LUMINESCENCE
The Journal of Biological and Chemical Luminescence

Microwave assisted extraction with micellar media for determination of fluoroquinolones in coastal marine sediments followed by HPLC with fluorescence detection

S. Montesdeoca–Esponda[1], Z. Sosa-Ferrera[1],
J. Santana-Rodriguez[1]
[1]University of Las Palmas de G.C., Chemistry, Las Palmas de G.C., Spain

Introduction

Contamination by pharmaceutical and personal care products (PPCPs) in sediments samples has recently been studied for the scientific community. Fluoroquinolones (FQs) are a class of antibiotics employed in human and animals medicine [1] that are accumulated in the wastewater treatment plants because there aren't mechanisms for remove it completely. Moreover, FQs are resistant to microbial degradation and may be persistent within environment [2].

Table 1. Experimental design for the optimization of extraction time, power, surfactant volume and concentration volume

RUN	POWER (V)	EXTRACTION TIME (min)	VOLUME (mL)	CONCENTRATION (%)
1	100	10	5	0,5
2	500	2	15	5
3	100	2	5	5
4	500	10	15	0,5
5	500	10	5	5
6	100	2	15	0,5
7	500	2	5	0,5
8	100	10	15	5
9	100	2	15	5
10	100	2	5	0,5
11	500	10	15	5
12	500	2	5	5
13	100	10	15	0,5
14	500	10	5	0,5
15	100	10	5	5
16	500	2	15	0,5

The aim of this work is to optimize a microwave assisted extraction method using micellar media as extractants (MAME) for the determination of FQs in different coastal marine sediment samples using high performance liquid chromatography with fluorescence detection.

Experimental

Chromatographic separation

A chromatograph system (C_{18} column) with fluorescence detector (excitation and emission wavelength 280 and 450 nm) from Varian (Madrid, Spain) was used. Analytes separation was carried out employed an isocratic mobile phase (15:85 v/v, methanol/water) at a flow rate of 1 mL.min^{-1}.

Microwave Assisted Micellar Extraction (MAME)

Microwave oven used was a Multiwave of Anton Paar (Graz, Austria).

Sediment samples were sieved to a particle size of lesser than 0.3 mm and we taken 2 g, which were spiked with a solution of FQs to obtain a concentration of each analyte of 2 µg/g. Then the sample was transferred to the vessel, the extractant agent was added and was subjected to the MAME process. The surfactants used as extractans were: Polyoxyethylene 10 lauryl ether (POLE), Hexaethylene glycol monododecyl ether ($C_{12}E_{6)}$, Hexadecyltrimethylammonium bromide (HTAB) and Sodium dodecyl sulphate, (NaLS). The extract solution was filtered through a 0.45 µm syringe filter.

The experimental design used for the optimization was obtained using Statgraphics Plus software 5.1 and the statistics study was done with SPSS 17.0.

Results

We optimize the extraction time, power, volume and concentration of surfactant with a 2^4 factorial design (Table 1) for researching the influence of each variable on the recovery and the variables correlation each other.

In the first experiments we observed that NaLS showed a very low amount of analyte extracted, so we not included it in following studies. In table 2 are shown the bivariate and partial correlations between each parameter for HTAB, which presents the major responses. The variable that more affects the process is the

Table 2. Relevant variables and correlation between variables using HTAB as extractant. The maxima correlations are +1 and −1

CORRELATION	Levofloxacin	Norfloxacin	Ciprofloxacin	Enrofloxacin	Sarafloxacin
Power	0.463	0.577	0.553	0.472	0.532
Time	0.157	0.339	0.346	0.024	0.045
Volume	–0.392	–0.251	–0.088	–0.308	–0.364
Concentration	0.449	0.968	0.331	0.521	0.465
Power/Concentration	–0.263	–0.265	–0.233	–0.327	–0.330
Time/Concentration	–0.080	–0.136	–0.129	–0.015	–0.024
Power/Time	–0.083	–0.255	–0.245	–0.013	–0.028
Volume/Concentration	0.214	0.097	0.031	0.198	0.205
Volume/Time	0.068	0.094	0.033	0.008	0.018
Volume/Power	0.0223	0.183	0.059	0.173	0.245

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