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Study of pharmaceuticals fate in the aquatic environment using microcosm experiments and SPE-UHPLC-MS/MS

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Abstract

Pharmaceuticals are considered contaminants of emerging concern (CEC). They may reach the aquatic environment by the discharge of treated sewages from wastewater treatment plants. The fate of CEC is still not completely understood and microcosm models has been employed for the evaluation of the fate and for ecotoxicological assays. Laboratory sediment/water microcosms allow a better understanding of biotic and abiotic processes involved in the fate of chemicals in water bodies. In this work, a multiresidue SPE-UHPLC-MS/MS method (LOQ 0.1 µg/L) was developed and validated to quantify three CEC (carbamazepine, sulfaquinoxaline and fexofenadine). The fate of these CEC was evaluated using a microcosm with sediment and surface water collected from the Rio Atibiaia.

Key words: *Pharmaceutical residues, microcosm, aquatic environment*

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Introduction

The main entrance of pharmaceuticals, or contaminants of emerging concern (CEC), in water resources is by the discharge of treated sewages form wastewater treatment plants (WWTPs). In general, the removal of pharmaceuticals in the WWTPs is not complete (Kümmerer, 2013). Therefore, it is important to understand the fate of these compounds when they reach the aquatic environment.

The aim of this study was to evaluate the fate of three pharmaceuticals, prioritized in a previois work: carbamazepine, CBZ; sulfaquinoxaline, SQX and fexofenadine, FXF in the aquatic environment, using microcosms (sediment and surface water of the Rio Atibaia). The determination of the CEC were carried out using ultra high performance liquid cromatography tandem mass spectrometry with on-line solid phase extraction (SPE-UHPLC-MS/MS). The method was developed and validated.

Hydrolysis, sorption onto sediment and biodegradation of the target CEC were evaluated in the microcosm using the experimental design presented in Figure 1.

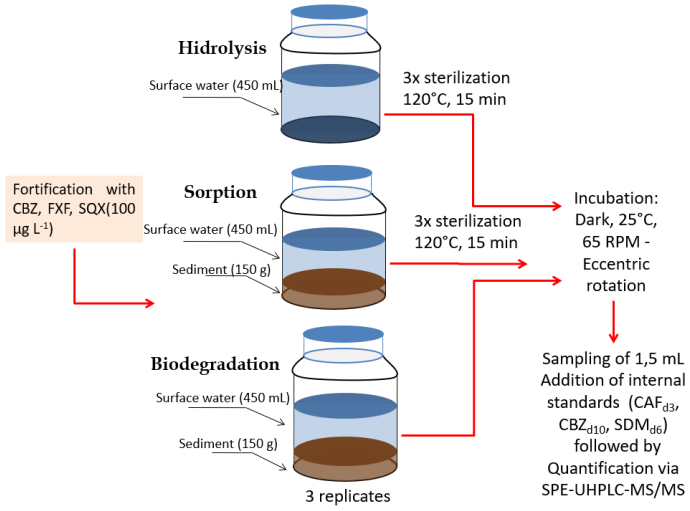

Figure 1. Experimental design.

Results and Discussion

The optimal analytical SPE-UHPLC-MS/MS conditions were as follows: SPE column XBridge C8 (10 µm), analytical column Acquity UHPLC HSS T3, sample loading solvent 0.1% aqueous formic acid, volume of loading solvent 0.2 mL and an injection volume of 200 µL. Formic acid 0.1%(v/v):methanol was the mobile phase (gradient elution), and the flow rate was 0.30 mL min^{-1}. Quantification was performed in selected reaction monitoring mode and sulfadimethoxine-d_6, caffeine-d_6 and carbamazepine-d_{10} were used as internal standards. The linearity was higher than 0.99 and the limit of quantitation of the method was set as 100 ng L^{-1}.

Abiotic process (hydrolysis and sorption) were not effective for the degradation of all three evaluated CEC during 10 days (25 ºC). Nevertheless, SQX and FXF underwent biodegradation (10 days, 25 ºC) in the microcosm containing a sediment:surface water ratio of 1:3.

Conclusions

The analytical method developed presented adequate validation parameters, including detectability to assess the fate of CBZ, SQX and FXF in the microcosm. Carbamazepine did not degrade under abiotic and biotic processes, indicating persistence in the aquatic environment. Abiotic processes did not contribute to the degradation of the three evaluated pharmaceuticals.

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