From Macro- to Nanoplastics and Beyond:

Advances in Analytical Techniques

Milica Velimirović Fanfani

We are VITO - An independent research institute focused on accelerating the transition to a sustainable world

In 2023

1296	61	268M€	25	277	11/3
employees	nationalities	revenues	patents/year	publications	11 sites on 3 continents


ANALYTICAL
LABORATORY

TEAM GOAL









R&D 9



POST-DOC 1


PHD 2


ANALYST 17













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#30

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Nanoplastics analysis - Platform integration based on nanoscience methods

Field flow fractionation

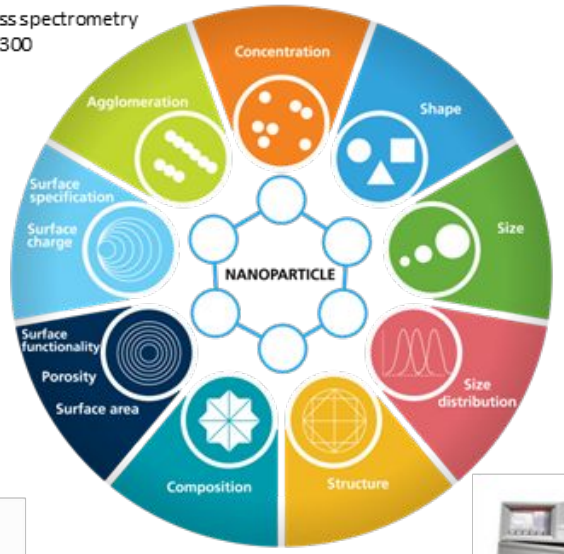
Single particle inductively coupled plasma mass spectrometry (sp ICP-MS) Perkin Elmer Nexion 300

Dynamic Light Scattering (DLS) ZetaSizer

Ambient ionization-high resolution mass spectrometer DART – Thermo Q Exactive

Pyrolysis-gas chromatography-mass spectrometry (Py-GC-MS)

Energy dispersive X-ray fluorescence ED-XRF, XEPOS, Spectro



Field Emission Gun Scanning Electron Microscopy with STEM detector (FEI, Nova NanoSEM 450)

Bruker Multimode 8 AFM

Gel Permeation Chromatography –UV/VIS-MALS

FTIR Spectrometer

Agilent 7100 CE System

Inductively coupled plasma atomic emission spectroscopy Agilent 5100 ICP-OES

Micro-XRF Analyzer EDAX Orbis PC

Nanoparticle Tracking Analysis (NTA) NanoSight NS500

CPS Disc Centrifuge DC2400

Microwave digestion, Multiwave PRO Anton Paar

μRaman

spICPMS (Nu Attom)

Detailed characterization of plastic debris: A case study of the Sea Scheldt estuary

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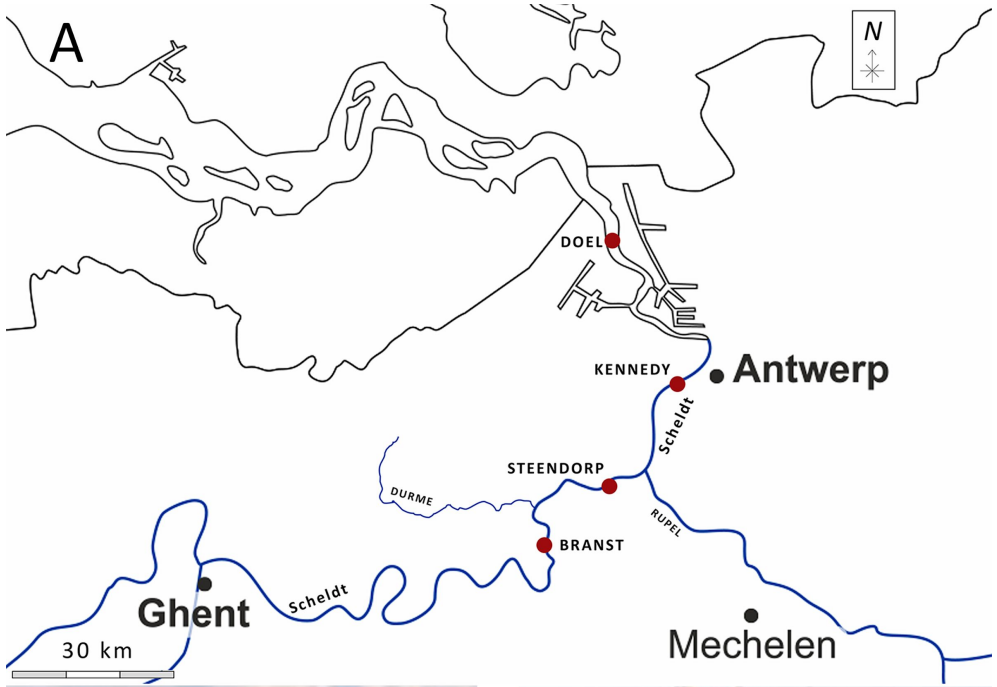
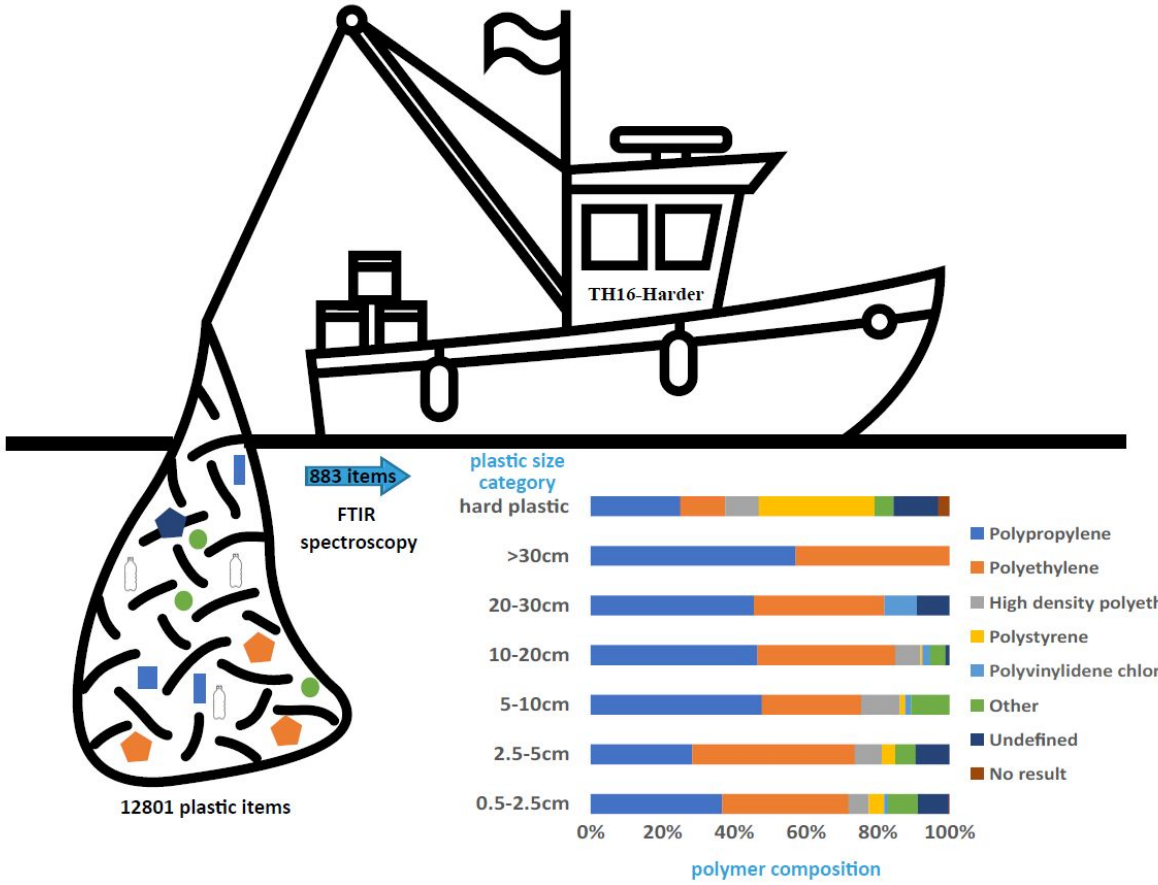
The Sea Scheldt estuary: plastic debris monitoring in 2018 (spring, summer and autumn)



anchor netting



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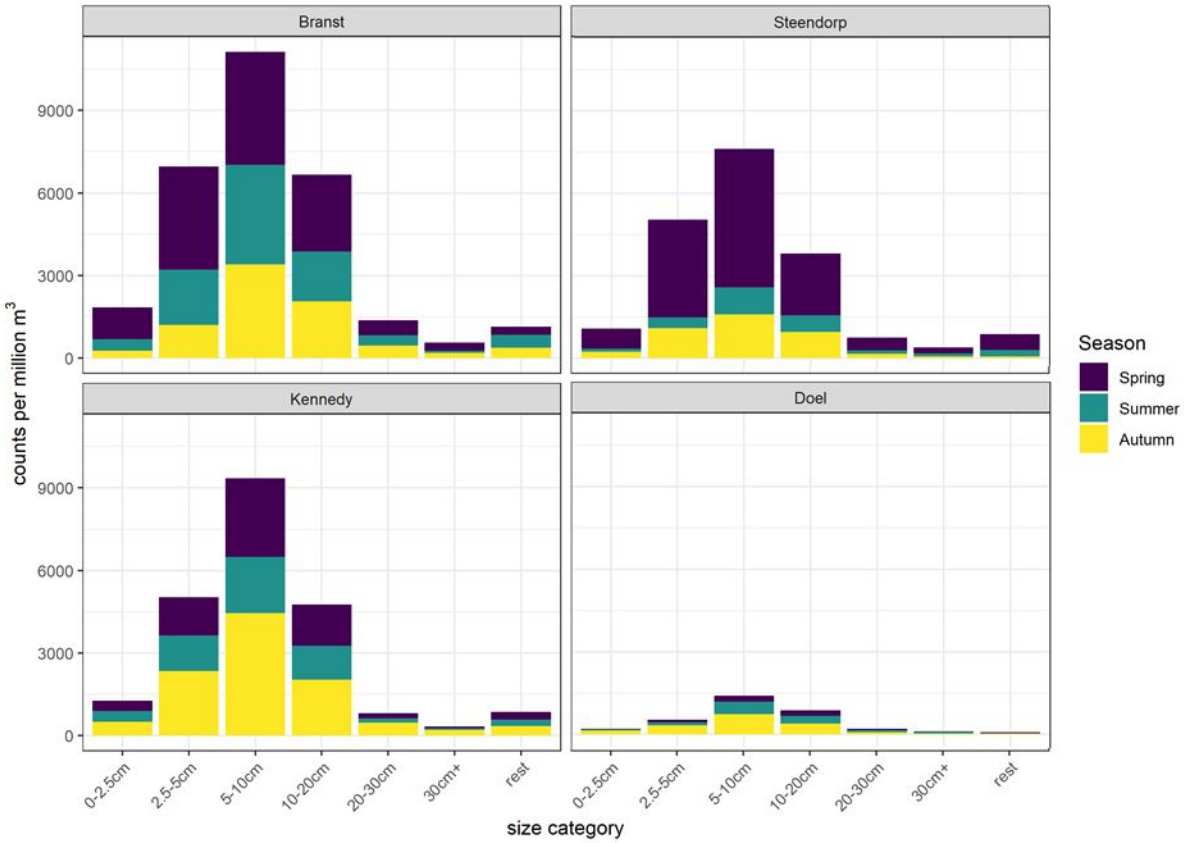
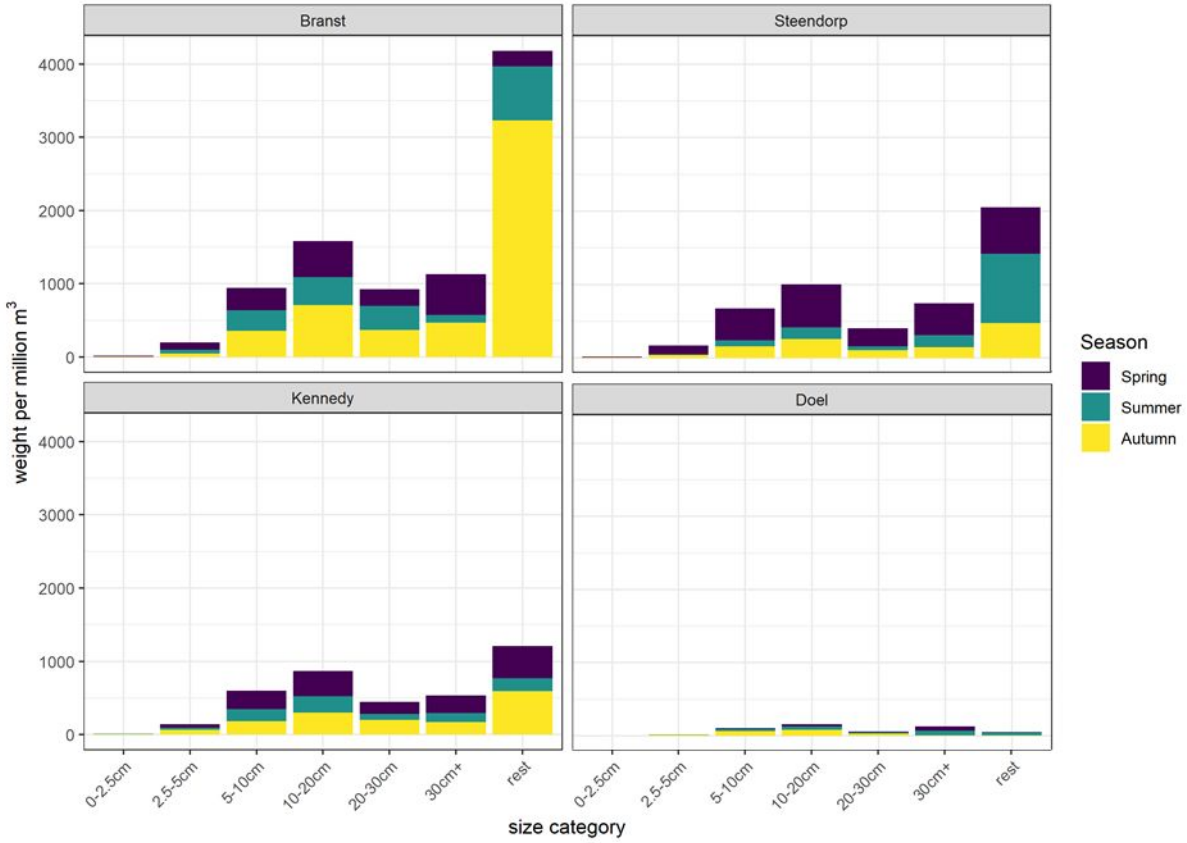
University of Antwerp

The Sea Scheldt estuary: plastic debris monitoring in 2018 (spring, summer and autumn)





Distributions of the number of items and weight in grams of items collected per million m^3 of water, by size, at the four locations

Science of The Total Environment
Volume 851, Part 1, 10 December 2022, 158226

What can we learn from studying plastic debris in the Sea Scheldt estuary?

Milica Velimirovic [a b 1], Bert Teunkens [c 1], Hossein Ghorbanfekr [b], Bart Buelens [b], Tom Hermans [d], Stefan Van Damme [c], Kristof Tirez [b], Frank Vanhaecke [a]

From Macro to Mesoplastics characterization

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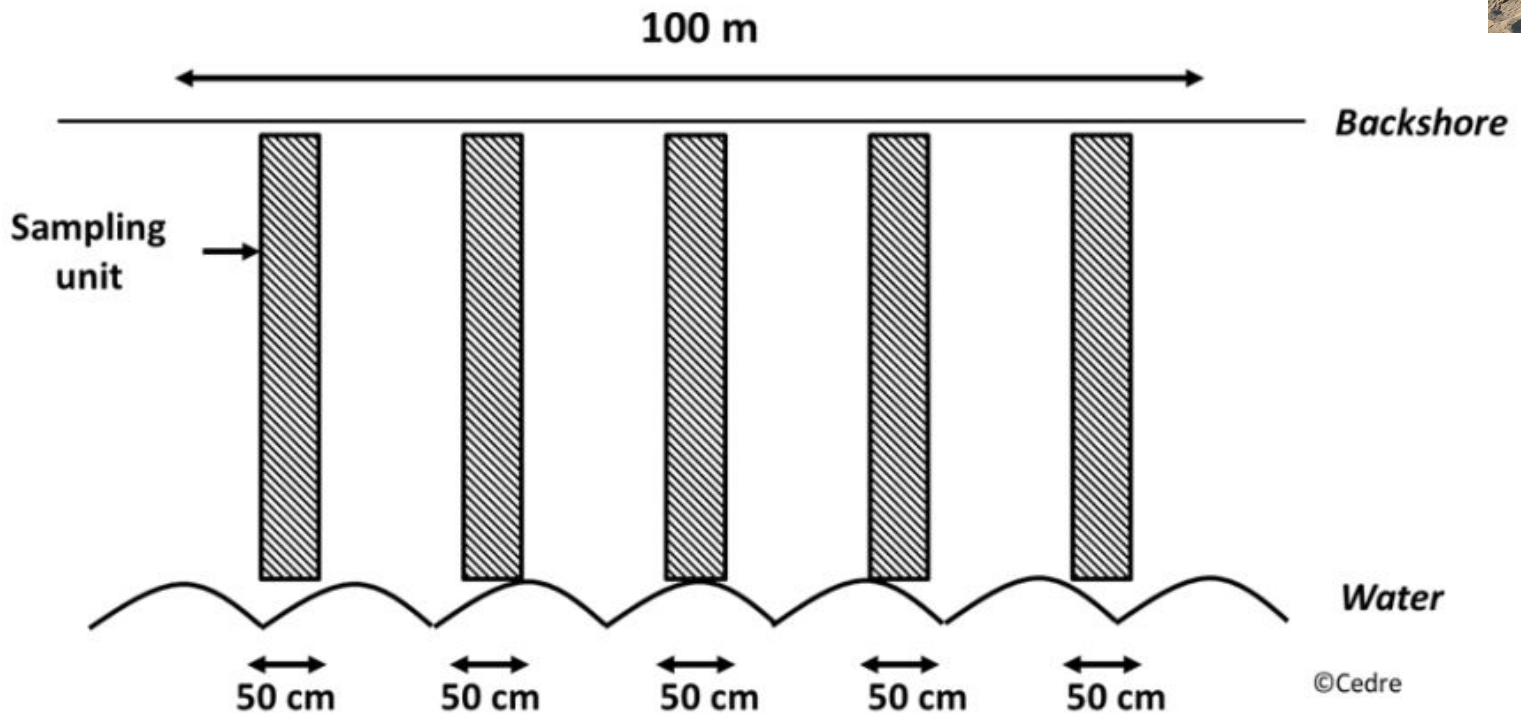
Remedies: Sampling campaign in Albania March 17th 2024 - methodology

Hamallaj Beach, Albania

Mesoplastics sampling unit



Source: Marine Strategy Framework Directive - MSFD TG ML. Guidance on the Monitoring of Marine Litter in European Seas. 2023

Remedies: Beach mesoplastics separation and characterization

Manual sorting after drying for 24 hours at 40°C

Separating mesoplastics

Flotation & stirring

Morphology

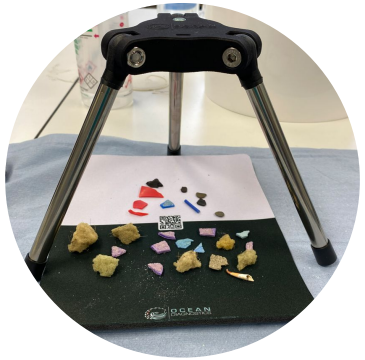
Color

Size

Polymer composition

Elemental fingerprint

© MSFD TG ML. 2023



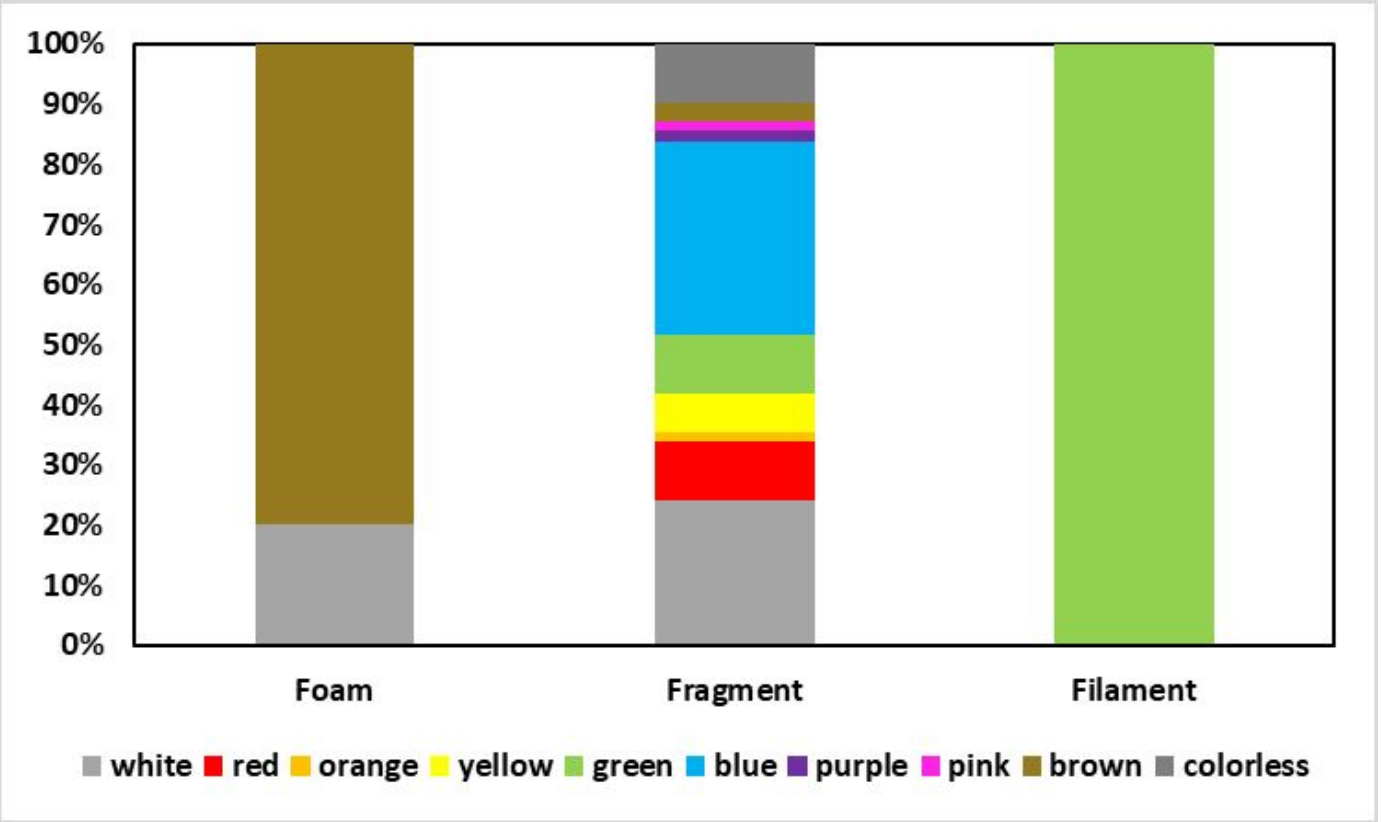
Saturna imagining system

ATR-FTIR

µXRF

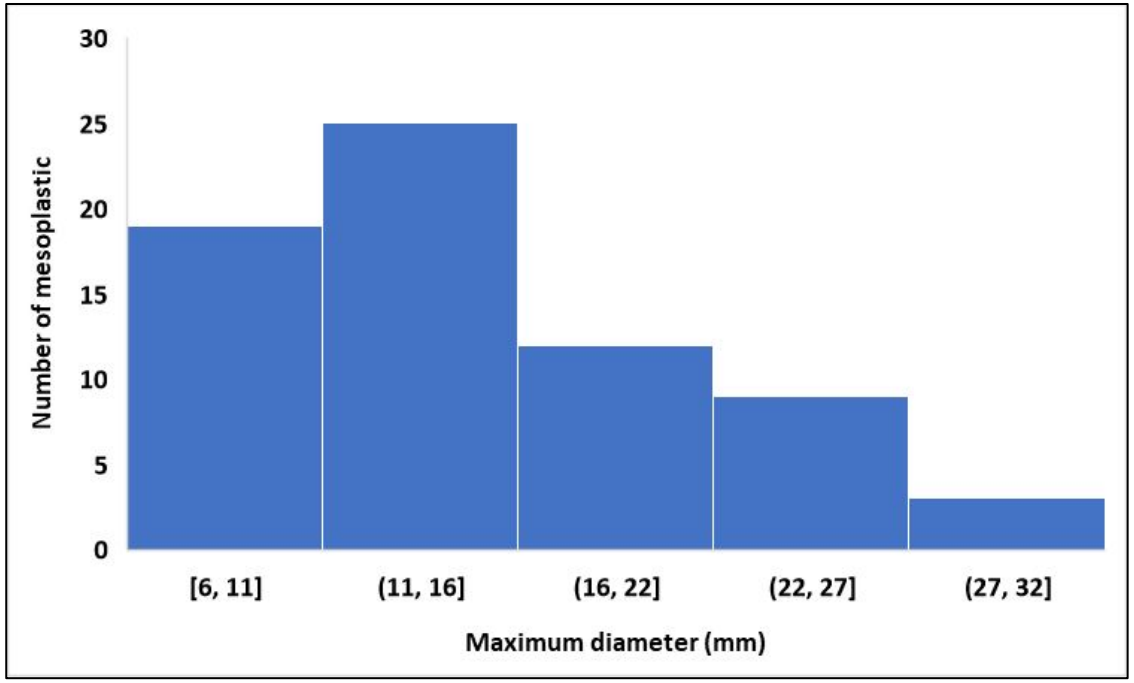
REMEDIES

Remedies: Mesoplastics – morphology and color

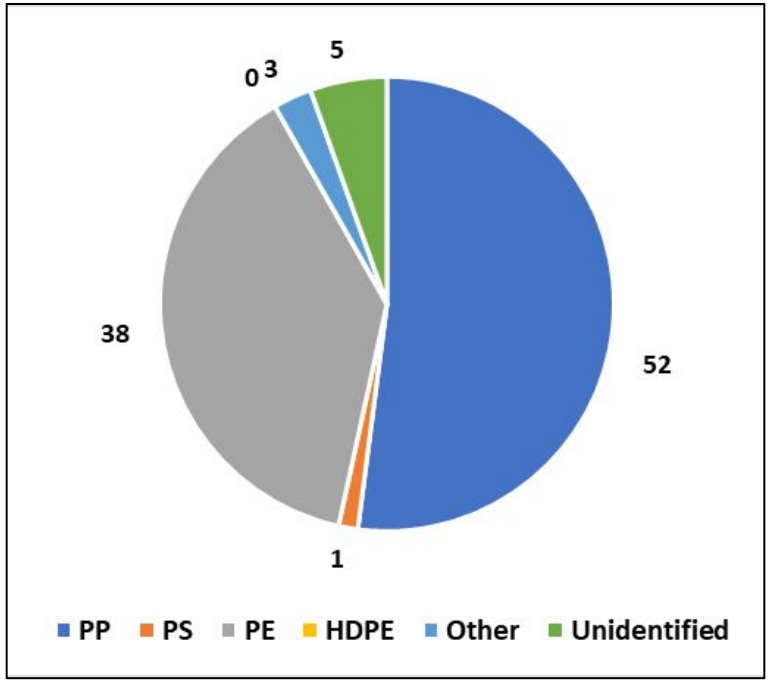


Percentage of different colors of mesoplastics present in different plastics morphologies – foam, fragment and filament (n=73)

Remedies: Mesoplastics – size and polymer composition

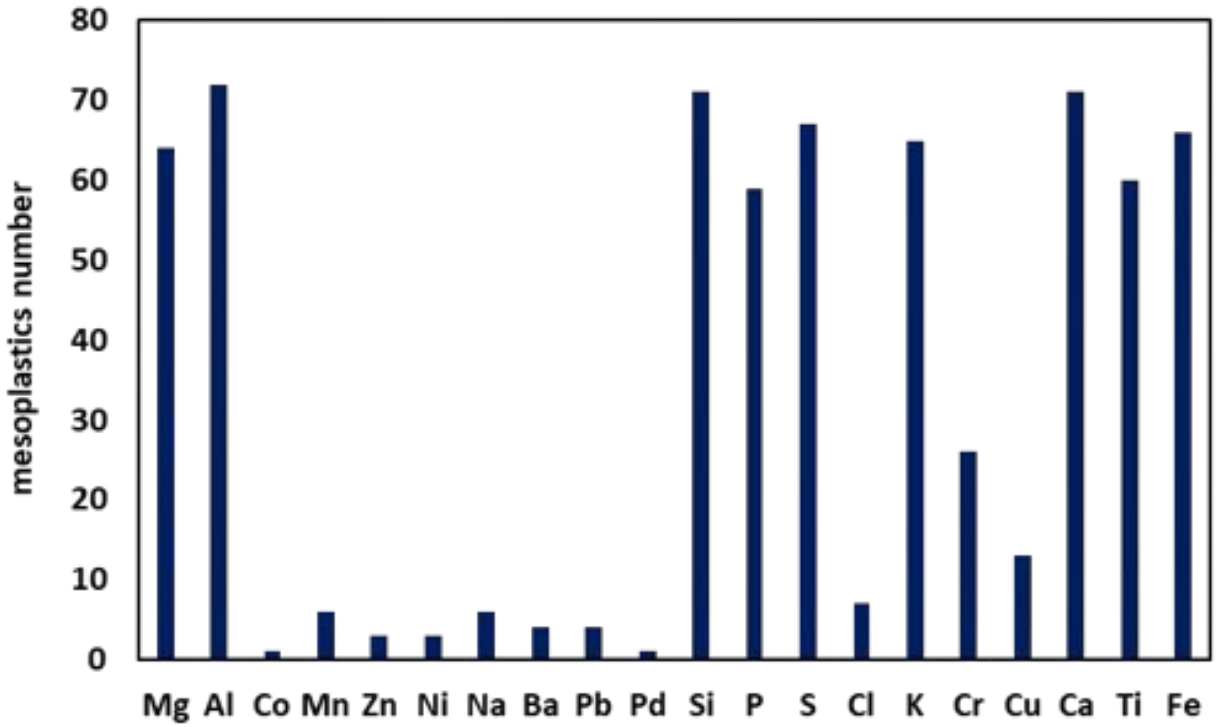


Maximum diameter (mm) of mesoplastics (n=73)



Polymer composition (%) of mesoplastics (n=73)

Remedies: Mesoplastics – elemental fingerprint



Detected elements in mesoplastics (n=73)

spICP-MS for Microplastics Characterization

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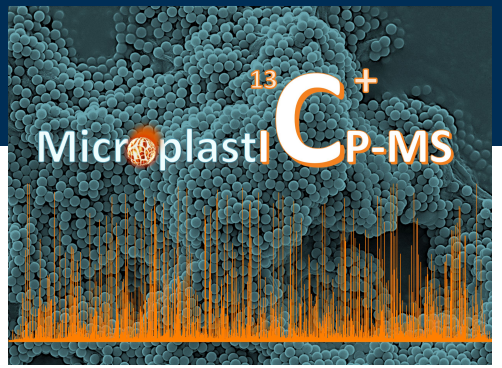
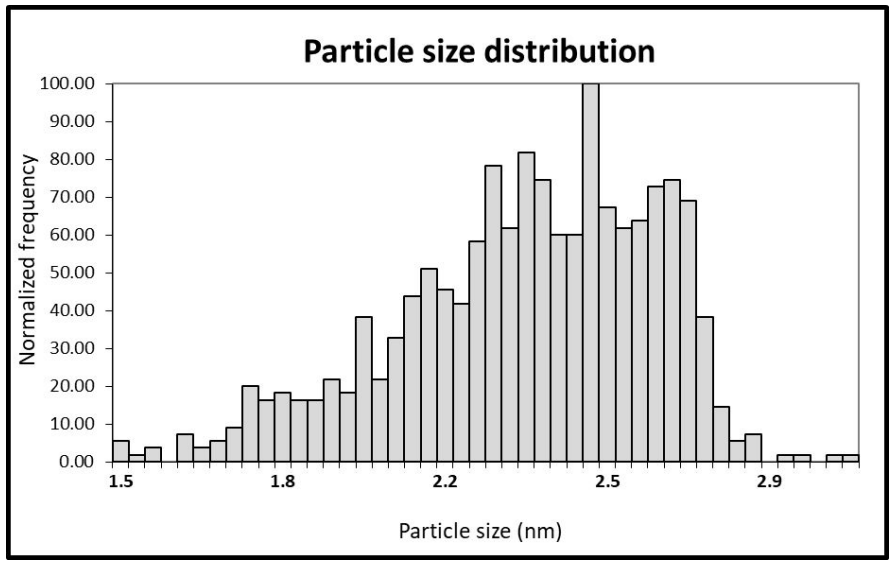
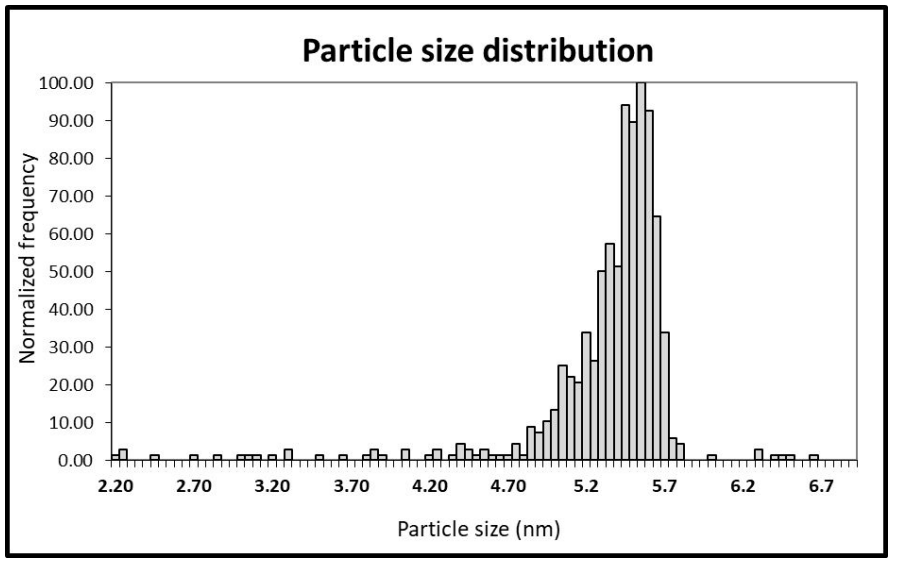
Microplastics and spICP-MS: a perfect match?



spICP-MS for low µm range MPs (1-10 µm) size determination





Particle size distribution obtained for 2.5 µm and 5 µm polystyrene (PS) microspheres measured using ICP-MS operated in single-event mode via the monitoring of $13C^+$.

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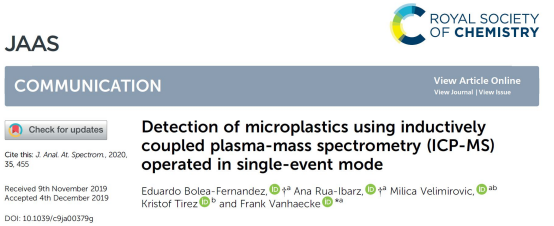
GHENT UNIVERSITY

A&MS

fwo

12ZD120N


ROYAL SOCIETY OF CHEMISTRY

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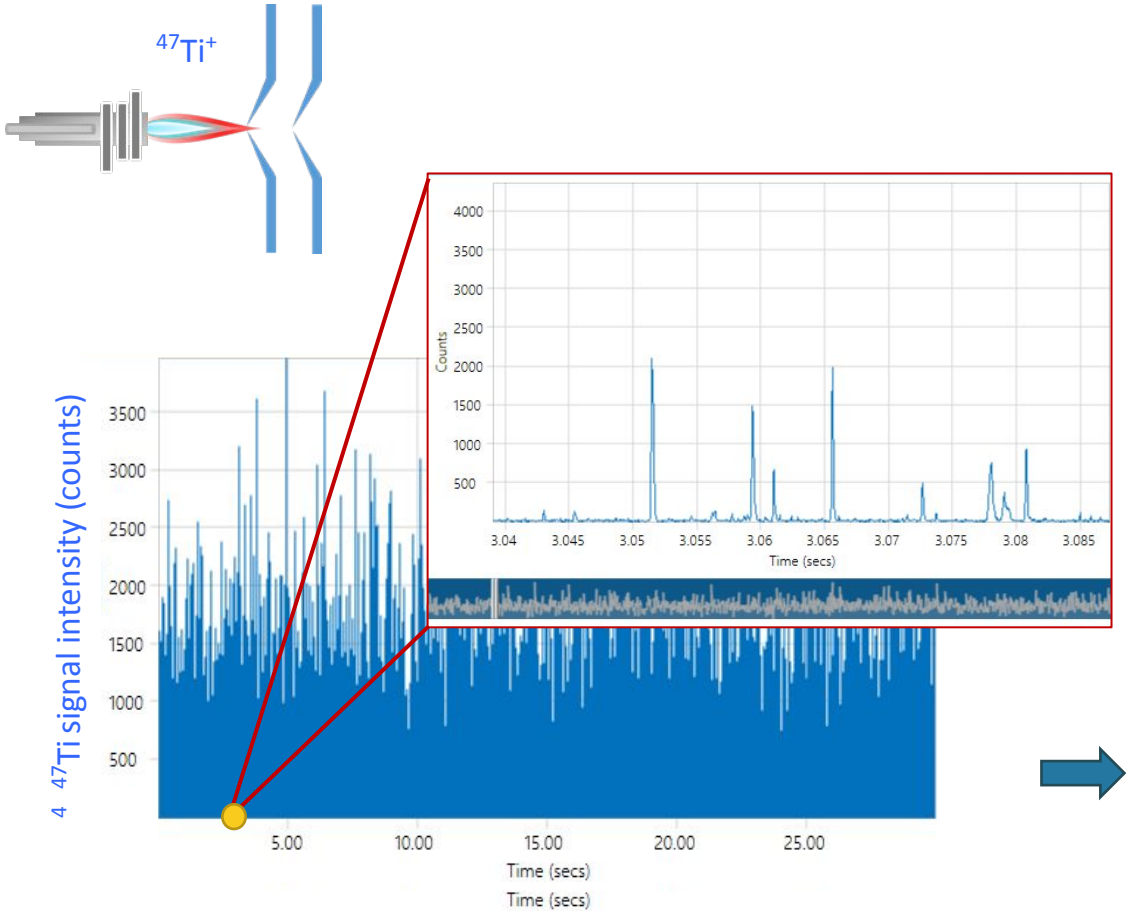
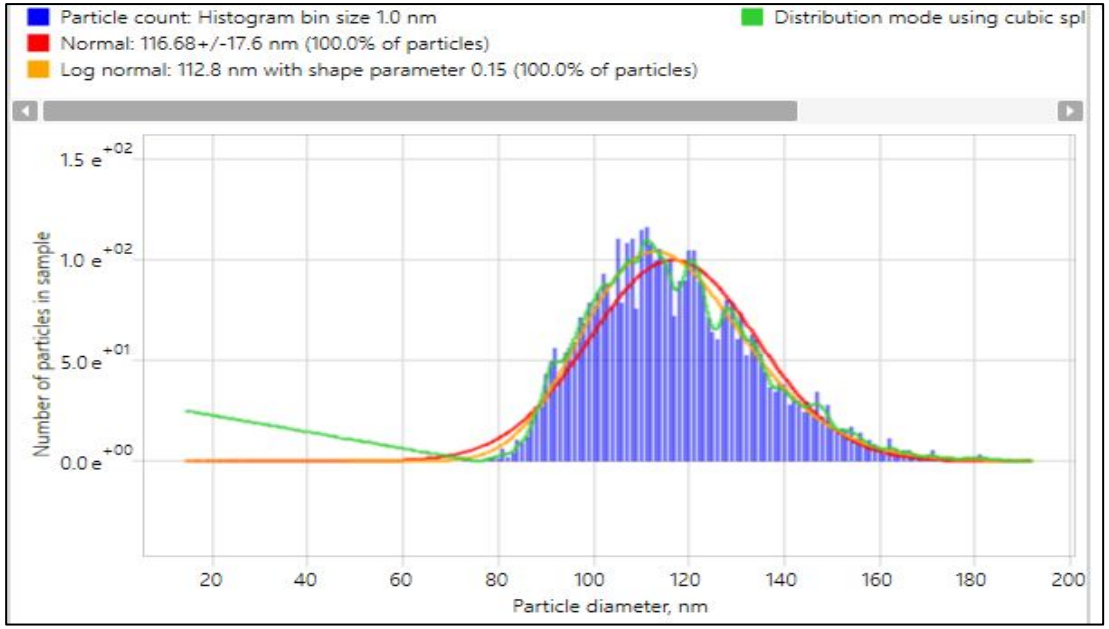
Detection of microplastics using inductively coupled plasma-mass spectrometry (ICP-MS) operated in single-event mode

Eduardo Bolea-Fernandez, Ana Rua-Ibarz, Milica Velimirovic, Kristof Tirez and Frank Vanhaecke

Metal fingerprinting as an approach to detect nanoplastics (<1 µm)





Ti particle size distribution

12ZD120N

From Micro to Nanoplastics characterization

Simulated UV weathering of disposable plastic face masks

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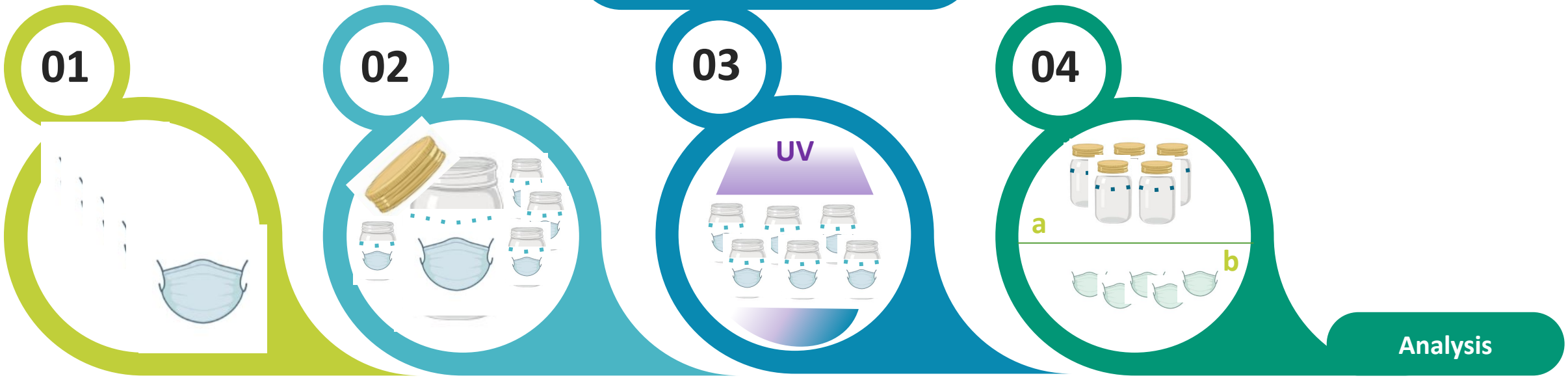
Cost action PRIORITY: Simulated UV weathering of disposable plastic face masks

Simulation of **5, 10, 20, 30**, **50** and **365 days** of sunlight exposure in Thessaloniki.



DPFMs

- Disposable surgical mask, Type II
- 3 layered
- Confirmed filtration BFE≥98% according to EN14683 Standard

DPFM + H_2O

One mask was inserted into a glass jar, filled with 300 mL of ultrapure H_2O.

System and procedural blanks were obtained to control any contamination

UV chamber

- Jars were inserted into simulating chambers for certain time intervals.
- Every 4h of aging, the media was stirred gently with a glass rod.
- Twice a day, UP H_2O was added to keep the volume of 300mL stable (evaporation).

Completion of weathering

After the completion of the required weathering period, the masks were carefully removed from the aqueous media and left in clean filter paper to dry in RT (fume hood).

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cost
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Cost action PRIORITY: Simulated UV weathering of disposable plastic face masks

a

DPFMs' leachates

b

Weathered masks

Low concentration of the samples

DLS

Particle size characterization

Microscopy

Capture formed fibers & particles

Atomic force microscopy - Bruker

AFM

Topography during aging

Asymmetric filed flow fractionation – Wyatt

AF4

Particle size distribution of NPs

Monitor oxidation processes

ATR-FTIR

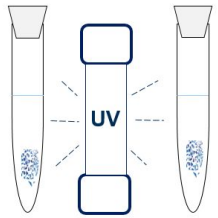
Attenuated Total Reflectance Fourie transform infrared spetrocsopy Nicolet™ iS™ 10

Identify newly-for med products

Py-GC/MS

EGA/PY-3030D, Frontier lab; GC 7890B and 5977 MSD, Agilent

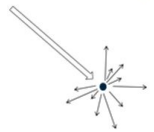
60 mg of the DPFMs
10ml of Milli-Q water



UV-C = 280 to 200 nm

120 hours
temp = 60°

Dynamic light scattering



Polydispersity index

Intensity weighted mean hydrodynamic size
z-average (d.nm)

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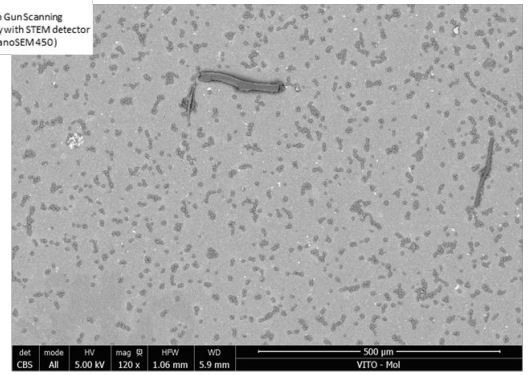
Cost action PRIORITY: Simulated UV weathering of disposable plastic face masks

DPFMs' leachates

SEM

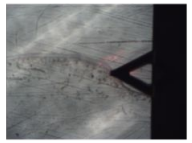
Field Emission Gun Scanning Electron Microscopy with STEM detector (FEI, Nova NanoSEM 450)



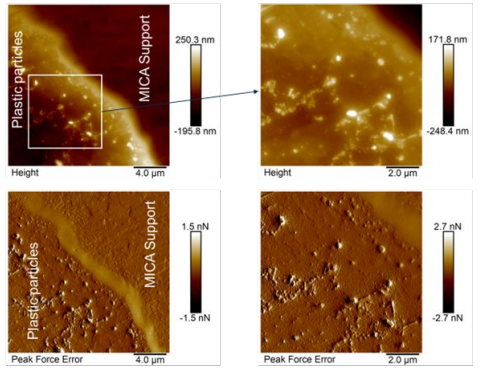
After 50 days

AFM

Bruker Multimode 8 AFM

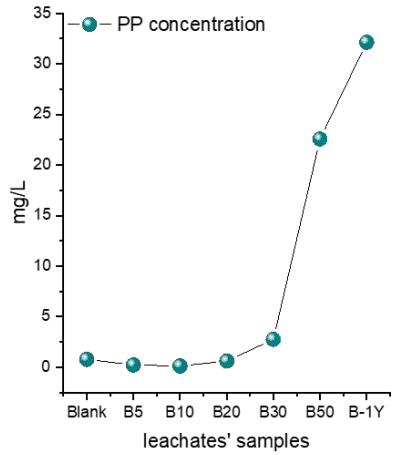


Optical image



After 50 days

Pyr-GC-MS



Nanoplastics release from face masks under simulated UV weathering

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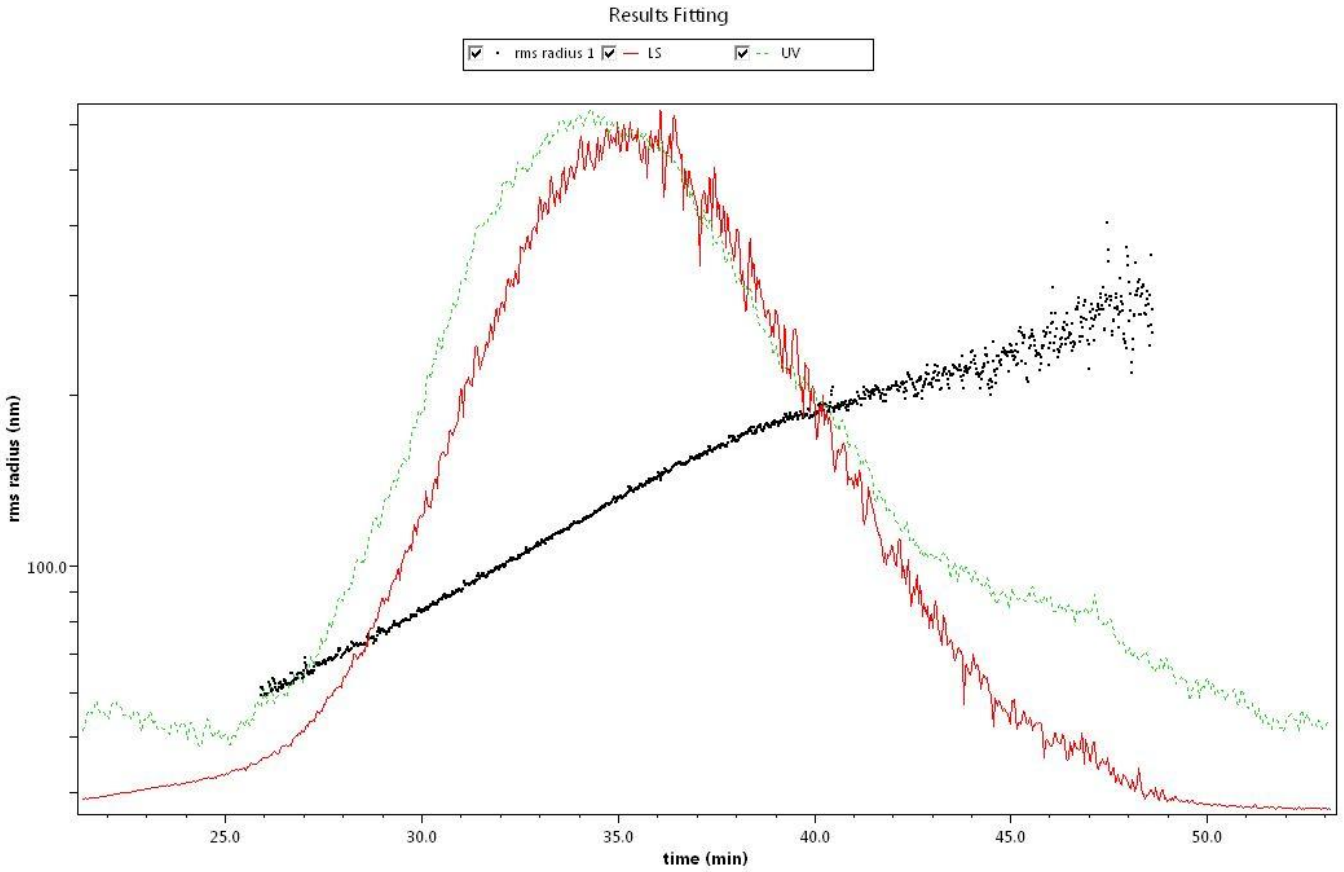
Cost action PRIORITY: Accelerated UV weathering of disposable plastic face masks

DPFMs' leachates



Simple and straightforward separation of the nanoplastics after accelerated UV degradation of surgical face masks.

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From Micro to Nanoplastics characterization

Washing water of PET and recycled PET textile

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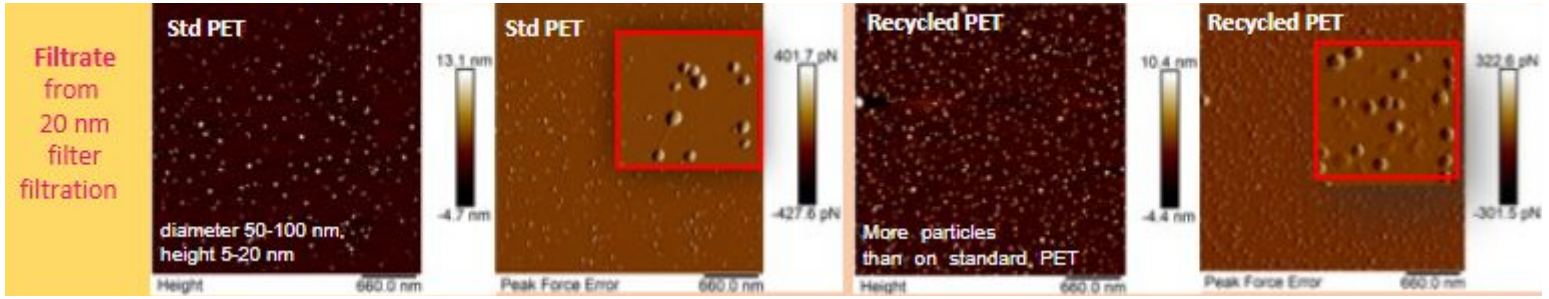
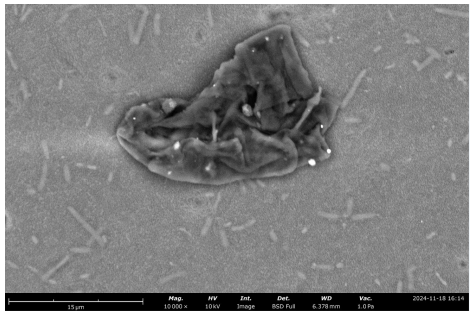
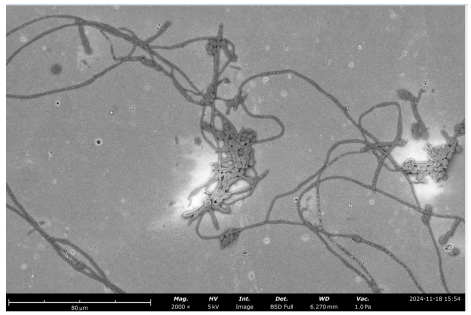
Cost action PRIORITY: Accelerated UV weathering of disposable plastic face masks

AFM:

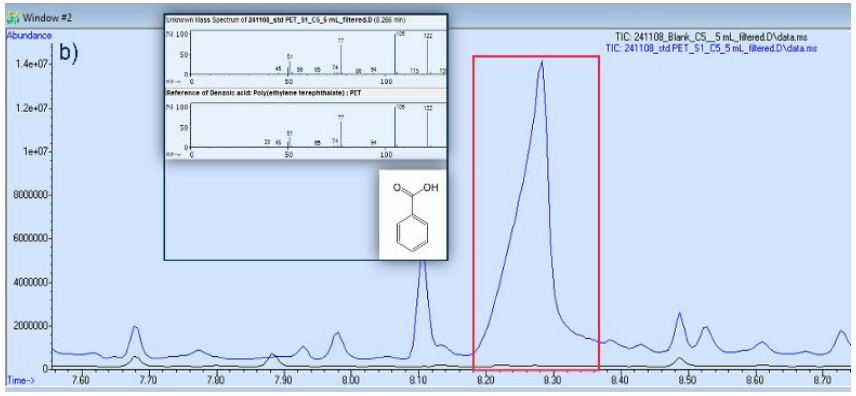


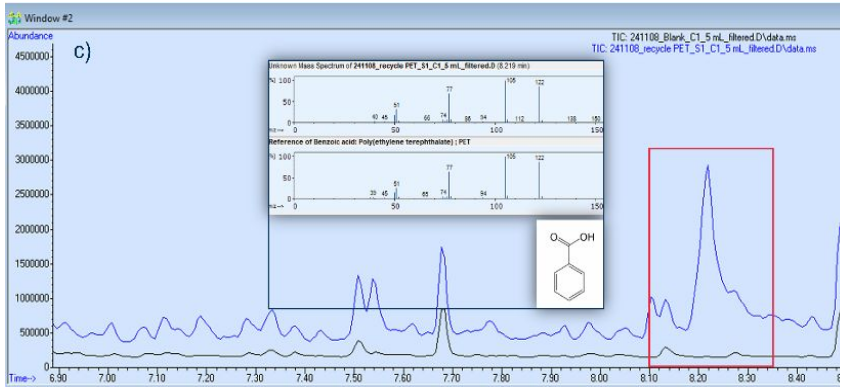
SEM:





Py-GC/MS:





SSbD4CheM

From Micro to Nanoplastics characterization

92 nm polystyrene spiked fish samples

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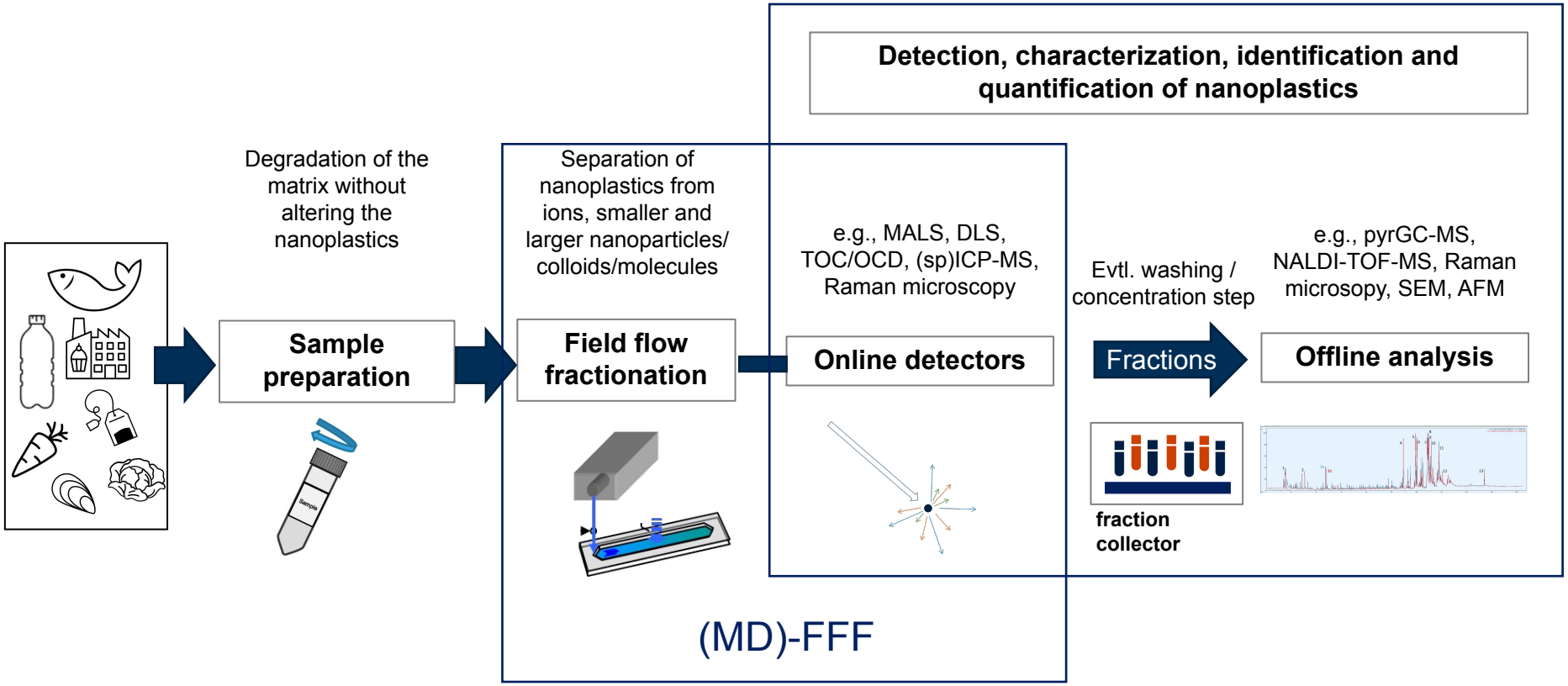
MS4Plastics: Multi detector field flow fractionation for nanoplastics characterization

Wyatt FFF system



Analytical and Bioanalytical Chemistry
https://doi.org/10.1007/s00216-022-04321-y

TRENDS

Finding the tiny plastic needle in the haystack: how field flow fractionation can help to analyze nanoplastics in food

Katrin Loeschner[1] · Janja Vidmar[2] · Nanna B. Hartmann[3] · André Marcel Bienfait[4] · Milica Velimirovic[5,6]

Received: 7 July 2022 / Revised: 31 August 2022 / Accepted: 3 September 2022
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Marie Skłodowska-Curie Actions
GA: 101023205

MS4Plastics: Multi detector field flow fractionation for nanoplastics characterization

92 nm PS spiked fish sample

Digestion:

Proteinase K digestion

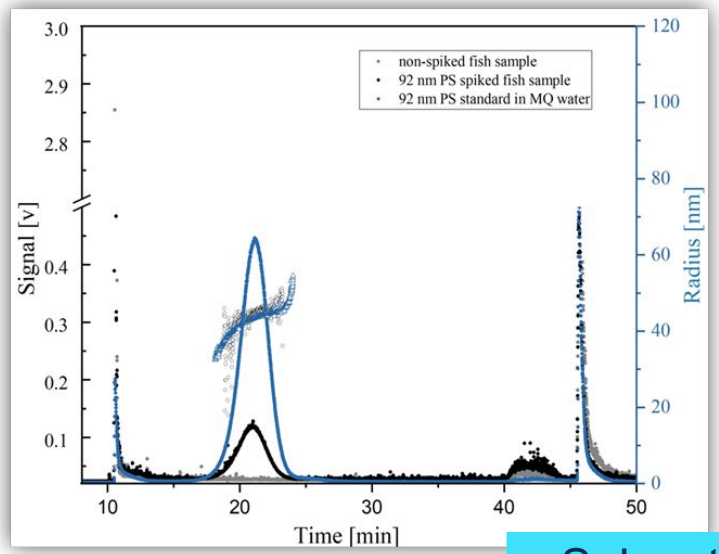
37 degree
17 h
Water bath

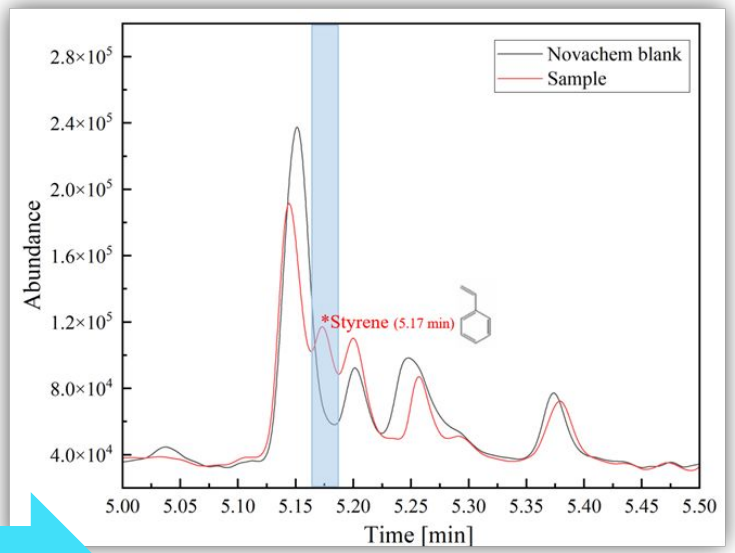
Wyatt FFF system

AF4-MALS:

Py-GC/MS:

Polystyrene size determination





Polystyrene identification and quantification

Solvent extraction

Integrated approach

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GA: 101023205

From Micro to Nanoplastics characterization

Sewage sludge

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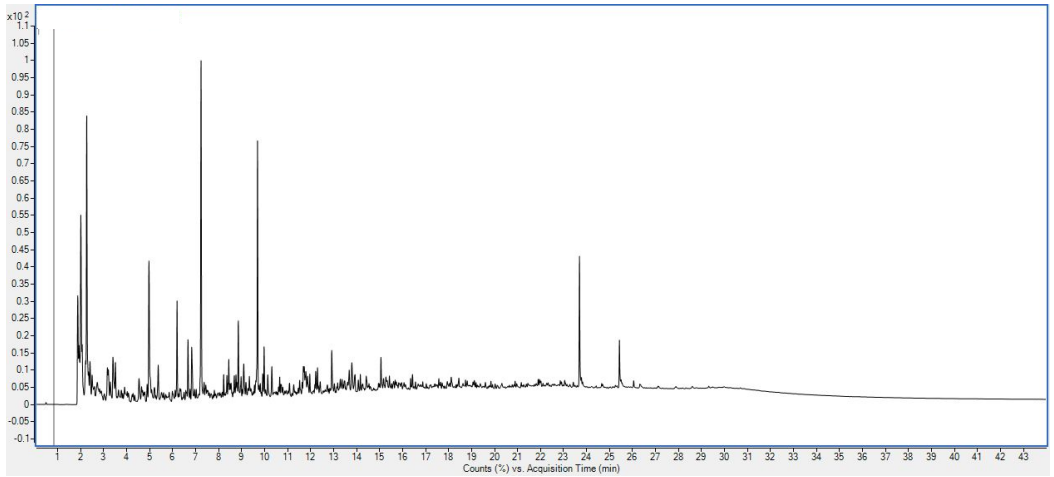
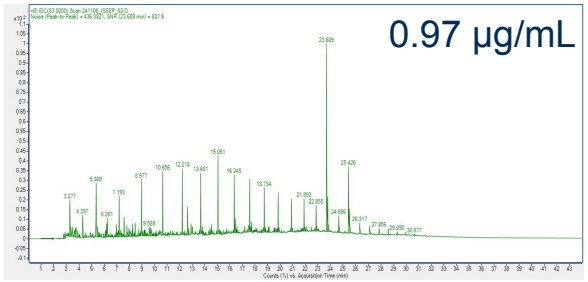
Pyr-GC-MS method as a powerful tool for nanoplastics detection

Pyr-GC-MS method as a powerful tool for MNPs detection: sewage sludge

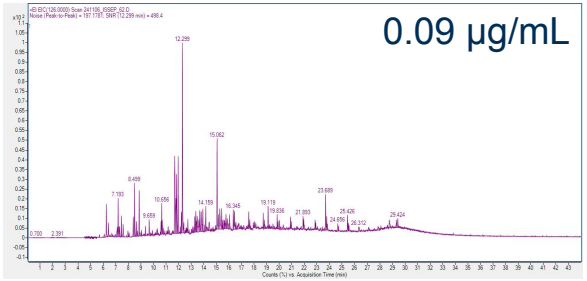
Extensive sample preparation is required



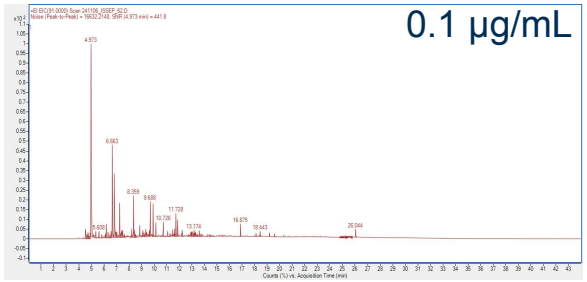
PE



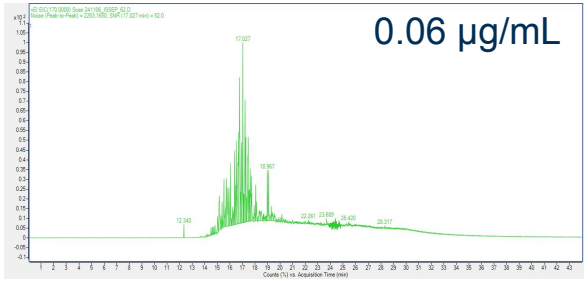
PP



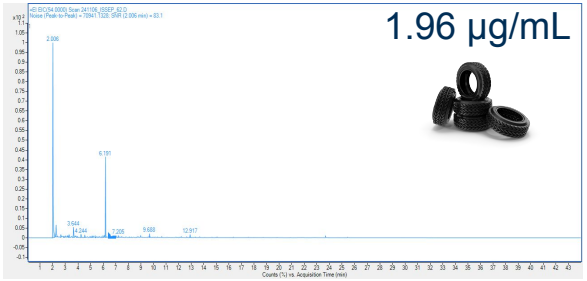
PS



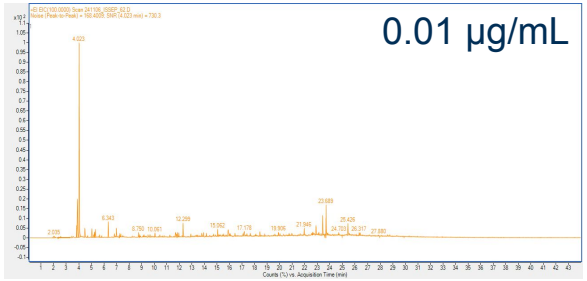
ABS



SBR



PMMA



From Nanoplastics and Beyond

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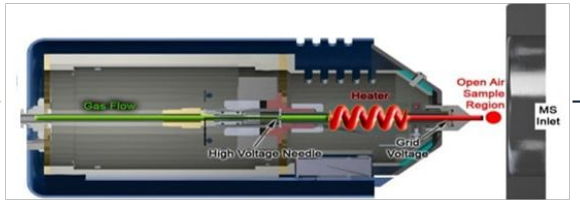
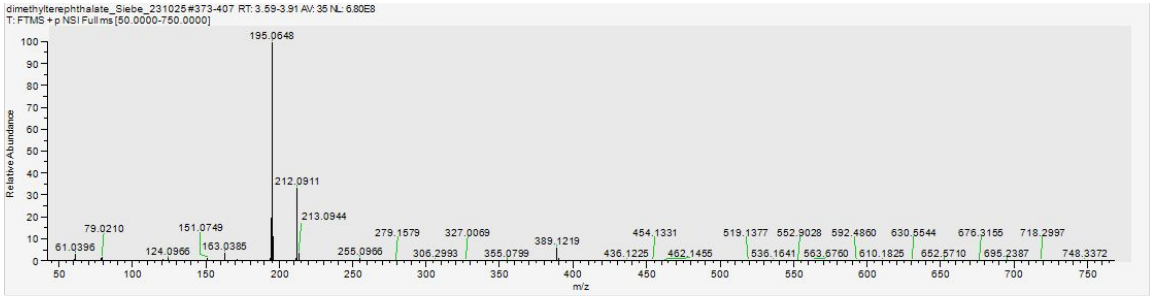
Upstream: DART-MS for additives/leachable compounds detection

a Fast Screening using Ambient Pressure Ionization

Standards

DART ion source



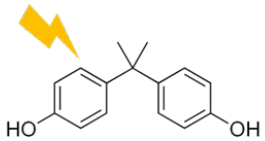


Samples

Database

Formula	Compound
C18H15OP H+	Triphenylphosphine oxide
C9H10O H+	acetyltoluene
C9H18ON2 H+	4-piperidin-4-yl-morpholine (blank)
C18H15OP H+ isotope	Triphenylphosphine oxide
C7H14O7 NH4+	sugar?
C10H10O4 NH4+ isotope	Dimethyl phthalate*
C9H16ON2	unk 1
C8H18O5 H+	PEG-4 (PEG200 monolaurate)*
#N/A	#N/A
C8H4O3 H+	phthalate fragment
C8H9ON H+	Penicillin G fragment*
C10H9O4 H+	Diethylphthalate fragment*
#N/A	#N/A
#N/A	#N/A
C6H14O4 H+	PEG-3 (PEG200 monolaurate*)
C16H22O4 H+	dibutylphthalate
C9H14O2 H+	unk
C12H14O4 H+	Diethylphthalate*
C8H14O4 H+	Suberic acid

Ambient ionization-high resolution mass spectrometer
DART – Thermo Q Exactive



Upstream
FOR WASTE-FREE EUROPEAN RIVERS

Take home message

- **From macro to nanoplastics, size dictates complexity:** As plastics degrade, their physical and chemical behaviors shift—altering how they interact with organisms and ecosystems. This makes size-resolved analysis not just valuable, but essential.
- **It all starts with the sample:** Robust and standardized sample preparation needed.
- **Analytical tools are evolving:** Cutting-edge techniques like µRaman spectroscopy, pyrolysis-GC-MS, and atomic force microscopy (AFM) are expanding our ability to detect, characterize, and quantify plastics.
- **A glimpse of the future:** Emerging methods are now turning toward the detection of chemical additives and degradation products in environemnt that may pose hidden risks.

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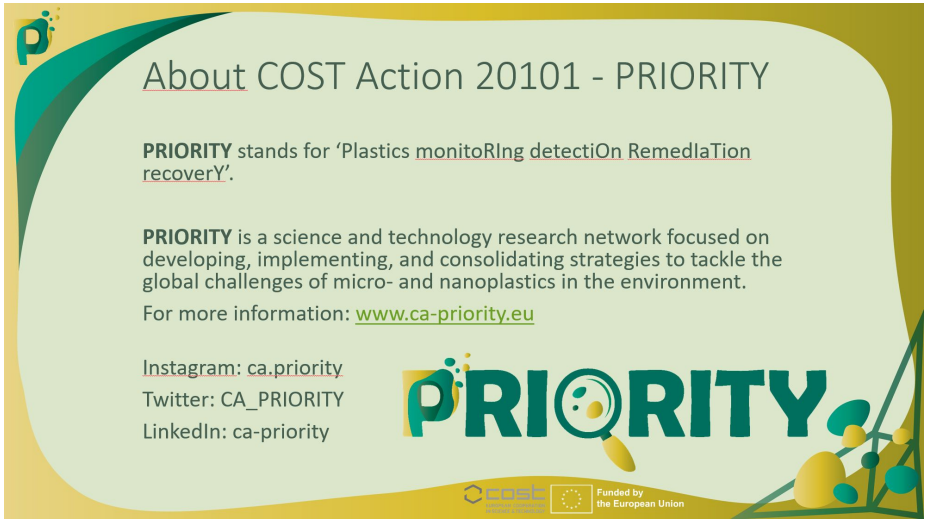

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