

ANALYSIS OF HORMONES IN SLUDGE SAMPLES USING MICROWAVE-ASSISTED EXTRACTION AND ULTRA-HIGH PERFORMANCE LIQUID CHROMATOGRAPHY TANDEM MASS SPECTROMETRY

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Introduction

Steroid hormones are natural and synthetic compounds which are considered as endocrine disruptor compounds due to their characteristics and possible toxic effects over aquatic biota [1]. Wastewater treatment plants (WWTPs) are the major sources of these compounds into the environment and because of their non-polar nature, they can be adsorbed on solid matrices.

Nowadays, studies about determination of steroid hormones in solid samples are scarce in comparison with studies in liquid samples. However, as happen in liquid samples, concentrations of steroid hormones in solids are very low and it is necessary the development of methodologies of extraction and preconcentration which permits the determination of this kind of micropollutants at measurable concentrations [2]. Microwave-assisted extraction (MAE) is an easy technique which allows the extraction of analytes in complex matrices using small solvent volumes and short extraction times [3].

Instrumental



Anton Paar Multiwave Microwave



Waters ACQUITY UHPLC-MS/MS

Chromatographic and detection conditions

Separation in gradient mode

Column: ACQUITY BEH C18 (50x2.1 mm, 1,7µm)

Detection mode:

- ESI – for oestrogens
- ESI + for androgens, progestogens, glucocorticoids

| Time (min) | %A Water + 0.1 NH ₃ | %B Methanol |
|------------|-----------------------------------|----------------|
| 0.00 | 80 | 20 |
| 2.75 | 25 | 75 |
| 3.75 | 0 | 100 |
| 6.00 | 80 | 20 |

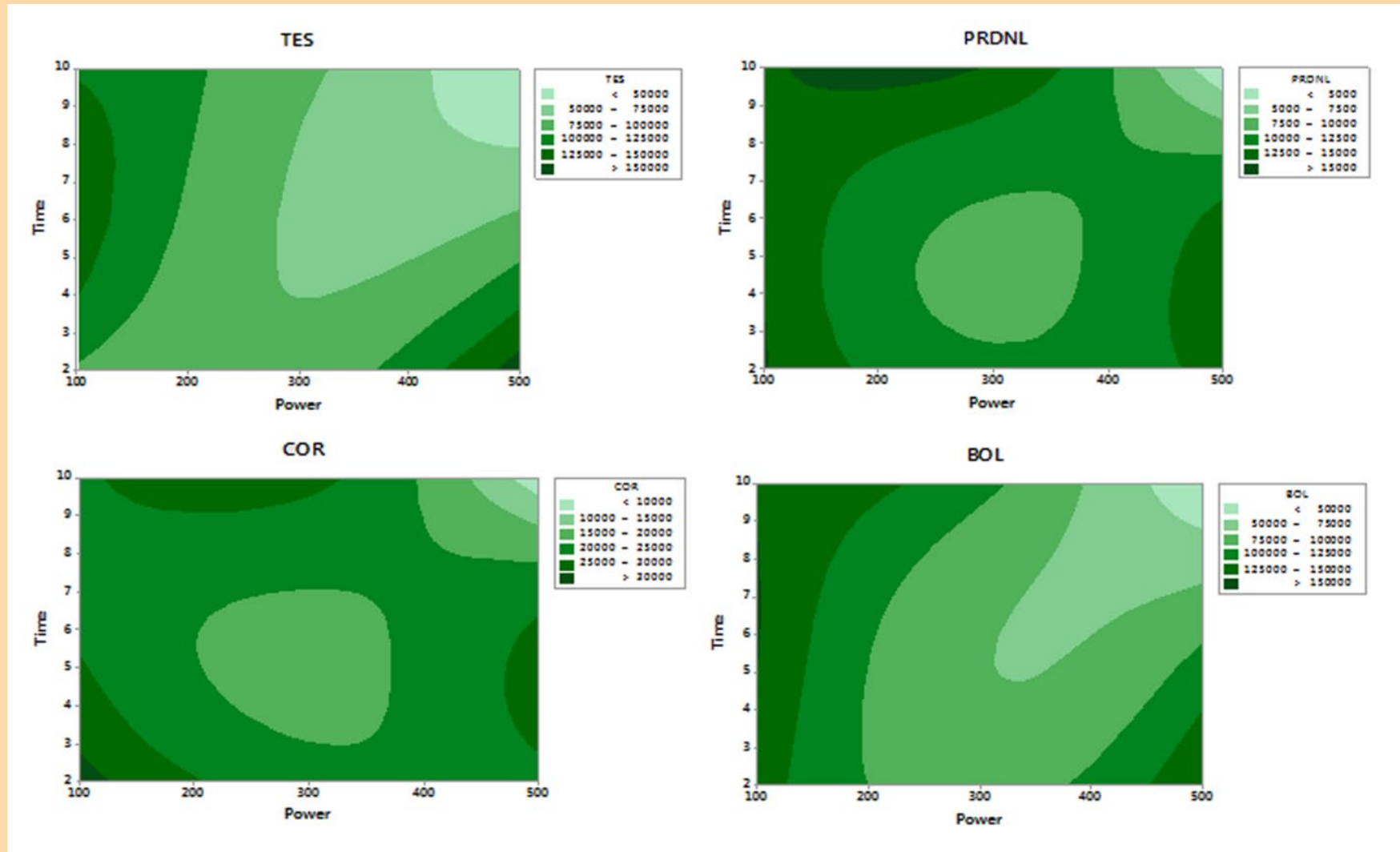
Experimental

Results

MAE Optimization

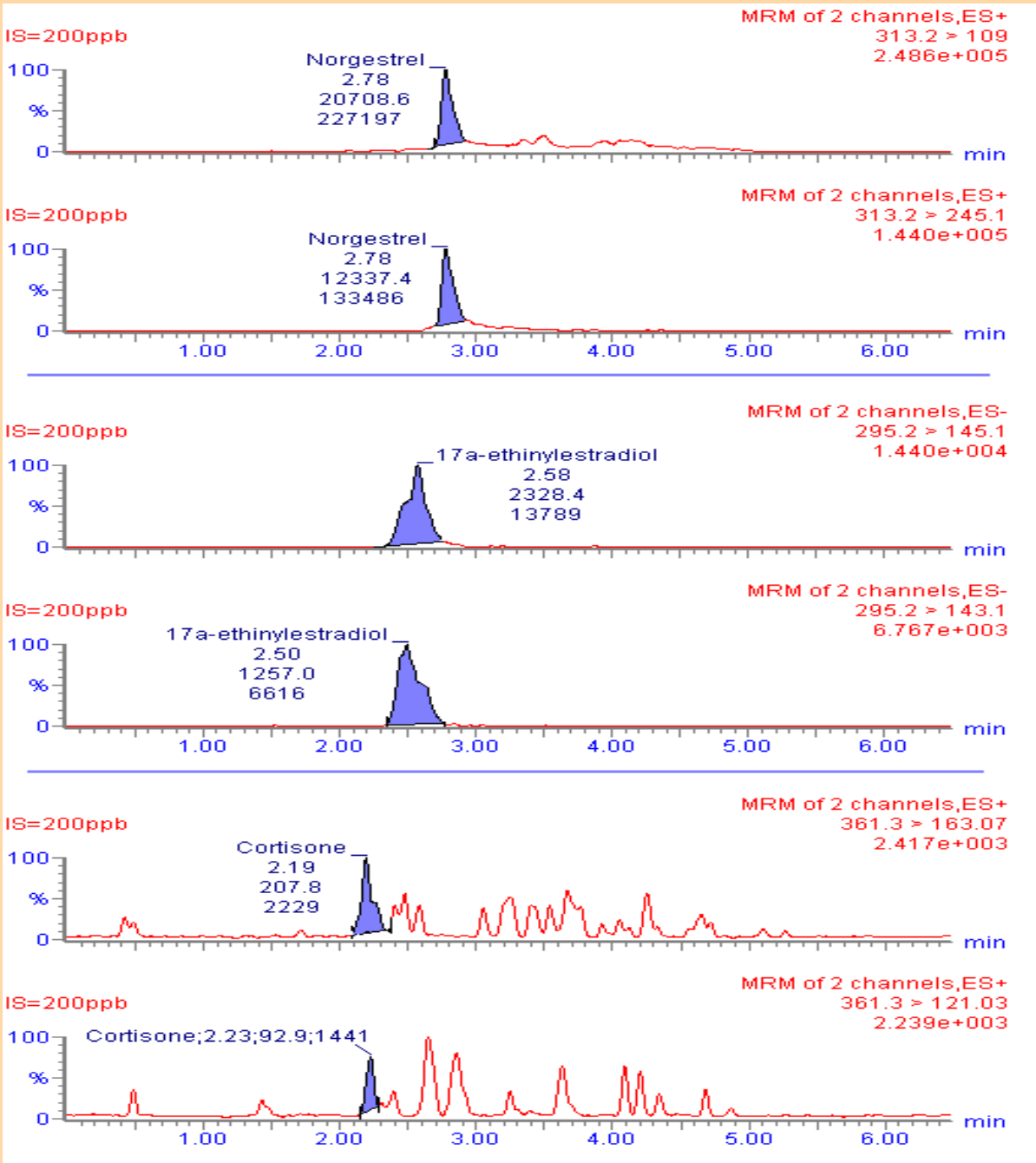
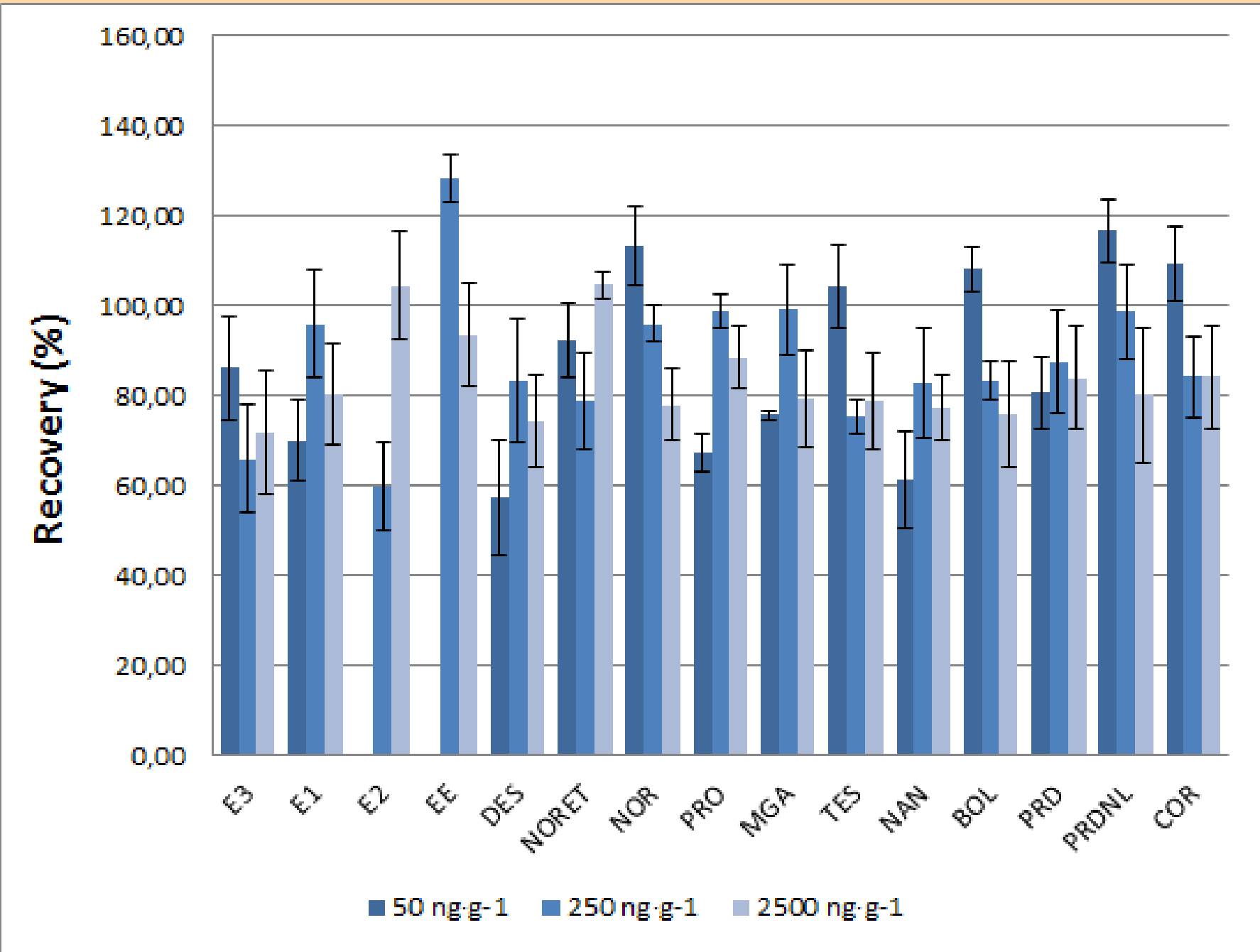
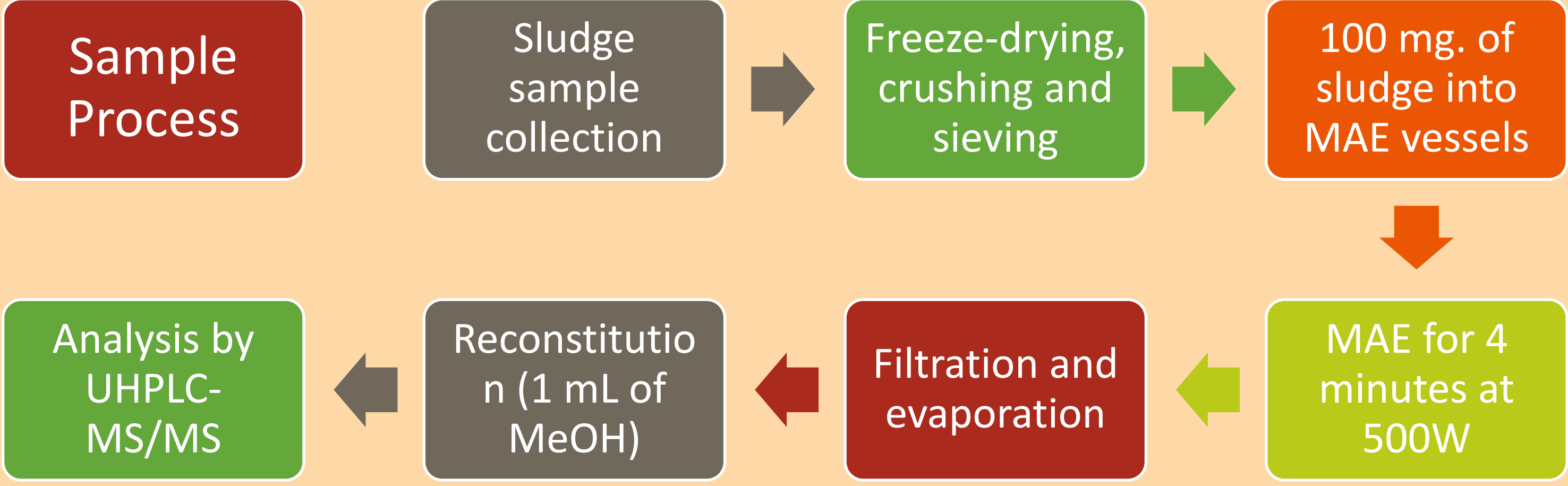
Experimental design

Power: 100-500W
Extraction time: 2-10 min
Extractant volume: 5-10 mL



Selective and sensitive method

LODs: 1.11 to 7.90 ng·g⁻¹
RSDs < 21%



Conclusions

A **MAE-UHPLC-MS/MS** method for the determination of **sex hormones and corticosteroids** in sludge samples is presented for **the first time**. The method has **appropriate detection limits**, shows **good selectivity and reproducibility** and It has been **satisfactory applied** to real samples.

Satisfactory applied to real samples

Norgestrel
430-1350 ng·g⁻¹
17α-ethynylestradiol
31.5-1440ng·g⁻¹
Cortisone
17.3 ng·g⁻¹

References

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2. R. Guedes-Alonso, S. Montesdeoca-Esponda, Z. Sosa-Ferrera, J.J. Santana-Rodríguez, Trends Environ. Anal. Chem. 3–4 (2014) 14–27.
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