



Analytical method using SPE and LC-MS/MS for determination UV filters in aquatic matrices

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Abstract

Nowadays, UV filters are considered emerging contaminants, once they are present in environment due to anthropic action, and they can also be harmful to human health and environment. So, in order to analyze the presence of these target compounds in water, this work describes the development of a sensitive method for the determination of five UV filters in water samples using solid phase extraction (SPE) followed by liquid chromatography- tandem mass spectrometry (LC-MS/MS).

Key words:

Emerging contaminants, water, analytical chemistry

Introduction

Some UV filters are harmful to human health and environment, depending on their concentration and exposure time. In this work, five UV filters are studied. This project intends to do the validation of a sensitive analytical method using solid phase extraction followed by liquid chromatography- tandem mass spectrometry (LC-MS/MS) to quantify the target compounds in different sources of water in Metropolitan Region of Campinas.

Results and Discussion

The UV filters were analyzed by ESI-MS/MS in the positive mode by multiple reaction monitoring (MRM). The MRM specific conditions for each analyte are present in Table 1.

Table 1. MRM conditions for each analyte

Analyte	Precursor ion (m/z)	Product ions (m/z)	Collision energy (eV)
BP-3	229.1	105; 151.1	20; 20
4-MBC	255.3	105; 212.1	30; 30
OC	361.7	231.7; 249.7	21; 7
OD-PABA	278.3	151.1; 166.1	35; 20
EHMC	290.8	178.8; 160.7; 132.9	0; 10; 30

The ion source parameters were: temperature: 325 °C; ion spray voltage: 4000 V; curtain gas: 50 psi; gas flow: 11 mL/minute.

In the chromatographic method, the mobile phase was constituted by water (eluent A) and methanol (eluent B), with eluent A containing 0.1% (v/v) of formic acid. The gradient profile was as follows: 0 min, 60% B; 5 min, 80% B; 6 min, 90% B; 7 min, 90% B; 10 min, 60% B. The post run time was 5 minutes.

The solid phase extraction (SPE) was performed by using Oasis HLB cartridges (Waters, 60 mg) [1]. These cartridges were conditioned with 5 mL of methanol and 5 mL of water. Once conditioned, the Milli-Q water sample of 500 mL with pH=2 [1] was passed through the cartridge. After that, the cartridges were dried using a manifold and then the target compounds were eluted with 30 mL of methanol [1]. The eluate was collected and solvents were removed to dryness at night, because some UV filters can

degrade if they are exposed to light [2]. Finally, the residue was reconstituted with 500 µL of methanol: water (60:40, v/v). The average recovery with its respective RSD of each analyte is present in Table 2.

Table 2. Recovery for each analyte

Analyte	Recovery ± RSD (%)
BP-3	58 ± 1
4-MBC	43 ± 17
OC	33 ± 14
OD-PABA	46 ± 35
EHMC	14 ± 41

The limit of detection (LOD) and the limit of quantification (LOQ) [3] for each target compound are shown in Table 3.

Table 3. LOD and LOQ for each compound

Analyte	LOD (ng/L)	LOQ (ng/L)
BP-3	11	32
4-MBC	9	28
OC	9	27
OD-PABA	40	122
EHMC	11	32

Conclusions

A method based on SPE and followed by LC-MS/MS has been developing and the results are satisfactory until this moment, once the method is sensitive (low LOD and LOQ). Breakthrough, precision of the method and matrix effect for drinking water and surface water will be analyzed.

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