



Ultra-high performance liquid chromatography with fluorescence detection as a powerful analytical tool for the analysis of oestrogens in environmental waters

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Introduction

Oestrogens are naturally excreted by mammals and are also widely used as pharmaceuticals in many therapeutic treatments¹ which means that they could reach aquatic compartments through wastewaters producing changes in aquatic biota, even at trace concentrations². In this work we present different studies which use Ultra-high performance liquid chromatography with fluorescence detection (UHPLC-FD) for the optimization of extraction and preconcentration processes as well as to determine the levels of some hormones in water samples.

Materials and Methods

In a first study, the optimization of a solid phase extraction (SPE) procedure is shown. In this study, several parameters of SPE as cartridge type, sample volume, ionic strength and pH of the sample or elution volume were optimized following a factorial experimental design. Chromatographic and detection parameters such as chromatographic gradient or excitation and emission wavelengths were also optimized. In this work, the separation and detection of the four oestrogens (estriol, 17 β -estradiol, 17 β -estradiol glucuronide and 17 α -ethynylestradiol) were performed in less than 7 minutes.

In the second work, it was studied the use of molecularly imprinted polymers (MIPs) as sorbent in solid phase extraction. This type of sorbents is designed using a molecule as a template and for that reason, only analytes with a similar structure of the template molecule could be adsorbed, which means that the selectivity is greater in comparison with conventional polymeric sorbents. All the variables involved in the extraction process were optimized and the results were compared with those obtained for traditional sorbents.

Results and Discussion

In the optimization of the conventional SPE method, it was observed that the highest recoveries were using C₁₈ cartridges and also it was checked that ionic strength of the sample had a negative influence in the extraction process. The optimized SPE-UHPLC-FD method showed very satisfactory analytical parameters and a high preconcentration factor of 125.

Regarding the optimization of molecular imprinted solid phase extraction (MISPE), it was observed that the selectivity of the cartridges was really good. The optimized MISPE-UHPLC-FD method was applied to real wastewater samples from a wastewater treatment plant and from a veterinary hospital. The analytical parameters obtained were really good, with recoveries which were over 60% and detection limits which ranged from 0.18 to 0.45 ng·mL⁻¹.

Conclusion

The development of UHPLC-FD methods could be used to evaluate the presence of steroid hormones in environmental samples, achieving appropriate analytical parameters and quantification limits. Moreover, UHPLC-FD technique could be also very useful in the optimization and development of extraction and preconcentration procedures which are absolutely essential to quantify trace levels of this type of pollutants in environmental systems.

Bibliography

¹ R. Guedes-Alonso, *et al.*, Liquid chromatography methodologies for the determination of steroid hormones in aquatic environmental systems, *Trends Env. Anal. Chem.*, 2014, 3, 14-27

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