Hydro Nation Scholarship

Simultaneous Determination of PPCPs and EDCs in Waters by SPE-GC-MS and LC-MS/MS

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Introduction

➢The ubiquitous occurrence in the aquatic and their adverse effect of Endocrine Disrupting Chemicals (EDCs) and Pharmaceutical and Personal Care Products (PPCPs) on animal and human health have garnered global attention.

➢Totally, twenty two compounds including PPCPs and EDCs were selected as target compounds in this study.

Research Objective

➢ The objective of this study was to develop fast and reliable methods for the simultaneous determination of selected PPCPs and EDCs in waters.

Conclusion

- ✓ The satisfactory recovery rates were achieved by SPE when Oasis HLB cartridge showed higher enrichment with ethyl acetate/acetone as optimal extraction solvents
- ✓ Analytical methods for the simultaneous determination of various 22 PCPPs and EDCs were developed by LC-MS/MS while GC-MS achieved 11 analytes. pH value 2 was validated to be the best condition for these 11 compounds in GC-MS
- ✓ Compared to GC-MS, LC-MS/MS methods showed better performance in a wider range of PPCPs with lower LOD values

Results and Discussion

A. Sample pre-treatment technique – Solid phase extraction

✓ **Effect of SPE cartridge type**

Oasis HLB cartridges showed higher enrichment efficiency of target analytes (47%-119%) from waters than the other SPE cartridges i.e. Strata X, Supelclean and ENVI-18.

✓ **Effect of elution solvents**

Acetone/ Ethyl acetate (v/v 50/50) was selected as optimal elution solvent with satisfactory recoveries for EDCs (84%-95%) and PPCPs (67%-117%)

B. Instrumentation analytical methods – GC-MS and LC-MS/MS

✓ **Liquid Chromatography and MS/MS Observations**

- A good separation of 14 pharmaceuticals chemicals was achieved in the positive ionization mode (in red) as shown in table 1.
- 8 chemicals (5 EDCs and 3 pharmaceuticals) were detected in the negative ionization mode (in blue)

Antidiabetics	Metformin
ß-Blockers	Atenolol
NSAID	*Paracetamol
	Ketoprofen
	*Ibuprofen
Antibacterials & Antiinfectives	Trimethroprim
	*Triclosan
	*Diclofenac
Fluoroquinolone Antibiotic	Levofloxacin
	Ciprofloxacin
Analgesics	*Tramadol
	Azithromycin
	Erythromycin
	Clarithromycin
Antiepileptics	Carbamazepine-10-11-epoxide
Anti-convulsant	*Carbamazepine
SSRI	Fluoxetine
Chemical Additive	*Bisphenol A
Natural Hormone	*Estrone
	*17β-Estradiol
	*Estriol
Synthetic Hormone	*17α-Ethynylestradiol

Table1. Target compounds list
SSRI-selective serotonin reuptake inhibitors
NSAID-Nonsteroidal anti-inflammatory drugs

- Instrument parameters i.e. mobile phase, column temperature, precursor ions, optimal fragment voltage and collision energies were optimized

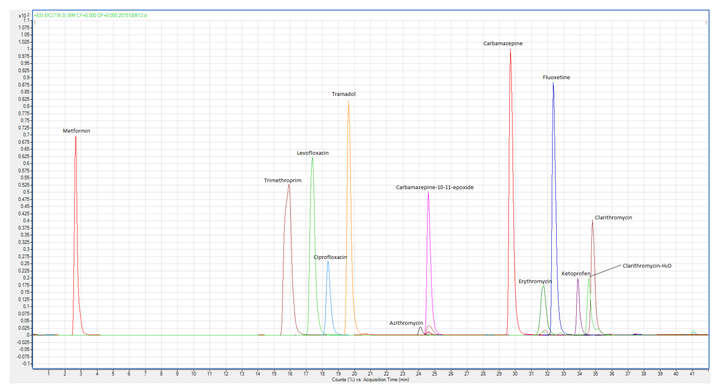

Figure1. Liquid chromatography of 12 target compounds

✓ **Gas Chromatography and MS Observations**

- A good separation of 11 chemicals (5 EDCs, 6 pharmaceuticals marked as*) was achieved.

✓ **Effect of pH**

- pH value 2 was validated to be the best condition for these 11 compounds recoveries (76%-145%)

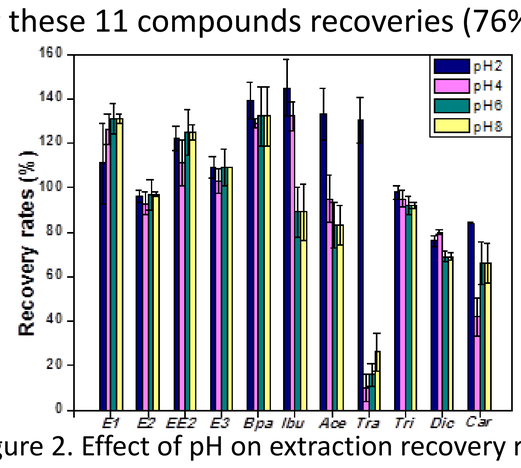

Figure 2. Effect of pH on extraction recovery rates

C. Method performance and comparison

✓ **Detection performance**

- LC-MS/MS methods achieved simultaneous determination in a wider range of PPCPs (22 analytes in total) while a good separation of 11 analytes in GC-MS was attained

✓ **Limit of detection comparison**

- LOD of GC-MS ranged from 0.48 to 2.27µg/L, indicating environmental water concentrations (e.g. 100 mL) of 0.36-1.73 ng/L can be detected by using SPE concentration step
- The instrumental LOD values of LC-MS/MS ranged from 0.03 to 0.39µg/L (methods LOD 0.002-0.030ng/L), which provided reliable and applicable tools for the further investigation of target compounds

crew

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Acknowledgements

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