	51.1 ± 1.0	67.0 ± 1.6	83.4 ± 1.6
7	(0)	0.06	(0)	0.08	(0)	4.0	74.4 ± 1.2	80.0 ± 1.5	99.5 ± 1.8
8	(0)	0.06	(0)	0.08	(0)	4.0	73.3 ± 1.2	80.1 ± 1.6	99.2 ± 2.0
9	(0)	0.06	(0)	0.08	(0)	4.0	74.4 ± 1.5	79.6 ± 1.5	99.9 ± 1.9
10	(0)	0.06	(1)	0.12	(0)	4.0	100.0 ± 2.1	93.2 ± 2.0	95.8 ± 2.0
11	(−0.5)	0.04	(−0.5)	0.06	(0.707)	4.5	55.8 ± 0.9	64.5 ± 1.1	67.9 ± 1.6
12	(−0.5)	0.04	(−0.5)	0.06	(−0.707)	3.5	52.2 ± 1.0	69.8 ± 1.3	70.6 ± 1.9
13	(−0.5)	0.04	(0.5)	0.10	(0.707)	4.5	81.6 ± 1.6	75.4 ± 1.4	69.6 ± 2.1
14	(−0.5)	0.04	(0.5)	0.10	(−0.707)	3.5	81.1 ± 1.4	74.1 ± 1.2	73.0 ± 1.7
15	(−1)	0.02	(0)	0.08	(0)	4.0	53.8 ± 1.2	59.8 ± 1.0	63.8 ± 2.1

X_1: Triton X-100 concentration (mol L^{-1}); X_2: KCl concentration (mol L^{-1}); X_3: initial pH.

yellow [1], allura red [2,3], carmoisine and brilliant blue [12], malachite green [13], malachite green and crystal violet [14] in food samples.

A major role of experimental design in analytical chemistry concerns method optimizations and produce the best possible analytical performance. The multivariate procedure has the following advantages in relation to the univariate strategy [15]: (a) it is possible to know thoroughly the studied system, having a global knowledge of it in the whole experimental domain. From the results obtained, a mathematical model can be constructed to relate the response to the experimental conditions. The response for any point of the experimental domain can be predicted after an estimation of the coefficients of the model; (b) the number of experiments is smaller than the number of experiments required in the one at a time approach, reducing cost, effort and time; (c) it is possible to study the interactions between factors and the nonlinear relations with the responses; (d) generally, it is possible to find the absolute optimum in the studied domain, while the one at

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