OPTIMIZATION OF MICROWAVE ASSISTED EXTRACTION COMBINED WITH UHPLC WITH FLUORESCENCE DETECTION FOR THE DETERMINATION OF ESTROGENS IN SLUDGE SAMPLES

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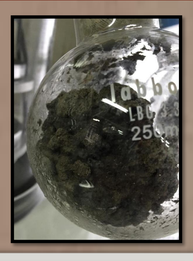
INTRODUCTION

Emerging contaminants are a group of substances that has been detected at very low concentrations in the environment .Within this group of pollutants, this work is focused specifically on estrogens. Estrogens are a type of hormones that are proved to be endocrine disruptors. The conventional purifying treatments do not completely eliminate them and they are been adsorbed in the sludge [1].
New methodologies for extraction and preconcentration are required because these type of compounds are usually detected at trace level concentrations. Microwave assisted extraction (MAE) is a rapid technique which uses minimum volumes of solvents and it allows the extraction of analytes in complex matrices such as sludges [2]. In this work, it has been optimized a microwave assisted extraction combined with ultra-high performance liquid chromatography with fluorescence detection (UHPLC-FD) for the determination of a group of for estrogens in sludge samples from wastewater treatment plant.

EXPERIMENTAL DESIGN


Sludge → freeze-dried sludge → 100 mg freeze-dried sludge

2^3

- P= 100 W, 500 W
- V= 2 ml, 5 ml
- t= 2 min, 15 min

	E3	E2	EE	E2-3ME
Power (W)	-0,054	-0,160	-0,020	-0,178
Time (min)	0,167	0,140	0,006	0,346
Volume (ml)	0,937	0,802	0,887	0,765
Pow x Vol	0,145	0,214	0,038	0,215
Pow x t	-0,499	-0,489	-0,5	-0,475
Vol x t	-0,456	-0,190	-0,012	-0,439

Less influence

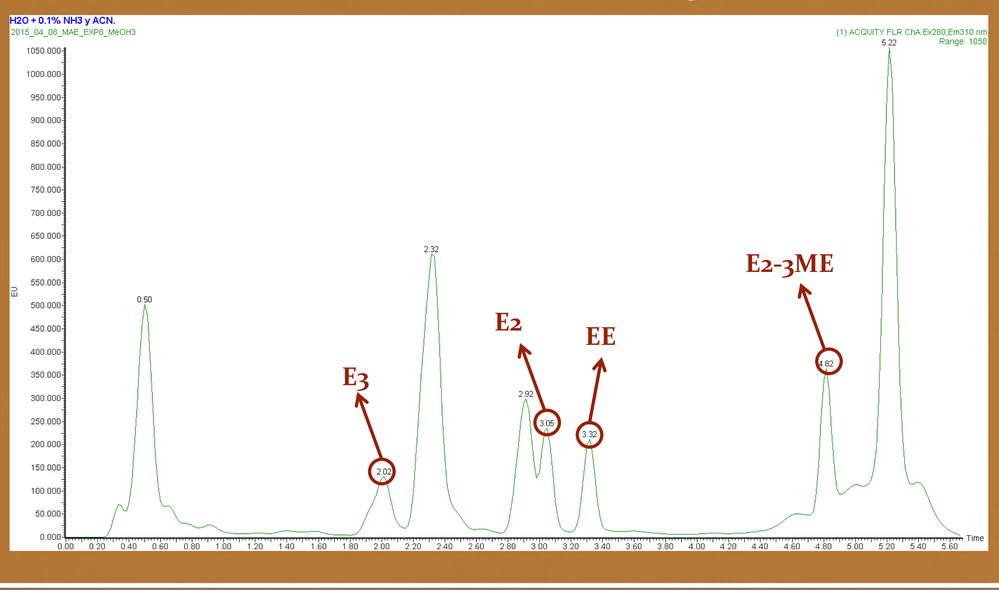
Greater influence

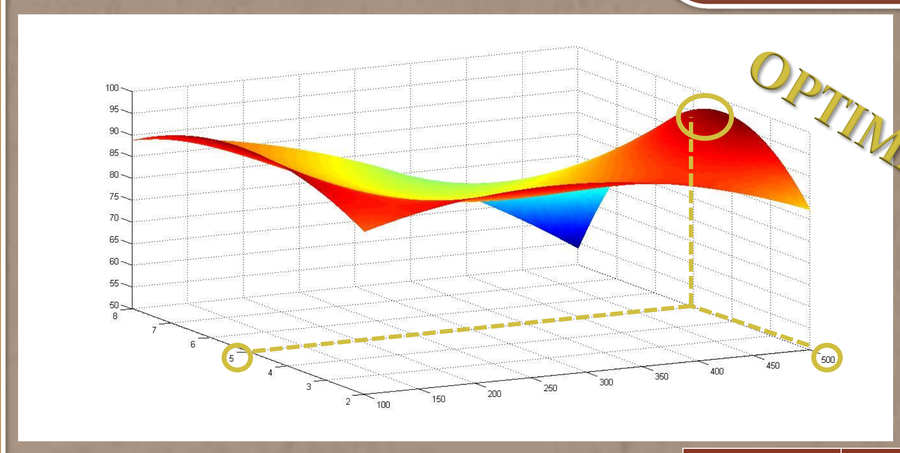
3^2

t\Pow	100 W	300 W	500 W
2 min	●	●	●
5 min	●	●	◉
8 min	●	●	●

UHPLC-FD CONDITIONS

- C_{18} column
- Phase A: Mili-Q water with 0,1% NH_3
- Phase B: ACN
- Gradient mode
- Excitation wavelength: 280 nm
- Emission wavelength: 310 nm





	RSD (%)	
	Interday	Intraday
E3	13,44	22,19
E2	17,85	18,16
EE	16,53	20,53
E2-3ME	18,05	11,04

CONCLUSIONS

- A MAE-UHPLC-FD method for the determination of estrogens in sludge have been developed.
- All variables affecting the process such as time, power, volume of solvent and solvent, have been optimized.
- The Optimal Conditions were 10 ml of MeOH, 500 W and 5 min.
- Recoveries were around 50%.
- Interday RSD are below 20%, whereas Intraday RSD are between 20 – 30%.
- The method is suitable for determination of estrogens in sludge.

REFERENCES

1. Guedes-Alonso, R., Montesdeoca-Esponda, S., Sosa-Ferrera, Z., Santana-Rodríguez, J.J., 2014. Trends Environ. Anal. Chem. 3–4, 14–27.
2. Vega-Morales, T., Sosa-Ferrera, Z., Santana-Rodríguez, J.J., 2013. Water. Air. Soil Pollut. 224, 1–15.

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