8 – 10 November, TFMM workshop :
Towards a harmonized approach for atmospheric monitoring of Chemicals of Emerging Concern (CECs)

IMT Nord Europe
École Mines-Télécom
IMT-Université de Lille

Methodological approaches to the monitoring of atmospheric microplastics in France.

08
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2023

Liselotte Tinel
Liselotte.tinel@imt-nord-europe.fr

ISO 24187: Principles for the analysis of microplastics present in the environment (September 2023)

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Definitions:

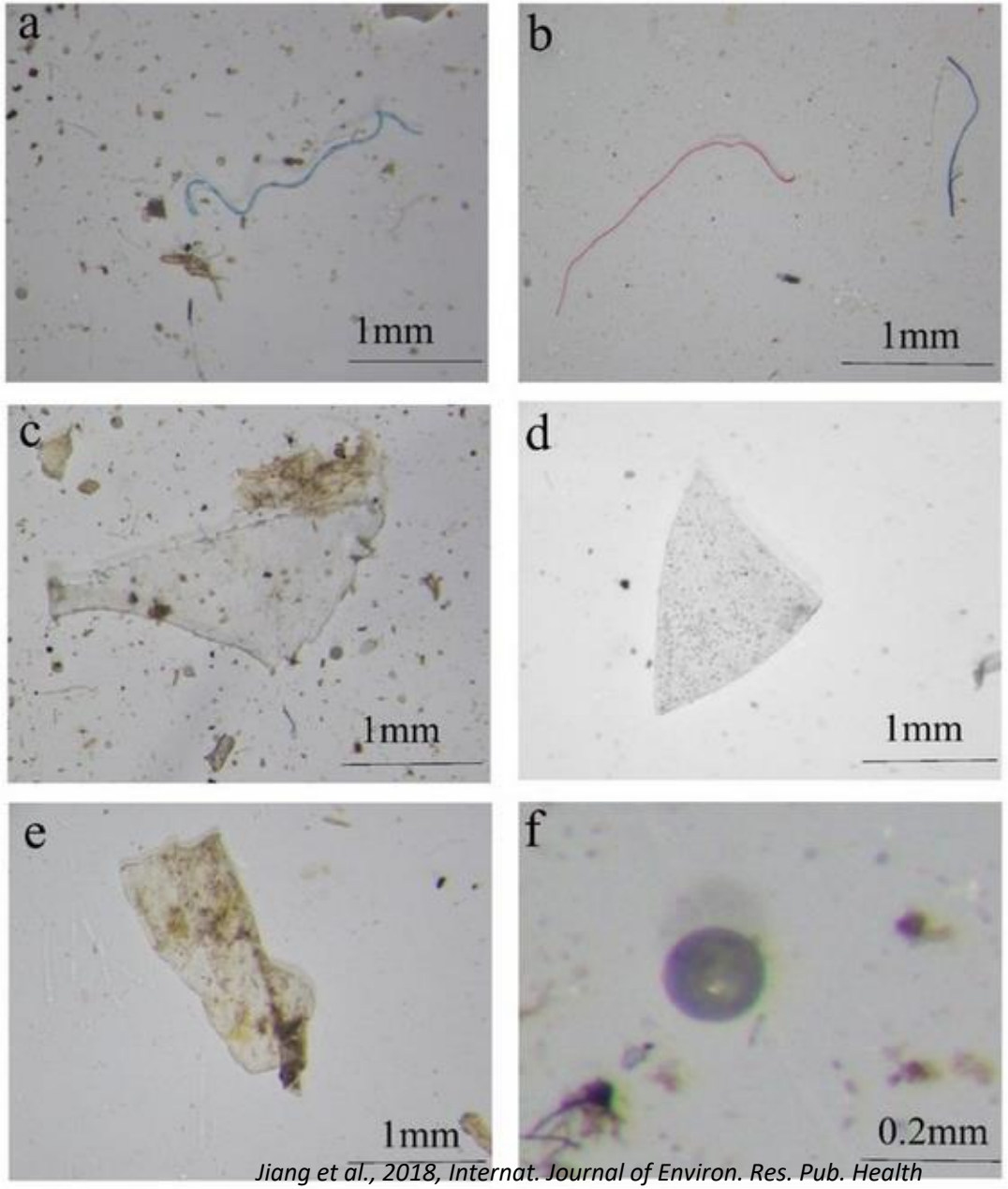
Large microplastics: 1-5 mm
Microplastics: 1-1000 µm

Any solid plastic particle, unsoluble in water.

Irregular size and forms
⇒ Size classification according to the longest length of the particle

The amount of microplastic in matrix can be expressed in numbers or mass per unit of matrix (and subcategories e.g. form, color, polymers…)

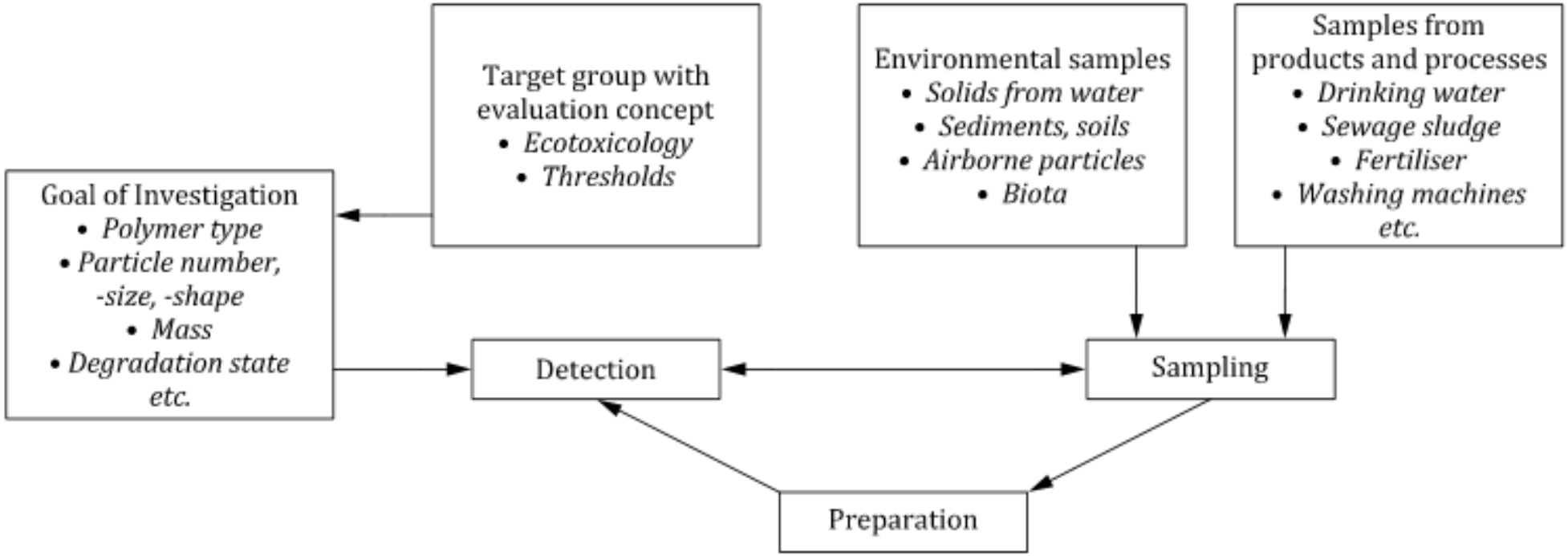
⇒ Clear question or evaluation concept needed to target the right parameters to measurement



Jiang et al., 2018, Internat. Journal of Environ. Res. Pub. Health

ISO 24187: Principles for the analysis of microplastics present in the environment (September 2023)

⇒ Clear question or evaluation concept needed to target the right parameters to measure



Representation of interdependancies in microplastic analysis in environmental samples

⇒ Choose the best adapted detection method(s)

ISO 24187: Principles for the analysis of microplastics present in the environment (September 2023)

⇒ Clear question or evaluation concept needed to target the right parameters to measure

⇒ **Multitude of detection methods**

Spectroscopy

FTIR
ATR-FTIR
FPA-FTIR
Raman
NIR, SWIR
QCL-IR

Mass spectrometry

ICP-MS (additives or ^{13}C)

MS coupled with separative techniques

GC-MS
coupled with
Py (pyrolysis)
TED (Thermal Extraction Desorption)

LC-MS/UV
for specific polymers (PET, PC, PA…)

Optical methods

Microscopy (fluorescence)
Hot needle test
Visual sorting (subjective!)

ISO 24187: Principles for the analysis of microplastics present in the environment (September 2023)

⇒ **Clear question or evaluation concept needed to target the right parameters to measure**

⇒ **Multitude of detection methods**

Optical methods — information on particle number

→ Sampling and analysis of small MP challenging
(size limit for optical ~20 µm; for spectroscopic ~5 µm)

Spectroscopy — information on polymer, particle number and size

→ Crucial for toxicological studies

Mass spectrometry — information on chemical composition and mass concentration

→ ! Large MP: high mass concentration but low number concentration

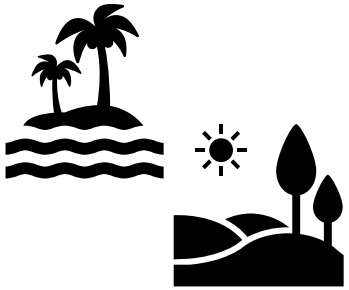
→ Estimate the occurrence (trends, monitoring)

ISO 24187: Principles for the analysis of microplastics present in the environment (September 2023)

What are the air sampling recommendations?

Indoor air*:*

refer to ISO 16000 – 34 : Indoor air: Strategies for the measurement of suspended particles
general indoor air sampling strategies for airborne particles from 1 nm to 100 µm
⇒ *nothing specific for MP*



Ambient air:

recommendation for sampling over long time periods and using stratified sampling (as described in ISO 9359: Air Quality: Stratified sampling method for assessment of ambient air quality)
no indications on how to differentiate MP from other PM
refer to 2020 BMBF German statuspaper is cited, which states that “Methods for identifying and quantifying plastics are therefore recommended”
⇒ *nothing specific for MP*

No international consensus on sampling or analytical strategies and techniques

And in France?

Spoiler alert: no unified approach for airborne MP so far

And for aqueous matrixes ?

2 French norms in the making for MPs in aqueous matrix:

XP T 90-968-1 Water quality - Analysis of microplastics in human drinking water and groundwater - Part 1: Methods using vibrational spectroscopy
⇒ In final stage of writing
⇒ French mirror of EN ISO 16094-2 ➔ to be validated end 2024

XP T 90-968-1 Water quality - Analysis of microplastics in human drinking water and groundwater - Part 2: PY/GC/MS method after microscopy
⇒ Comparative studies ongoing
⇒ Mirror of EN ISO 16094-3 – XP probably out before the ISO norm

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International context : aqueous matrixes

International: ISO norms in progress for MPs in water:

✓ ISO/NP 16094-1 "MP in water - General and sampling"
✓ ISO/NP 16094-2 "MP in water - Vibrational spectroscopy" : **call for participation until 2023**
✓ ISO/NP 16094-3 "MP in water - Thermoanalytical methods" : **technical comments ongoing**
✓ ISO/NP 16094-4 "MP in water – Automatic Sample Preparation" : **project**
✓ ISO/NP 16094-5 "MP in water – Ecotoxicological" : **conceptual stage**

French context : airborne microplastics

Monitoring of international literature

INERIS (National Institute for the Industrial Environment and Risks)

LCSQA (Central Air Quality Monitoring Laboratory)

Research projects:

ANSES (National Agency for Food, Environmental and Occupational Health Safety; 3 projects)

INERIS (few projects on aqueous matrix)

ADEME (Agency for the Ecological Transition) & OFB (French Office for Biodiversity) call for microplastic fluxes:

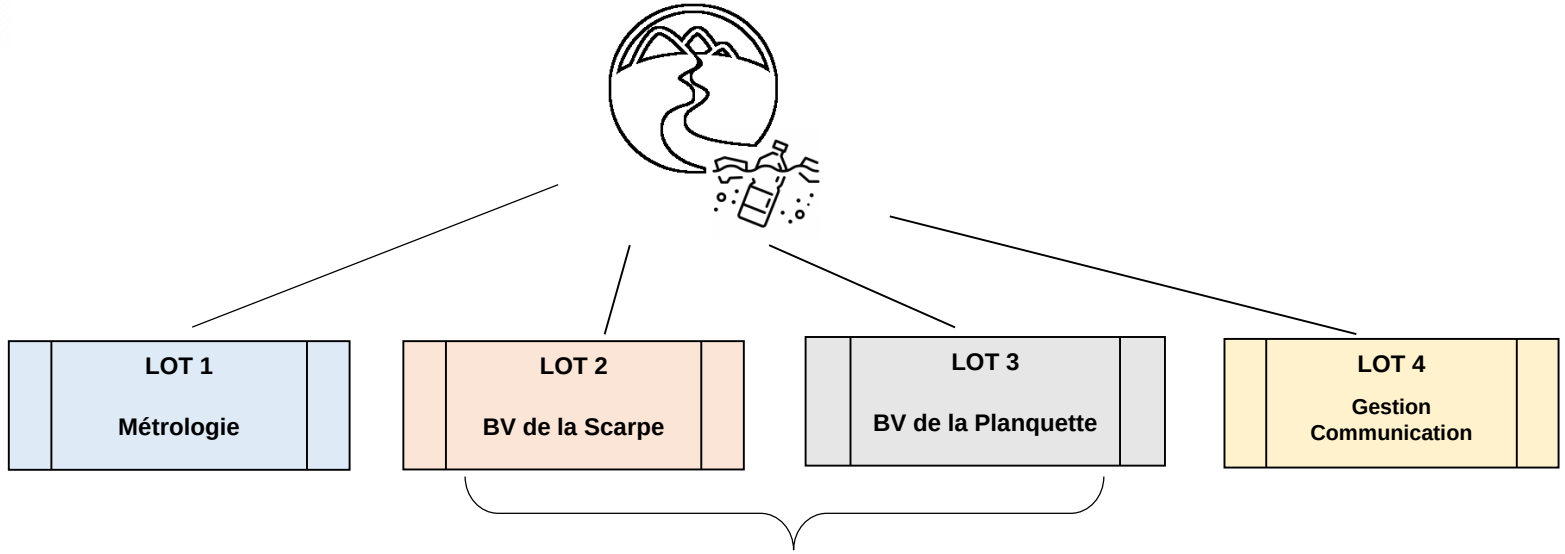
<u>Characterization and quantification of microplastics in continental environments – soil, water and transfers</u>

4 selected projects:

- Plastival
- Minuscule
- Dymitria
- Plastranfer

+ intercomparison excercise

DyMiTRiA : Dynamics of the deposition and transfer of microplastics in rivers in a region subject to heavy urban and agricultural anthropisation



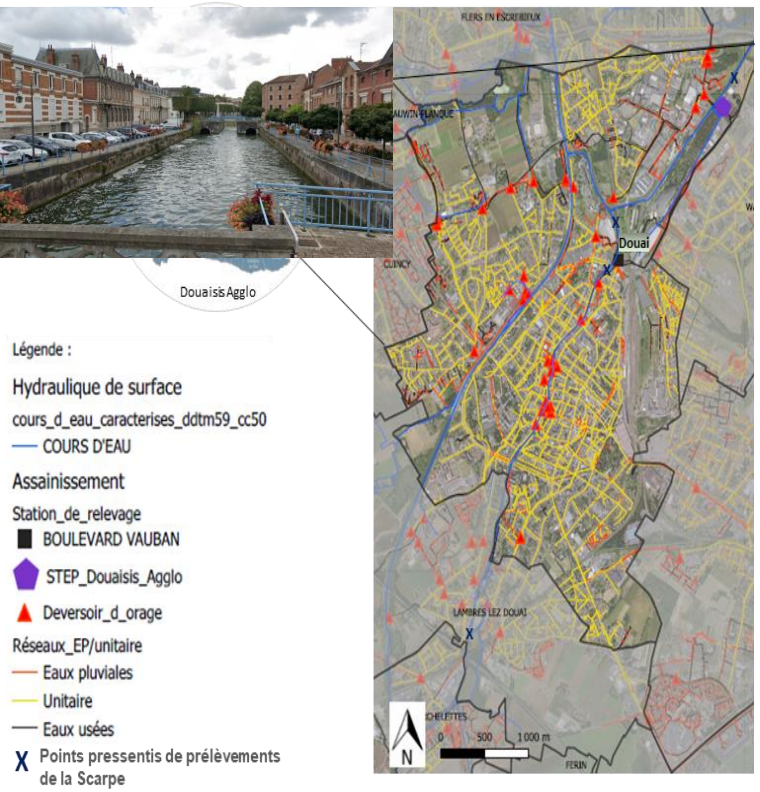
Intercomparison exercice
Methodology

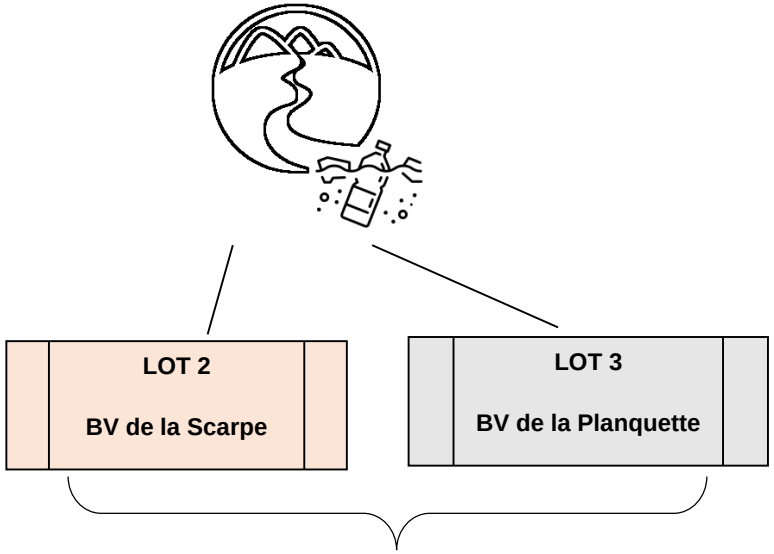
Sampling of MP in 4 matrices:

- Water (suspended)
- Sediments
- Soil
- Air (fall-out)

DyMiTRiA : Dynamics of the deposition and transfer of microplastics in rivers in a region subject to heavy urban and agricultural anthropisation

Scarpe: urban

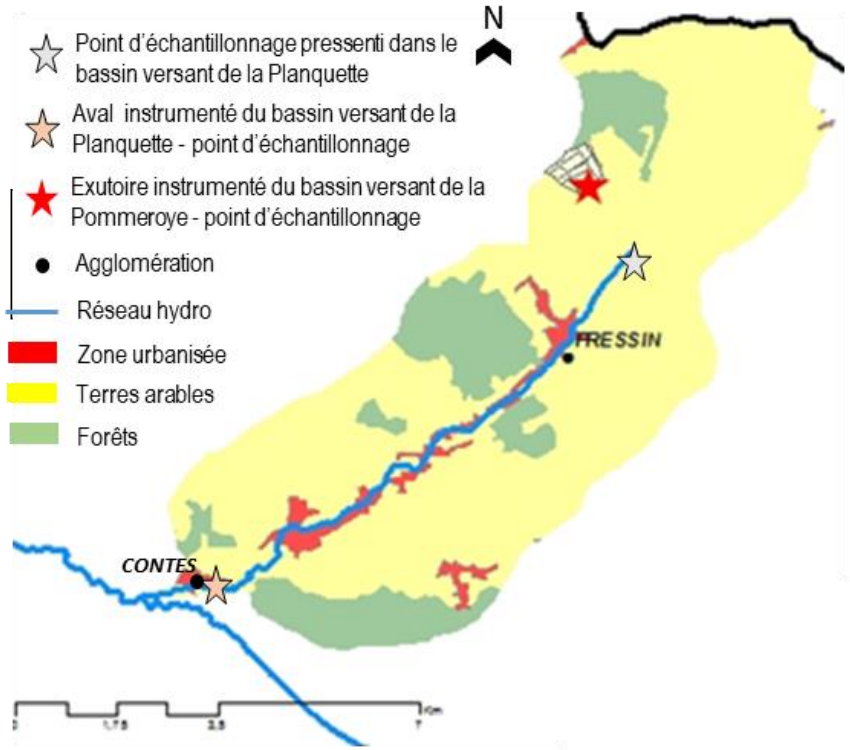




Sampling of MP in 4 matrices:

- Water (suspended)
- Sediments
- Soil
- Air (fall-out)

Planquette: rural



Sampling of atmospheric microplastics

Other matrixes: Constant et al., Environ. Sci. & Technol., 2021, https://pubs.acs.org/doi/10.1021/acs.est.0c08386
Constant et al., J. Haz. Mat., 2021, https://doi.org/10.1016/j.jhazmat.2021.126571

Diameter:
26 cm

1 wet deposition sampler

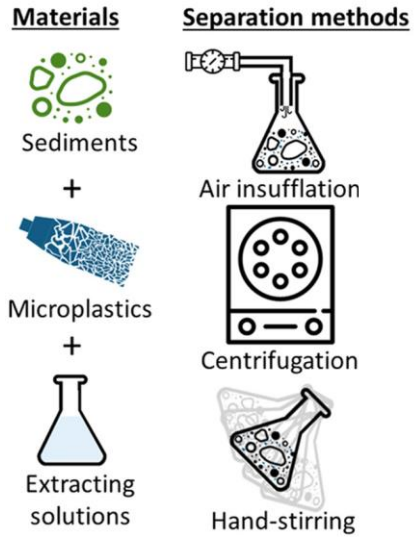
Replaced by a glass bottle (Duran, 5L)

Sampling method for atmospheric microplastics

- Sampling time: between 2 weeks and 1 month in function of the environment (rural/urban)
- Cleaning of sampling material: rinsing 5x with ultrapure water (better: 540°C); packed in tin foil for transport – precautions for contamination
- Sample preparation: (similar for all matrixes)
  - Rinsing with ultrapure water to collect and
  - Digestion with H_2O_2 (3%)
  - Extraction by density in aqueous NaI solution (1.6 g/mL)
  - Centrifugation (5 min, 2000 rpm)
  - Supernatant collected and sieved for size fractionation
  - Filtration on filter paper (Whatman©; 47 mm diameter; 2µm 37 porosity)

Repeated 4 times

- Analysis: stereo-microscope, SEM, ATR-FTIR, µFTIR
- Blanks: lab blank = open petri dish with filter in the lab
  field blank = opening and closing recipient, treatment as sample



Constant et al., *J. Haz. Mat.*, 2021

Sampling method for microplastics

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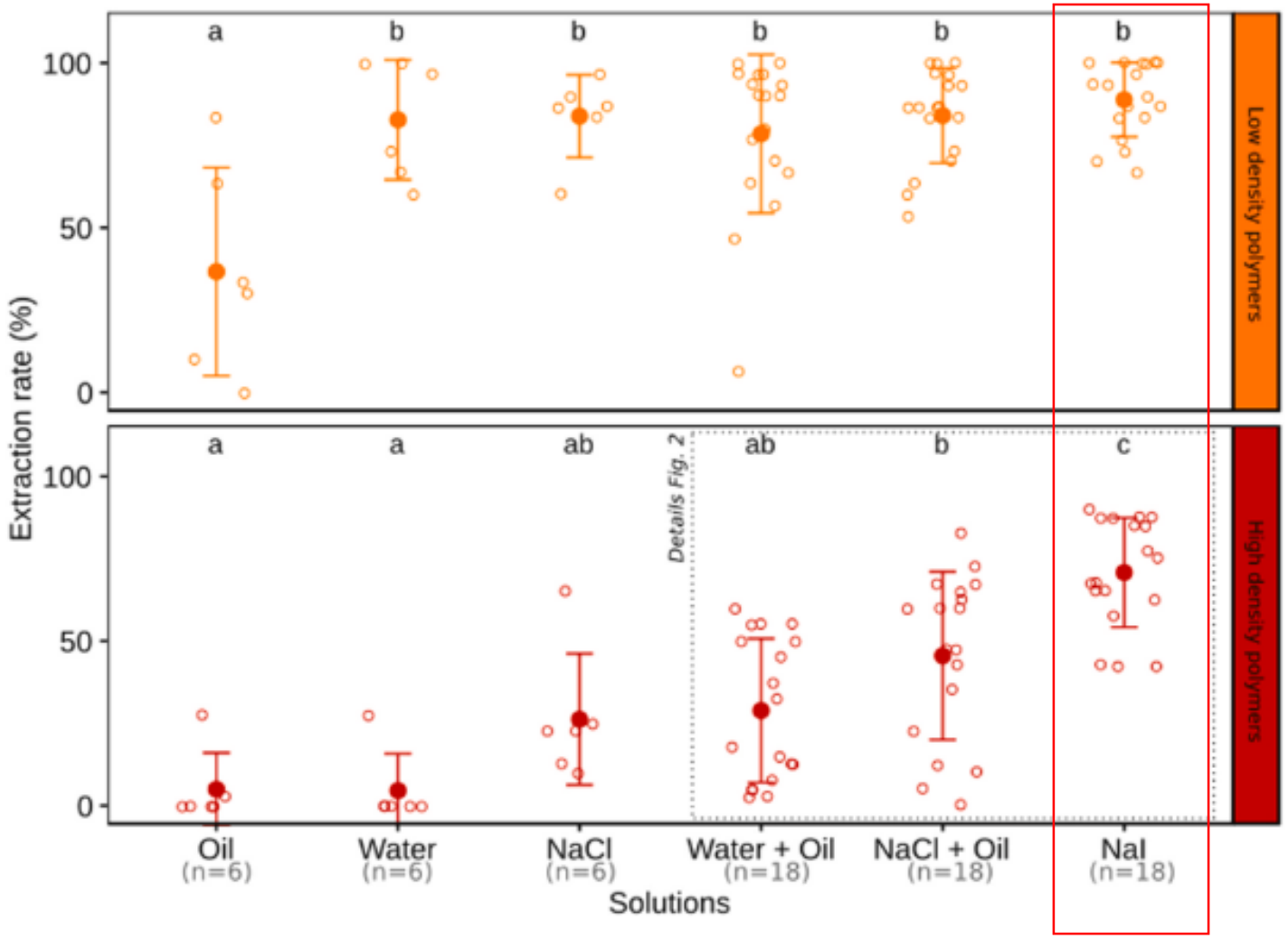

Sampling method for microplastics

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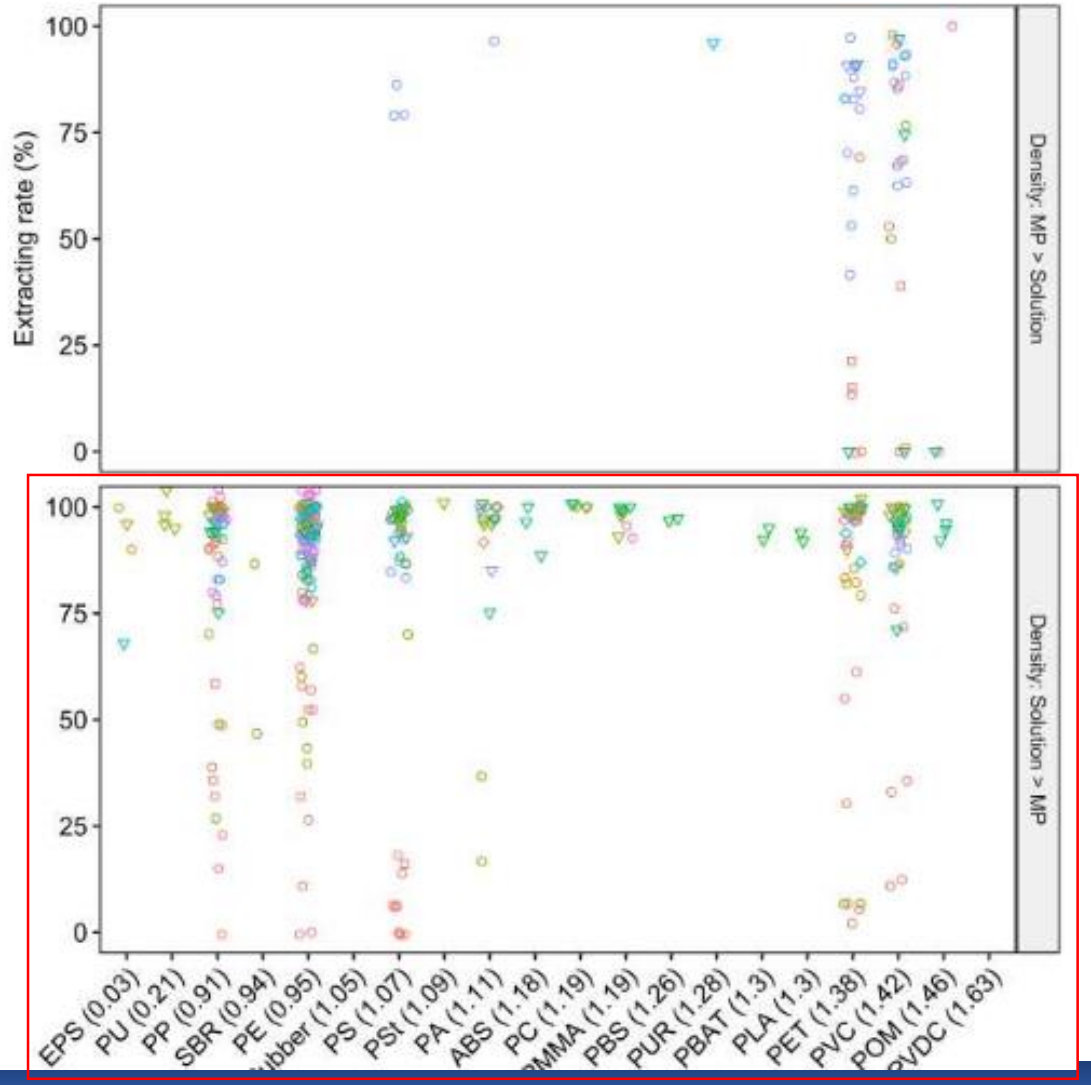

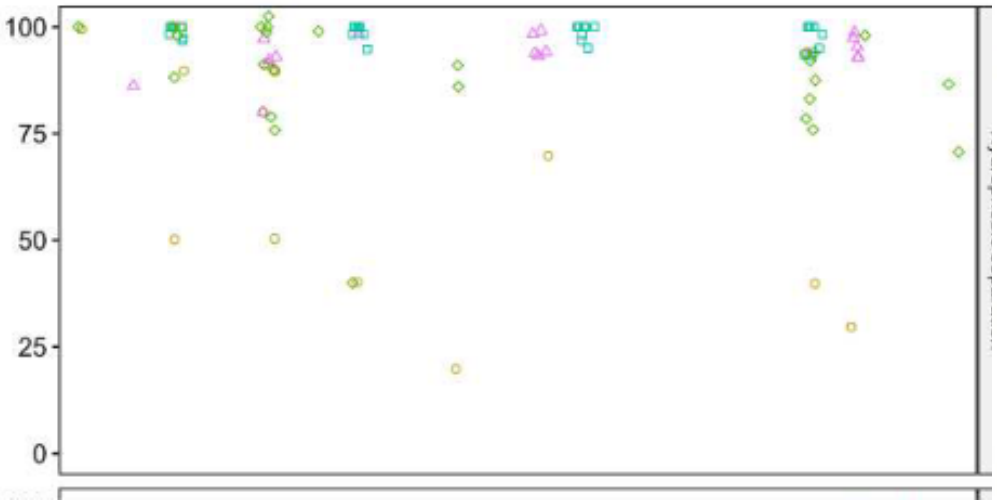

Best extraction rates for solutions density > MP

Intercomparison excercice

For all matrixes : sizes: relevant for each matrix?

⇒ different size fractions for each matrix

Atmospheric MP:

- analysis of 'common sample' - spiked

  BUT: heterogeneous samples and different size fractions analysed

  ⇒ complicated comparisons!

- sampling and analysis on 'reference site'

  BUT: different sampling devices (1 active, 4 passive) ⇒ difference in sampling period and duration

  different analytical techniques: PyGC-MS and FTIR ⇒ comparison of *chemical information* only

Attention to be paid to the intercomparability of different matrixes : air, water and soil/sediment

⇒ Py-GC-MS more complicated on heavy organic matrix

Thank you for your attention!

liselotte.tinel@imt-nord-europe.fr