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How important is the sample preparation step for non-target screening of micropollutants in urban waters?

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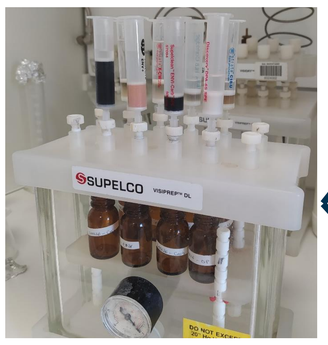
Introduction

Non-target screening (NTS) has gained interest in recent years for environmental monitoring purposes because it enables the **analysis of a large number of pollutants** without predefined lists of molecules. However, **sample preparation methods are diverse**, and few have been systematically compared in terms of **amount and relevance of the information** obtained by subsequent NTS analysis.

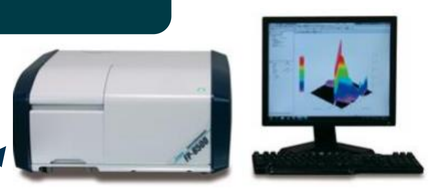
Objectives

- **Testing different natures of phases for solid phases extraction (SPE)** to retain a large range of micropollutants on real samples
- Developing a strategy based on different types of **relevant indicators to compare the results in NTS**
- Applying the developed strategy on **urban water samples**

Methods



Panel of **9 commercially-available** (HLB, C18, X-AW, X-CW, SDBL, ENV+, C18/ENV+, X-A, X-C) stationary phases + 1 **home-made multilayer** cartridge



UV at 254 nm + Excitation [240-450 nm] - Emission [250-600 nm] matrix (EEM)

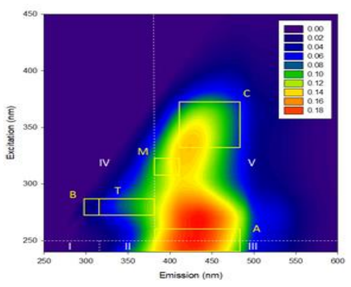
UV-vis & 3D fluorescence
Organic matter retention

C18 chromatography and ESI + with m/z [100-1000]

HRMS analysis
Number and properties of retained features

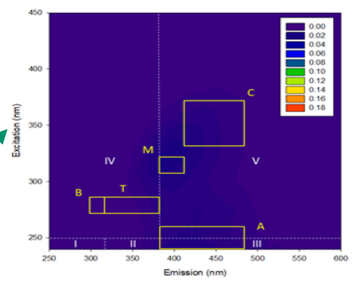
Results

Evaluation of the retention ability

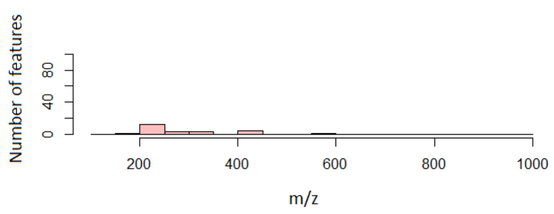


EEM before extraction

UV_{254} retention = 100%

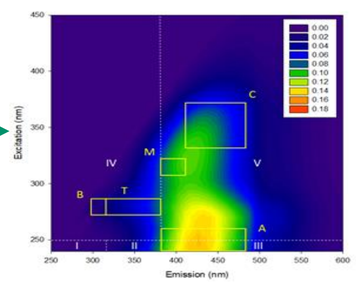


EEM after SPE on X-AW

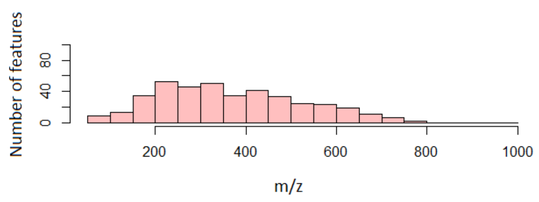


Distribution of HRMS features retained on X-AW

UV_{254} retention = 32%

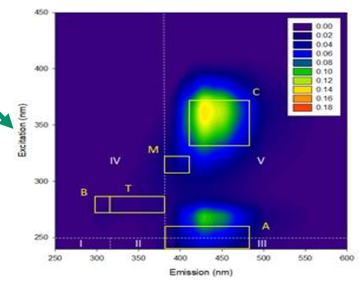


EEM after SPE on HLB

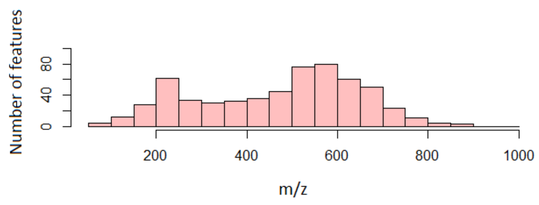


Distribution of HRMS features retained on HLB

UV_{254} retention = 90%



EEM after SPE on Multilayer (HLB, ENV+, X-AW, X-CW, based on *Kern et al., 2009*)



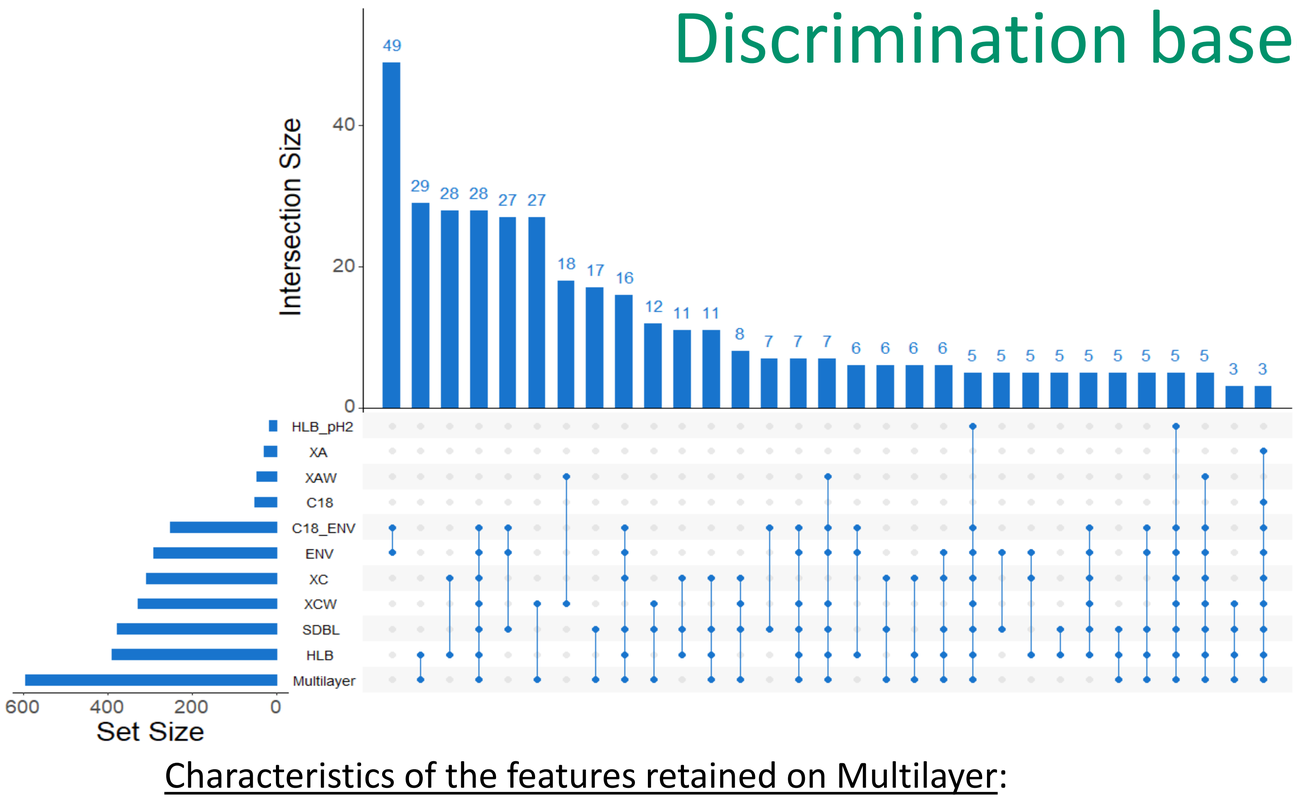
Distribution of HRMS features retained on Multilayer

Based on EEM spectra, regions II and IV were well retained by **X-AW and Multilayer** (≥ 80%). Those regions can be associated with **simpler, more polar and nitrogenous compounds** (*Zhang et al., 2008*). These cartridges seem to be the most efficient to retain **hydrophobic materials** as well as **smaller and more polar** compounds for a global screening of the molecules present in the sample.

X-AW exhibits **high retention of fluorescent materials but few features in HRMS** which is the total opposite of HLB.
This can be associated with a higher **retention of dissolved organic matter** which could contribute to **a competition** with the retention of organic compounds.

However, **Multilayer** does not seem to suffer such matrix effects, **retaining both high content of fluorophores and organic compounds.** Moreover, Multilayer retains molecules with a **wider range of molecular weight**, making it interesting for the **screening of a large range of micropollutants.**

Discrimination based on HRMS features



Multilayer is the cartridge with the **highest number of specific features** (323 features only found in this cartridge), but also the one with the **highest number of intersected features** (20 intersections with features find on other tested cartridges for a total of 212 features intersected), making it the most interesting cartridge for non-target studies by **retaining the most extensive set of features** (*i.e.*, with diverse properties).

Characteristics of the features retained on Multilayer:

- 594 retained features = 47 % **more than HLB** which is often considered as an **universal cartridge**
- Average m/z = 470.6467 (on a range from 100 to 1000) which shows its **ability to retain molecules with low and high molecular weight together**
- Average retention time = 47% of the chromatographic gradient, which shows that the compounds retained have a **large range of polarity**

Conclusions

The **Multilayer** cartridge (HLB, ENV+, X-AW, X-CW):

- is the most effective at retaining a **large range of HRMS features**
- is **less affected by the adsorption of dissolved organic matter**
- takes **advantage** of the **four phases** in terms of **diversity of compounds**

Ongoing investigation: **better characterize the chemical space** (log Kow, pKa, chemical functions…) covered by each cartridge. This could help determining the **relevance of each type of cartridge** towards specific purposes (*e.g.*, focus on more polar compounds).

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