Extraction and quantification of pharmaceutical drugs in aqueous matrices

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Introduction

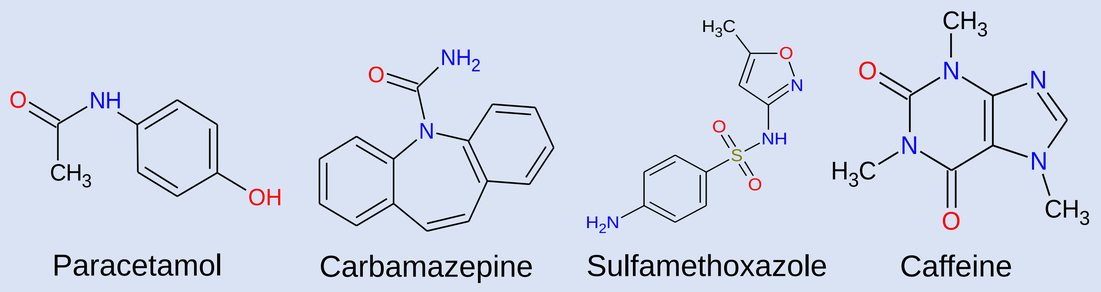
The increasing production and use of chemical compounds, coupled with inefficient sewage treatment systems, results in an inadequate release of all types of pollutants into the environment.

Emerging pollutants are potentially toxic substances normally found in very small concentrations but that can produce harmful effects on the environment. These compounds are not yet included in water quality monitoring programs nor in environmental control legislation standards.

Pharmaceutical and Personal Care Products (PPCPs) are an important group of emerging pollutants due to their continuing increase in world consumption and their inherent capacity to induce physiological effects at very low doses, raising concerns about potential adverse effects in humans, animals and environmental systems.

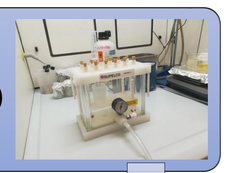
Pharmaceutical drugs

This study includes four pharmaceutical drugs that belong to four different pharmacological classes, namely, an analgesic (paracetamol), one antibiotic (sulfamethoxazole), an anticonvulsant (carbamazepine) and a central nervous system stimulant (caffeine).



Experimental Methodology

Optimization of Solid Phase Extraction method (SPE)



Development of a method for detection and quantification by High Performance Liquid Chromatography (HPLC-DAD) in aqueous matrices

Validation of the analytical method of extraction and quantification using real samples

Results

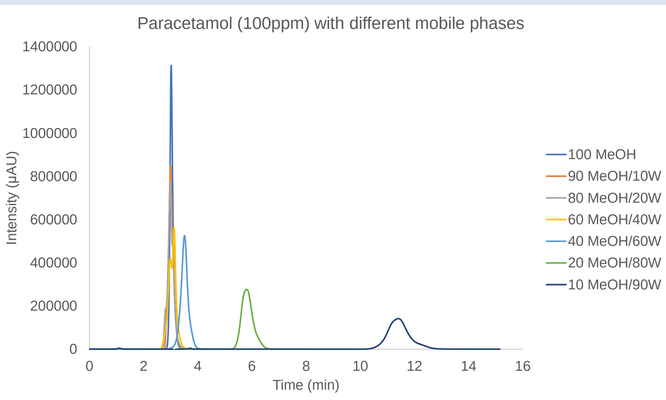


Figure 1. Effect of the mobile phase composition on retention time and dispersion for HPLC-DAD analysis of paracetamol.

Table 1. HPLC-DAD solvent gradient used for the separation of the pharmaceutical compounds.

Time (min)	% MeOH	% Water	% DEA	Flow rate (mL/min)
0	35	65	0,005	1
5	75	25	0,005	

With the results of Figure 1 it can be concluded that the higher the amount of water the higher the retention time and dispersion.

With these results a gradient was created so that all the compounds were separated and the retention time was shorter (Table 1).

After selection of the best gradient, all compounds were identified as well as their retention time and the optimum wavelength (Table 2) that allows the maximization of the detector signal.

Table 2. Optimal wavelength and retention time for the compounds under study.

Compound	"Optimal" wavelength (nm)	Retention time (min)
Sulfamethoxazole	255	1,673
Paracetamol	245	3,350
Caffeine	272	4,870
Carbamazepine	280	8,503

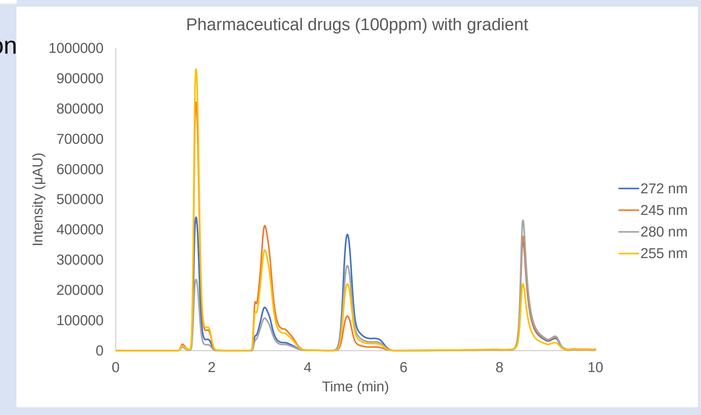


Figure 2. Chromatograms obtained by HPLC-DAD for the mixture with compounds under study (100 ppm) at their "optimal" wavelength with the gradient of Table 1.

On-going Work

- Calibration Curves, Limits of Detection and Limits of Quantification;
- Experimental Measurements of SPE Recoveries;
- Method Validation using real aqueous samples.

References

1. Raimundo, C. C. M., "Contaminantes emergentes em água tratada e seus mananciais: sazonalidade, remoção e atividade estrogénica", **2011**, *Tese de Doutoramento*, Universidade Estadual de Campinas*, Brasil.*

2. Ebele A. J.; Abdallah M. A.; Harrad S., "Pharmaceuticals and personal care products (PPCPs) in the freshwater aquatic environment", *Emerging Contaminants* , **2017**, vol. *3*, issue 1, 1-16**.**