Organic matter digestion methods for microplastic extraction from estuarine samplings

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CONTEXT OF THE STUDY

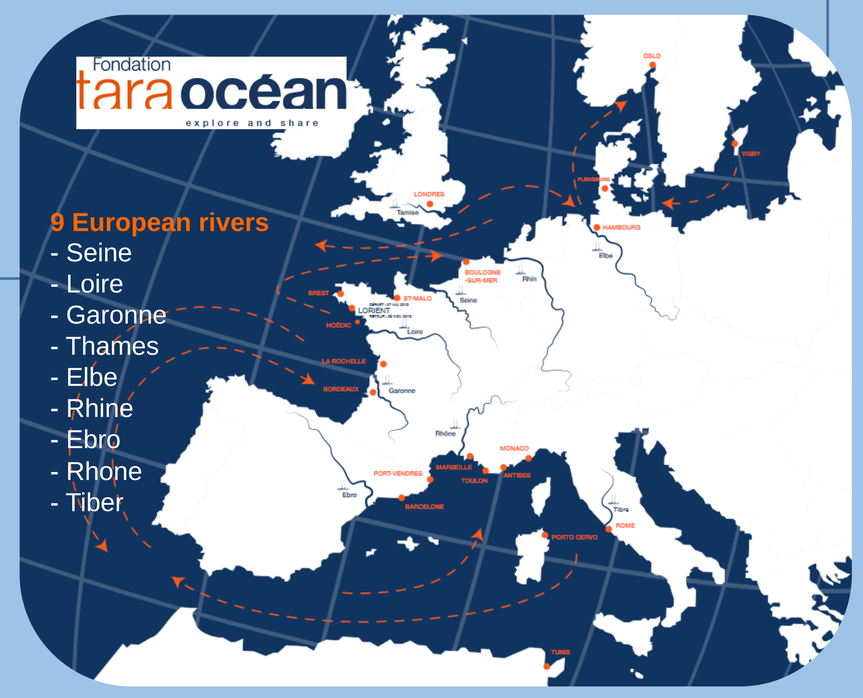
The TARA Microplastics 2019 [1] mission aims to investigate plastic pollution in European rivers across different scientific fields of study:

Plastic chemistry | **Physical oceanography** | **Marine biology**

Among the 19 partner laboratories, the IRDL objectives are:

- **Quantify the microplastic pollution,**
- **Identify the chemical nature of microplastics (MP).**

The very first observation is the **presence of plastic particles in all the sampled sites** during the mission.



ISSUES

- About **45 samplings** using **a 300µm manta trawl:** a large number of samples with **high concentrations of organic matter (OM)** of **various types** (algae, leaves, branches, etc.)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How to **efficiently** extract MP from a **large number** of field-collected samples while **preserving MP integrity**?

MATERIALS AND METHODS

① In-lab "artificial" samples were produced by **mixing MP from the most common polymers** in the environment **with complex organic matter**.

② To remove OM, two digestion protocols using hydrogen peroxide (H_2O_2) were tested.

KOH followed by H_2O_2
Digestion in two successive steps:
KOH (10%, 40°C, 48h) then H_2O_2 (30%, room T°, overnight) (~72h in total)

Fenton's reagent [2]
H_2O_2 (30%) with an iron catalyst: $FeSO_4$. $7H_2O$
T°C <40°C (in ice bath) (1 to 4h)
Iron oxides rinsed with HCl (1 M)

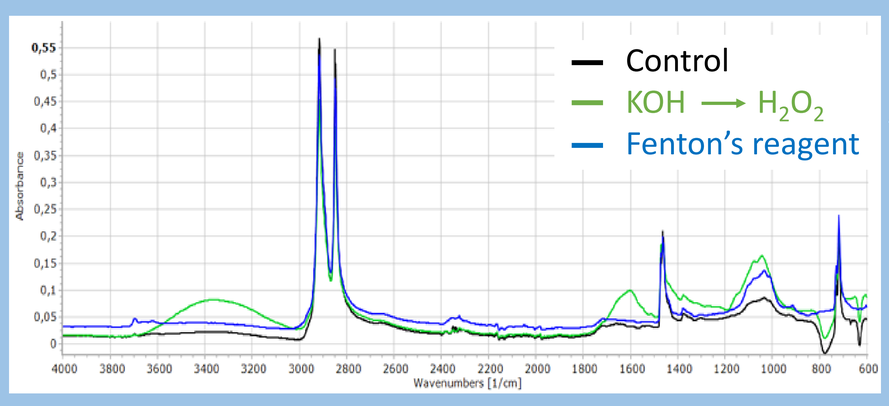
Both protocols were completed with a density separation step to remove inorganic matter (clay, sand, etc.).

③ Protocol selection criteria

- ✓ Effects on microplastics
  - Physical integrity : Weight comparison & visual inspection under binocular magnifier
  - Chemical integrity : Identification by infrared spectroscopy (ATR-FTIR)
- ✓ Organic and inorganic matter removal efficiency (dry weight before and after digestion and density separation)
- ✓ Time efficiency, ability to process a large number of samples

RESULTS AND DISCUSSION

- Weighting and visual inspection of MP showed that both protocols maintain MP physical integrity.
- Both protocols allowed the **identification** of the chemical nature of the polymers **by infrared spectroscopy**.



ATR-FTIR spectra of a PE microplastic before and after organic matter removal

	KOH → H_2O_2	Fenton's reagent
Effect on MP • Physical integrity • Chemical integrity	 + + + + +	 + + + + + +
Organic and inorganic matter removal efficiency	81%	89%
Time efficiency	+ (~72h in total)	+ + + (~4h)
Technician involvement	Minimum assistance	Full assistance

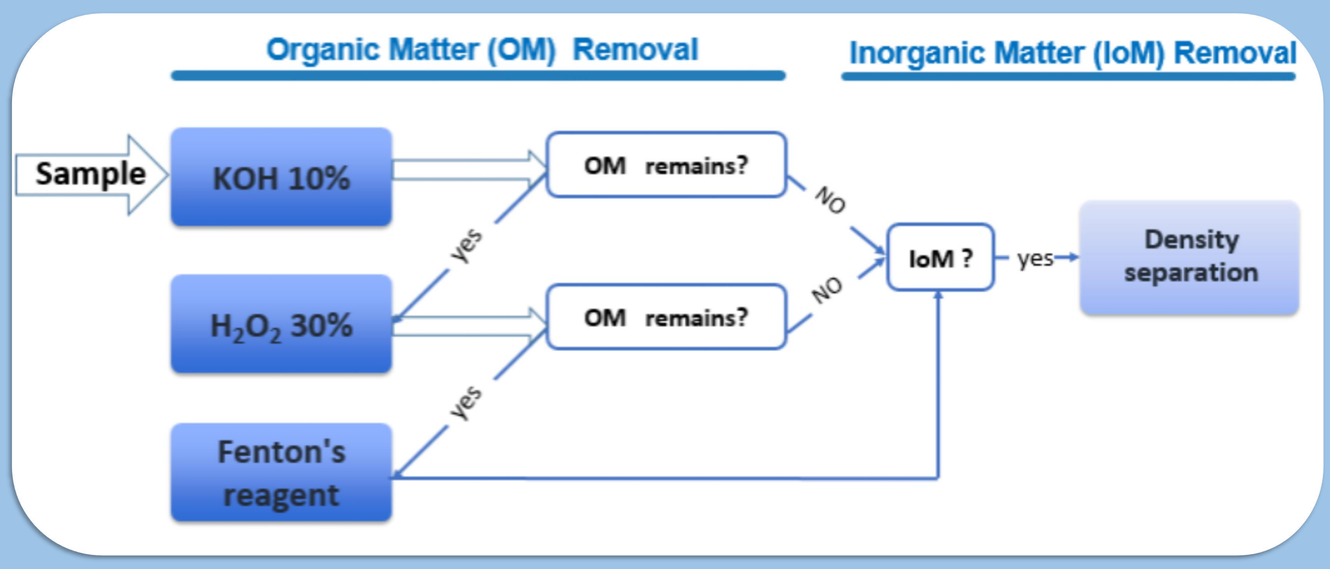
Although the Fenton's reagent is faster and slightly more efficient at digesting OM, it requires full assistance, hence the simultaneous processing of multiple samples is limited.

CONCLUSION

Given the wide variety of the Tara 2019 mission samples, a new protocol was necessary **to adapt to the varying concentrations and natures of OM**.

Taking in consideration the need to **efficiently process a large set of samples**, a new protocol is suggested. Firstly, all samples undergo a KOH digestion. Depending on the amount of remaining organic matter, more advanced digestion methods with H_2O_2 may be applied successively.

This new protocol is more time efficient, avoids unnecessary steps and allows the processing of a high number of samples.



[1] Tara's Blue Book: Tracing the Origins of Plastic Pollution (2020), 69 p.
[2] Hurley, R. R., Lusher, A. L., Olsen, M., & Nizzetto, L. (2018). Validation of a Method for Extracting Microplastics from Complex, Organic-Rich, Environmental Matrices. Environ. Sci. Technol., 52(13), 7409–7417.