

# NO MICROPLASTICS, JUST WAVES.

**Deliverable ACTION B4** 

**Teaching Material and Lessons** 



info@lifebluelakes.eu







'PlasticsEurope'



Dipartimento di Scienze e Ingegneria della Materia, dell'Ambiente ed Urbanistica **SIMAU** 



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#### Executive summary

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Four different training courses editions were carried out during the B4 action. This deliverable includes:

- a) Detailed Program of each edition;
- b) Numbers and general affiliation of the participants;
- c) Mini photo report of the courses;
- d) Final Survey results from the participants;
- e) Teaching materials.





#### SIMAU 1. First Edition: Ancona 05<sup>th</sup>-07<sup>th</sup> December 2022-In person for Italian stakeholders

Dipartimento di Scienze

#### a) Detailed program

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#### Le Microplastiche nel Servizio Integrato

Corso Teorico- Pratico finanziato nell'ambito del progetto europeo Life Bluelakes.https://lifebluelakes.eu/

#### Prima edizione 05 - 06 - 07 Dicembre 2022 (Italiano)

#### Modalità in presenza

Luogo Lezioni teoriche: Università Politecnica delle Marche (UNIVPM), Scienze (Edificio 3) Aula B6-Blocco Aule Sud, Via Brecce Bianche 12, 60131, Ancona https://goo.gl/maps/KGD98tG8dUNYJzi6 Sessioni tecnico-pratiche: impianto di potabilizzazione-impianto di depurazione-piattaforma sperimentale SIMAU e Laboratori Didattici DISVA (LS1) -Università Politecnica delle Marche

#### Programma

| -                  |  |   |
|--------------------|--|---|
| 05 Dicembre        | 13.30<br>14.15-14.30<br>14.30-15.20<br>15.20-16.10<br>16.10-16.30<br>16.30-17.20   | Inizio registrazione<br>Saluti Istituzionali<br>Le Microplastiche: aspetti generali conoscitivi<br>Le microplastiche nel servizio idrico integrato: stato dell'arte e casi studio<br>Coffee Break<br>Metodologie di campionamento   |
| 06 Disembre        | 17.20-18.15<br>09.00<br>09.30-12.30<br>12.30-13.00<br>13.00-14.00<br>14.00-15.00<br>15.00-16.00<br>16.00-16.15<br>16.15-17.00<br>17.00-18.00 | Metodologie di caratterizzazione<br>Incontro presso UNIVPM e trasferimento in Pullman per la sessione<br>pratica<br>Sessione di campionamento in campo<br>Trasferimento ad UNIVPM<br>Light Lunch<br>Impianti di depurazione e di potabilizzazione: ruolo dei processi e delle<br>tecnologie nella rimozione delle microplastiche, esempi numerici di<br>bilanci di massa a scala impianto.<br>Controllo e ottimizzazione della rimozione delle microplastiche nel<br>servizio idrico e nelle diverse unità di trattamento: linee guida gestionali<br>e configurazioni progettuali ottimali, casi studio ed esempi pratici.<br>Coffee Break<br>Analisi di rischio associato alla presenza delle microplastiche nel settore<br>idrico e prospettive nella legislazione tecnica e ambientale<br>Processamento dei campioni per l'analisi della microplastica da matrici<br>del Servizio Idrico Integrato |
| 07 <u>Dicembre</u> | 9.00-13.00<br>13.00-14.00<br>14.30-15.30   | Descrizione della strumentazione tecnica di laboratorio<br>Sessione di pre-processamento dei campioni<br>Sessione di separazione e caratterizzazione delle MPS<br>Light Lunch<br>Visita Opzionale alla Piattaforma Sperimentale di Ingegneria Chimica<br>Ambientale e Sanitaria   |

La partecipazione al corso è gratuita e comprensiva dei materiali didattici e formativi e di eventuali trasferimenti presso gli impianti per le sessioni tecnico-pratiche.

Per qualsiasi informazione si prega di contattare i seguenti indirizzi: s.radini@univpm.it; a.l.eusebi@univpm.it; a.foglia@univpm.it; l.pittura@univpm.it; s.gorbi@univpm.it. Per contatti telefonici 0712204530.



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#### b) Numbers and general affiliation of the participants

| Total Participants                   | N° | 30 |
|--------------------------------------|----|----|
| Water Utilities                      | N° | 15 |
| Private Labs                         | N° | 3  |
| Health and Environmental agencies    | N° | 7  |
| Environmental Companies/Associations | N° | 2  |

#### c) Final Survey results



Che livello di importanza attribuirebbe ai singoli argomenti trattati?





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#### d) Mini photo report

UNIVERSITÀ Politecnica Delle Marche







## 2. Second Edition: 19th-21st April 2023-Ancona-In person for German Stakeholders

a) Detailed program

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#### Microplastics in the Integrated Water

#### Service

Theoretical- Practical Training course organised in the context of the European project Life Bluelakes https://lifebluelakes.eu/

| Second edition | 19 – 20 – 21 April 2023   |  |  |  |  |
|----------------|---|--|--|--|--|
| Modality       | In presence   |  |  |  |  |
| Language       | Practical sessions in German, theoretical sessions with live translation Italian/German   |  |  |  |  |
| Place          | <b>Theoretical sessions:</b> Università Politecnica delle Marche (UNIVPM), Via Brecce Bianche<br>12, 60131, Ancona<br><b>Practical sessions:</b> wastewater treatment plant (Ancona or Jesi), UNIVPM Laboratories<br>and (optional technical visit) UNIVPM pilot hall |  |  |  |  |
|                |   |  |  |  |  |

#### Program

| 10 April | Do be defined | Accival   |  |  |
|----------|---------------|---|--|--|
| 19 April | 19.30         | Welcome dinner  |  |  |
|          | 09.00 - 10.30 | <ul> <li>Microplastics in urban water cycle: Origins – Pathways – Fate</li> <li>Microplastic determinations: Literature and state of the art,<br/>critical aspects and challenges</li> </ul>                        |  |  |
|          | 10.30 - 10.50 | Coffee break  |  |  |
|          | 10.50 - 13.00 | <ul> <li>Sampling methodologies</li> <li>Analytical characterisation methodologies</li> </ul>   |  |  |
| 20 April | 13.00 - 14.00 | Light lunch   |  |  |
|          | 14.00 - 16.00 | <ul> <li>Risk considerations associated with Microplastics in water and<br/>in <u>environment</u></li> <li>Examples and case studies</li> </ul>   |  |  |
|          | 16.00 - 16.20 | Coffee break  |  |  |
|          | 16.20 - 18.00 | <ul> <li>Results and protocol from Blue Lakes project</li> <li>Benchmark assessment</li> </ul>  |  |  |
|          | 9.00-13.00    | <ul> <li>Technical visit to wastewater treatment plant/potabilization<br/>plant</li> <li>Experimental demonstrations using different sampling<br/>methods</li> <li>Sample pre-processing and preparation</li> </ul> |  |  |
| 21 April | 13.00-14.00   | Light Lunch   |  |  |
|          | 14.00 - 16.00 | <ul> <li>Description of the Lab equipment / instrumentation</li> <li>Lab activity for sample sorting</li> </ul>   |  |  |
|          | 16.00 - 16.20 | - Coffee break  |  |  |
|          | 16.20 - 18.00 | <ul> <li>Lab activity for sample characterization</li> </ul>  |  |  |

For any details please contact: <u>s.radini@univpm.it;</u> <u>a.l.eusebi@univpm.it;</u> <u>l.pittura@univpm.it;</u> <u>s.gorbi@univpm.it</u>. Telephone: 00390712204530.







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#### b) Numbers and general affiliation of the participants

| Total Participants                  | N° | 23 |
|-------------------------------------|----|----|
| Water Utilities                     | N° | 10 |
| Environmental Companies/Association | N° | 13 |

#### c) Final Survey results

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How would you assess the course content?





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#### d) Mini photo report





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## 3. Third and Fourth Edition: 12<sup>nd</sup> June 2023 -Ancona Online for Italian Stakeholders and 23<sup>rd</sup> June 2023 Ancona in Person for Italian Stakeholders

#### a) Detailed program



Sintesi dei protocolli e delle attività tecnico-pratiche del Modulo 2.

La partecipazione al corso di formazione è gratuita e comprensiva dei materiali didattici e formativi e di eventuali trasferimenti presso gli impianti e i laboratori per le sessioni tecnico-pratiche.

Il corso ha disponibilità limitata di partecipanti. Le iscrizioni dovranno essere finalizzate entro il 31 Maggio 2023, inviando una e-mail ad uno dei seguenti indirizzi: <u>chietimariagrazia@gmail.com</u>; <u>a.l.eusebi@univpm.it</u>; <u>a.foglia@univpm.it</u> e specificando Nome, Cognome, Ruolo, Azienda, Contatto e Modulo di Interesse.

www.lifebluelakes.eu / info@ilfebluelakes.eu





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#### Le Microplastiche nel Servizio Idrico Integrato - Terza edizione

Seminari Teorico-Pratici nell'ambito del progetto europeo Life Blue Lakes www.lifebluelakes.eu

#### SEMINARIO: METODOLOGIE DI CAMPIONAMENTO E CARATTERIZZAZIONE DELLE MPs

Modalità e data: In presenza -23 <u>Giugno</u> 2023 (Lingua Italiano) Luogo: impianto di potabilizzazione / impianto di depurazione / piattaforma sperimentale UNIVPM e Laboratori di UNIVPM

#### Crediti rilasciati dall'Ordine Professionale degli Ingegneri della Provincia di Ancona: 6 CFP

Modulo 2: Lezioni Tecnico-Pratiche- 23 Giugno 2023

|                   | 9.30          | Arrivo all'impianto di depurazione di Jesi e Inizio registrazione –<br>Via della Barchetta, 60035 Jesi AN - |
|-------------------|---------------|---|
|                   |               | https://goo.gl/maps/MXJVvHyTU82yV5Tt9   |
|                   | 9.45-10.30    | Saluti del Gestore e descrizione impianto   |
| 23/06/2023        | 10,30 - 11,15 | Metodologia di campionamento W86 con setacci  |
| matuna            | 11,15 - 12,00 | Metodologia di campionamento 🚜 con campionatore automatico  |
|                   | 12,00-12.30   | Descrizione sulla fase di recupero del campione   |
|                   | 12.30-13.00   | Trasferimento ad UNIVPM - Via Brecce Bianche 12, 60131 Ancona -<br>https://goo.gl/maps/16WcCwbNuQLegz3E7    |
| Bahsa,<br>Bradza, | 13,00-14,00   | Light Lunch → Aula Azzurra, Edificio Scienze 3  |
| 23/06/2023        | 14.00-15.00   | Descrizione della strumentazione tecnica di laboratorio   |
| 0000000           | 15,00-16,00   | Sessione di oto-processamento dei campioni  |
|                   | 16.00-17.00   | Sessione di separazione e caratterizzazione delle MBs   |

L'appuntamento mattutino per la sessione tecnico-pratica del corso <u>è prevista direttamente</u> all'impianto di depurazione di Jesi. Nell'eventualità di problemi logistici per gli iscritti preghiamo di mandare un'e-mail per tempo ai seguenti indirizzi: <u>chietimariagrazia@gmail.com</u>; <u>a.l.eusebi@univpm.it</u> o di chiamare ai numeri: 3420447463 e 3209666407.





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#### b) Numbers and general affiliation of the participants

| Total Participants  | N° | 166 |  |
|---|----|-----|--|
| Water Utilities   |    |     |  |
| Environmental Companies/Association                                   |    |     |  |
| Self-employed engineers mainly of civil/environmental infrastructures |    |     |  |

#### c) Final Survey results



Che livello di importanza attribuirebbe ai singoli argomenti trattati?





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#### d) Mini photo report

UNIVERSITÀ Politecnica Delle Marche





UNIVERSITÀ Politecnica delle marche Dipartimento di Scienze della Vita e dell'Ambiente **DISVA**  Dipartimento di Scienze e Ingegneria della Materia, dell'Ambiente ed Urbanistica **SIMAU** 



## **Teaching Material**



# The project LIFE BLUE LAKES

# Università Politecnica delle Marche



www.lifebluelakes.eu / info@lifebluelakes.eu

#### Università Politecnica delle Marche- UNIVPM-www.univpm.it













## **ENGINEERING FACULTY- Department of Science and Engineering** of Materials, Environment and Urban Planning – SIMAU

The research fields can be divided into the following 5 macro-areas:









**Digital solutions for** carbon footprinting and environmental /

> economic assessment

Advanced water and wastewater treatment





**Organic waste** treatment and valorisation

Prof. Eng. Francesco FATONE PhD Full Professor Chemical Engineering GROUP COORDINATOR

Prof. Eng. Anna Laura EUSEBI PhD Associate Professor Sanitary **Environmental Engineering** GROUP TECHNICAL DIRECTOR

Biologist

Eng. Massimiliano SGROI PhD Assistant Professor in Environmental Engineering





Eng. Nicola LANCIONI PhD candidate









Eng. Josue GONZALEZ-CAMEJO PhD PostDoc in Environmental Engineering

Doct. Valeria FRATESI

Eng. Giulia CIPOLLETTA PhD candidate









Eng. All POURZANGBAR

Data analyst

Dr. Marco PARLAPIANO

Chemist, PhD candidate

Eng. Alessia FOGLIA PhD candidate

Eng. Cecilia BRUNI PhD candidate

Eng. Paolo CROCETTI PhD candidate

Eng. Enrico MARINELLI PhD candidate

Eng. Serena RADINI PhD candidate

Eng. Corinne ANDREOLA PhD candidate





Eng. Çağrı AKYOL, PhD PostDoc in Environmental Engineering and Sciences Giorgio CONCETTONI Pilot Operator









# Lab. Ecotoxicology and Environmental Chemistry

## Dipartimento di Scienze della Vita e dell'Ambiente **DISVA**



Prof. Francesco Regoli







Prof. Stefania Gorbi Dr. Marica Mezzelani Dr. Alessandro Nardi Dr. Giuseppe d'Errico



Prof. Maura Benedetti



Dr. Giulia Lucia



Dr. Lucia Pittura



Dr. Michela Panni



Dr. Carola Mazzoli



Dr. Melissa Orsini



Dr. Veronica Vivani



Dr. Marta Di Carlo



Dr. Daniele Fattorini



|          | 09.00 - 10.45 | - Official Welcome  |  |  |  |  |
|----------|---------------|---|--|--|--|--|
|          |               | - Global Nature Fund Presentation   |  |  |  |  |
|          |               | <ul> <li>Bluelakes Project</li> </ul>   |  |  |  |  |
|          |               | <ul> <li>Microplastics in urban water cycle: Origins – Pathways – Fate</li> </ul>                               |  |  |  |  |
|          |               | <ul> <li>Microplastic determinations: Literature and state of the art,</li> </ul>                               |  |  |  |  |
|          |               | critical aspects and challenges   |  |  |  |  |
|          | 10.45 - 11.00 | offee break   |  |  |  |  |
| 20 A 11  | 11 00 - 13 00 | - Sampling methodologies  |  |  |  |  |
| 20 April | 11.00 15.00   | <ul> <li>Analytical characterization methodologies</li> </ul>   |  |  |  |  |
|          | 13.00 - 14.00 | Light lunch   |  |  |  |  |
|          |               | - Risk considerations associated with Microplastics in water and  |  |  |  |  |
|          | 14.00 - 16.00 | in environment  |  |  |  |  |
|          |               | <ul> <li>Examples and case studies</li> </ul>   |  |  |  |  |
|          | 16.00 - 16.20 | Coffee break  |  |  |  |  |
|          | 10.00 19.00   | <ul> <li>Results and protocol from Blue Lakes project</li> </ul>  |  |  |  |  |
|          | 10.20 - 18.00 | - Benchmark assesment   |  |  |  |  |
|          | 19.30         | - Welcome dinner in Giardino Restaurant   |  |  |  |  |
|          |               | https://ristorantegiardinoancona.it/  |  |  |  |  |
|          |               | - Technical visit to wastewater treatment plant/potabilization  |  |  |  |  |
|          | 9.00-13.00    | plant   |  |  |  |  |
|          |               | <ul> <li>Experimental demonstrations using different sampling</li> </ul>  |  |  |  |  |
|          |               | methods<br>Sample are proceeding and properties   |  |  |  |  |
| 21 April |               | - Sample pre-processing and preparation   |  |  |  |  |
| 21 April | 13.00-14.00   | Light Lunch   |  |  |  |  |
|          | 14.00 - 16.00 | <ul> <li>Description of the Lab equipment / instrumentation</li> <li>Lab activity for sample sorting</li> </ul> |  |  |  |  |
|          | 16.00 16.20   |   |  |  |  |  |
|          | 10.00 - 10.20 | - conee break   |  |  |  |  |
|          | 40.00 40.00   |   |  |  |  |  |

Special Thanks to



<image><image><image><image><image><image>

**DEPURAZIONI BENACENSI** 



## **Microplastics in the Integrated Water Service**



In the European Green Deal and in the new Action Plan for Circular Economy, the European Commission announced an initiative to face the unintentional release of microplastics in the environment.

The initiatives aim to develop measures for labelling, standardisation, certification and regulation on the unintentional release of microplastics in the environment, including:

- Measures to increase microplastics removal in all the phases of the life cycle of the products.
- Development and harmonisation of methods to detect the unintentional release of microplastics, especially from tires and tissues, and provide harmonised data on microplastics concentration in marine water.
- Fill the gap on scientific knowledge on risk about the presence of microplastics in the environment, in drinking water and in food.





www.lifebluelakes.eu

Life Blue Lakes aims to face the issue of microplastics in lakes through actions of governance, training, scientific research, information e sensibilization.

Main actions are being deployed in **Garda**, Bracciano, Trasimeno and Castreccioni lakes, in Italy and in Costanza and Chiemsee lakes in Germany.

BENEFICIARI ASSOCIATI





LEGAMBIENTE

BENEFICIARIO COORDINATORE



Autorità di Bacino Distrettuale Appennino Centra



Lake Constance Foundation





AZIONALE PER LE NUOVE TECNOLOGIE L'ENERGIA E LO SVILUPPO ECONOMICO SOSTENIBIL





ABDAC - Autorità di Bacino Distrettuale dell'Appennino Centrale ENEA – Agenzia Nazionale per le nuove tecnologie, l'energia e lo sviluppo economico UNIVP - Università Politecnica delle Marche **Global Nature Fund** 

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## Origin of Microplastics in the Integrated Wat Service

Microplastics are defined as all plastic fragments whose dimensions are < 5 millimeters.





## WHAT ARE MICROPLASTICS?

**Microplastics**: plastic fragments or particles smaller than 5 millimetres.

**Primary microplastics**: microbeads, fillers on artificial turf sports pitches or additives for cleaning, cosmetic products, exfoliation...

**Secondary microplastics**: result of chemical and physical ageing and degradation processes

## $\rightarrow$ IMPACTS ON ECOSYSTEMS AND

- Can easily enter in contact with aquatic organisms
- May be vectors for toxical compounds, which may act as potentially endocrines distructors, once entered in the organsim, interfering with the hormonal functions.
- Have elevated adsorbance capacity and can transport pollutant substances present in water
- Can be occupied by microorgansism and pathogens.









www.lifebluelakes.eu Blue Lakes

## Life Blue Lakes - Project areas in Italy and in Germany





## Lake Garda

370 km<sup>2</sup>, located between three regions (Trentino Alto Adige, Veneto and Lombardia). Tourism

## Lake Trasimeno

128 km<sup>2</sup>, Umbria. Nature conservation area, tourism

# Lake Bracciano

56.5 km<sup>2</sup>, Lazio. Drinking water reservoir, nature conservation area, tourism



## Lake Castrreccioni

128 km², Marche. Drinking water reservoir, tourism



## Lake Constance

536 km<sup>2</sup>, international border between Germany, Switzerland, and Austria

Drinking water reservoir, high standards for water treatment

Nature conservation area, tourism

## Lake Chiemsee

79.9 km<sup>2</sup>, Bavaria nature conservation area, tourism

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## **Microplastics in Italian lakes**







- Strategic basin for drinking water supply
- Presence of combined sewerage overflows
- Sewer network undergoing renovation
- Integrated Water Service managed by various companies
- Strong variability and relevance of anthropic and natural pressures

#### Plastic waste threatens lakes as well as oceans - BBC News 07 Ottobre 2013 - BBC

#### https://www.bbc.com/news/science-environment-24434378

# Plastic waste threatens lakes as well as oceans

By Matt McGrath Environment correspondent, BBC News

③ 7 October 2013

<



Researchers found levels of plastics in Lake Garda similar to those in marine environments

Pollution with plastic waste is not confined to the oceans but poses a growing threat to lakes as well.

## **Microplastics in Italian lakes**



| Lake<br>(Sampling date) | Sample<br>Location   | Wind<br>direction | Trawling direction | Transect | particles/km <sup>2</sup> |
|-------------------------|----------------------|-------------------|--------------------|----------|---------------------------|
| 10                      | Lovere-Pisogne       | SW                | NW-SE              | ISI      | 57000±36000               |
| IS<br>(28/06/2016)      | Riva di Solto-Castro | SW                | N-S                | IS2      | $15000 \pm 11000$         |
| (28/00/2010)            | Predore-Pilzone      | S                 | E-W                | IS3      | 50000±14000               |
| 2.64                    | Ispra-Monvalle       | Absent            | SW-NE              | MA1      | 45000±13000               |
| MA<br>(06/07/2016)      | Arona-Angera         | NE                | SW-NE              | MA2      | 41000±32000               |
| (00/07/2010)            | Lisanza-Dormelletto  | S                 | NW-SE              | MA3      | 29000±17000               |
|                         | Riva-Torbole         | N                 | SW-NE              | GAI      | 55000±29000               |
| GA<br>(11-12/07/2016)   | Sirmione             | Absent            | SW-NE              | GA2      | 4000±2700                 |
| (11-12/07/2010)         | Desenzano            | Ν                 | SW-NE              | GA3      | 16000±13000               |

43.000 part./km<sup>2</sup> Laurentian Great Lakes 11.000-36.000 part./km<sup>2</sup> Swiss lake

lie

22 TRANSECTS  $\checkmark$ **843 PARTICLES**  $\checkmark$ **390 FTIR SPECTRES**  $\checkmark$ 

82.4



Maggiore 39.000 part./km<sup>2</sup>

Environmental Pollution 236 (2018) 645-651 Contents lists available at ScienceDirect **Environmental Pollution** journal homepage: www.elsevier.com/locate/envpol

Microplastic pollution in the surface waters of Italian Subalpine

Maria Sighicelli a.\*, Loris Pietrelli a, Francesca Lecce a, Valentina Iannilli a, Mauro Falconieri<sup>a</sup>, Lucia Coscia<sup>b</sup>, Stefania Di Vito<sup>b</sup>, Simone Nuglio<sup>b</sup>, Giorgio Zampetti<sup>b</sup> <sup>4</sup> Italian National Agency for New Technologies, Energy and Sustainable Economic Development (ENEA) CR Casaccia, Rome, Italy

Transects near river inputs and narrowings are by far



www.lifebluelakes.eu

## **Microplastics in the Integrated Water Service**



## **PROJECT, OBJECTIVESAND ACTIONS**

The LIFE Blue Lakes project contributes to reducing the contamination of lakes with microplastics.

Exemplary project areas are the **lakes Garda**, **Bracciano** and **Trasimeno** in **Italy** and **Lake Constance** and **Lake Chiemsee** in **Germany**. Further Italian and European lake communities will be involved in the promotion and dissemination of good practices.

 $\circledast$ 

The LIFE programme is the EU's largest funding instrument for the environment and climate action created in 1992 for supporting a wide range of measures and projects to safeguard biodiversity and nature, for enhancing the environmental policy and awareness raising on environmental issues.

## LIFE BLUE LAKES SPECIFIC AIMS AND ACTIONS:

Supporting local administrations and enhancing the engagement of local economic entities located close to lakes by a Participatory Process to draft a Lake Paper. This document may suggest to local communities a set of solutions tailored to their specific territorial context, proposing monitoring programmes or techniques to improve wastewater treatment processes, discharge limits, provisions for reducing the impact resulting from companies and families as well as awareness raising initiatives for residents.

Design and test a standard microplastic monitoring protocol in the two pilot areas of the Trasimeno and Bracciano lakes in order to assess microplastics levels in these basins.

A technical protocol to reduce the release of microplastics from wastewater treatment plants based on the experiences of a pilot plant at Lake Garda. Wastewater treatment plant operators in Italy and Germany are closely involved and trained to ensure a comprehensive transfer of knowledge.

Cooperating with relevant companies (plastics, tyre, outdoor and cosmetics industries) and carrying out an advocacy campaign with the aim of reducing and preventing further contamination of lakes with microplastics.

Raising public awareness in Italy and Germany and promoting behaviours to prevent plastic waste.

Enhancing existing regulatory framework to face local microplastic pollution and influencing national and European political agenda in Italy and Germany through the development of a Lake White Paper which will be presented to the relevant authorities (Ministries of Environment, Agriculture, Health, Economic Development, Regions, Basin Authorities, etc.) to promote a legislative initiative to protect lakes from microplastics at national and European level.

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Blue
Lakes

- ✓ Support local authorities and stakeholders through a partecipative process for the development of LAKE PAPER: a volountary proposal for management good practices and the diffusion of circular economy models.
- ✓ Reduce microplastics presence connected to wastewater treatment plants, through the development of a Technical Protocol and training of water professionists.
- ✓ Cooperate with local industries (plastic, tires producers, cosmetics) to develop solutions to reduce and prevent further primary loads of microplastics in lakes.
- ✓ Raise awareness, to prevent the spread of plastic waste in the environment;
- ✓ Improve the existing regulatory framework to address microplastic pollution in lake basins, influencing the policy agenda at Italian, German and European level.













- Promote a participatory process to develop the Lake Paper: a voluntary commitment by local stakeholders for the implementation of good management practices and the dissemination of circular economy models aimed at protecting lakes and reducing the impact of economic activities.
- Monitor the release of microplastics from treatment plants by developing a technical protocol and training operators.
- Cooperate with stakeholders (plastics, pneumatics and cosmetics) to find and develop solutions to reduce and prevent further primary loads of microplastics;
- Raise public awareness and promote behaviors to prevent plastic waste;
- Improve the existing regulatory framework to address local microplastic pollution and influence the national and European policy agenda in Italy and Germany.

The measures are addressed to local authorities, businesses, industries and communities in the lake regions. Additional Italian and European lake communities will be involved in the promotion and dissemination of good practices.





## **Products of the project**

- **3 Lake Papers in Italy and 2 Lake Papers in Germany** drafted through a participatory process involving three main types of stakeholders (local administrators, cultural associations/school sector, economic/tourist sector);
- Manifesto del Lago, a tool that allows lake communities to undertake a participatory process aimed at protecting the lakes by reducing and mitigating the effects associated with the presence of microplastics in the waters;
- 2 summer awareness campaigns implemented for the promotion of the Lake Papers and the Manifesto del Lago;
- **Monitoring protocol** that provides methodological procedures for sampling and analyzing microplastic content in lakes, to support freshwater MP monitoring and assessment programmes.
- **Training courses** for ARPA technicians for the use/application of this Protocol (40 participants)





## **Products of the project**



- Monitoring protocol and operating manual to optimize the sampling and analysis procedures for microplastics detection in treatment plants
- Report on the results obtained from sampling campaigns in drinking water and wastewater treatment plants
- Training courses for Integrated Water Service professionals





- **Contest** for students
- Awareness/information campaign for stakeholders (plastics, pneumatics and cosmetics). More than 250 companies reached.



## UNIVPM activities in LIFE Blue Lakes project: MPs sampling campaign

- LAKE GARDA
- ✓ LAKE CASTRECCIONI
- WASTEWATER TREATMENT PLANTS
- DRINKING WATER TREATMENT PLANTS

- Strategic catchments
- Presence of overflows, WWTPs and DWTPs
- Ongoing upgrading of the sewer system
- Water service managed by various water utilities
- Variations in natural and anthropic pressures

## **PROMOTION OF GOOD PRACTICES EXPERIMENTED ON THE PILOT LAKES**

## Sampling in real environment

- evaluation of sampling systems and protocols
- ensure the transferability and replicability of the project results

## MPs sample preparation and characterization

#### **Optimization guidelines for the treatment stages**

critical audit of treatment technologies to improve the removal of microplastics

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# **Microplastics: general overview**

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# **THE AGE OF PLASTIC**

# Plastic production increased worldwide over the last 60 years, from 0.5 million tons y-1 in 1960, to 390 million tons in 2021



Plastic-the Facts 2022\_PlasticEurope


# **Global plastic production in relation to different** polymers typologies



Plastic-the Facts 2022 PlasticEurope

# PLASTIC POST-CONSUMER WASTE

treatment in 2020 (preliminary data)



# The plastic journey... fom the land to the sea



Karbalaei et al., 2018. Env. Sci. Pollut. Res. Int.

#### PLASTICS IN THE MARINE ENVIRONMENT: WHERE DO THEY COME FROM? WHERE DO THEY GO?

#### eunomia



# PLASTICS AND MICROPLASTICS...THE TIP OF THE ICEBERG

#### **Microplastics: plastic particles < 5 mm**





# **ORIGIN OF MICROPLASTICS**

Primary MPs - Direct introduction:

- the micron-sized plastic particles used as exfoliants in cosmetic formulations
- industrial abrasives in synthetic 'sandblasting' media (beads of acrylic plastics and polyester)
- "nurdle" pre-production plastic pellets



Secondary MPs - Weathering breakdown of meso- and macroplastics









**Textile fibers** 





Are we speaking the same language? Recommendations for a definition and categorization framework for plastic debris

# Spherical macroplastic?

Hartmann et al., 2019. Environmental Science & Tecnology.

# **Microplastic definitions from scientific literature and international agencies**

|                              |      |   | nanoplastics | microplastics | mesoplastics                   | macroplastics |
|------------------------------|------|---|--------------|---------------|--------------------------------|---------------|
| Gregory & Andrady4           | 2003 |   |              | 67–500 μm     | 5-10 1-                        | -15 cm        |
| Browne et al.52              | 2007 | <1 μm                                   | 1–100        | 00 μm         | •••••• >5 mm                   |               |
| Moore <sup>53</sup>          | 2008 |   | 5000 µm      |               | >5 mm                          | ***********   |
| Ryan et al.54                | 2009 | <200                                    | 0 µm         |               | 2–20 mm >2                     | 2 cm          |
| Costa et al.55               | 2010 | <1000                                   | ) µm         |               |                                |               |
| Desforges et al.56           | 2014 |   | 1            | –5000 µm      |                                |               |
| Wagner et al.57              | 2014 |   | <20 μm       | 20–5000 µm    | 5-25 mm                        | >2.5 cm       |
| Koelmans et al. <sup>7</sup> | 2015 | •• 1–100 nm ••••••                      | µm-scale-{   | 5000 µm       | >5 mm                          |               |
| Andrady <sup>58</sup>        | 2015 | <1 μm                                   | 1–100        | 00 µm         | 1–25 mm                        | 2.5–100 cm    |
| Koelmans ( NOAA              | 2017 | • |              | <335 µm 335–5 | 6000 µm <mark> &gt;5 mm</mark> |               |

| ΝΟΟΑ                        | 2009             |                  | *********        | <5000 µ          | um               |         |                  | •••••            |                   |
|-----------------------------|------------------|------------------|------------------|------------------|------------------|---------|------------------|------------------|-------------------|
| EU Commission <sup>22</sup> | 2011             | 1–100 nm         |                  |                  |                  |         |                  |                  |                   |
| EU MSFD WG-GES49            | 2013             |                  |                  |                  | •••••            | 20–5000 | ) µm             | 5-25 mm          | >2.5 cm           |
| GESAMP <sup>23</sup>        | 2015 ·····       | •••••            | <1               | μm               | 1–1              | 000 µm  | 1–2              | 5 mm             | 2.5–100 cm        |
| EFSA (CONTAM)60             | 2016             | 1–100 nm         |                  |                  | 0.1–50           | 00 µm   |                  | •••••            |                   |
|                             | L                | !                |                  | <u> </u>         | 1                |         | 1                |                  | <b></b>           |
|                             | 10 <sup>-9</sup> | 10 <sup>-8</sup> | 10 <sup>-7</sup> | 10 <sup>-6</sup> | 10 <sup>-5</sup> | 10-4    | 10 <sup>-3</sup> | 10 <sup>-2</sup> | particle size [m] |
| Hartmann et al., 2019       | 1 nm             |                  |                  | 1 µm             |                  |         | 1 mm             | 1 cm             |                   |

Lakes

## **CRITERIA FOR MACROPLASTIC CATEGORIZATION**







# PLASTIC E MICROPLASTIC... IT'S A SIZE MATTER

### MACROPLASTIC > 5 cm



MESO PLASTIC Small macroplastic 5mm - 5 cm



#### Large microplastic 1- 5 mm



#### MICROPLASTIC 1- 1000 µm (1mm)





<1µm (0,001mm) nano-plasticS



# **Considering shape MPs can be defined as**





ados unles miles andes miles miles miles Morenates



dee stales vales vales vales vales voles ender ender 10









FONDAZIONE SOLVING

Technologies and circular economy to counteract the impact of plastic in the Conero Riviera (Italy)



# **EPHEMARE**

#### ECOTOXICOLOGICAL EFFECTS OF MICROPLASTICS IN MARINE ECOSYSTEMS







### IL NOSTRO IMPEGNO A TUTELA DEI LAGHI. NO MICROPLASTICS, JUST WAVES.

| Ministeri | , della Salute |
|-----------|----------------|

Direzione generale della ricerca e dell'innovazione in sanità BANDO RICERCA FINALIZZATA 2019 fondi esercizio finanziario 2018-2019

Codice progetto: RF-2019-12370587

#### Titolo progetto:

MicroPLASTICs in edible aquatic organisms: ecotoxicological effects, transfer of chemical and biological CONtaminants and susceptibility to bacteria biodegradation (PLASTICON)

Destinatario Istituzionale: Istituto Zooprofilattico Sperimentale dell'Umbria e delle Marche



PlasticsEurope

1

Principles for the analysis of plastics and microplastics present in the environment

## MPs extraction from environmental matrices: from sample collection to visual sorting



























# Quantification of MP in biotic and abiotic matrices Different approach → Different results



# **GENERAL CONCERN FOR MICROPLASTICS IN BIOTA**

Accumulated in biota, they induce pseudo-satiety, physical damages, mechanical blockage of gastrointestinal system, respiratory and locomotory appendages...but they can also release chemical additives, adsorb pollutants from seawater and transfer to trophic webs...





# ...BIOACCUMULATION: MICROPLASTICS AS PAH VECTOR







BIOCELL 2022 46(1): 37-49

#### Emerging environmental stressors and oxidative pathways in marine organisms: Current knowledge on regulation mechanisms and functional effects

MAURA BENEDETTI\*; MARIA ELISA GIULIANI; MARICA MEZZELANI; ALESSANDRO NARDI; LUCIA PITTURA; STEFANIA GORBI; FRANCESCO REGOLI





# Morphological and polymeric characterization of MPs ingested by marine organisms



# **Microplastics in superficial sea water :** polymeric characterization



SOURCE: Plastics Europe Market Research Group (PEMRG)

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coatings, etc.

# **Field studies... biota investigations**

Thousands of analyzed organisms, more than 50 species, high ingestion frequency (about 20-30% reaching 90% in some cases), low number of MPs, independent from biological and ecological characteristics



# ...particular attention to micofibers due to airborne contamination...







25







#### **NO MICROPLASTICS, JUST WAVES.**

# Thank you for the attention!

Deliverable ACTION B3

#### TECHNICAL REPORT AND OPERATING MANUAL ON THE IMPROVEMENT OF THE TREATMENT PROCESS

Manuale tecnico-operativo per l'analisi delle microplastiche negli

impianti di trattamento acque

#### 

#### A cura di:

Dr. Lucia Pittura (DISVA, UNIVPM) Dr. Veronica Vivani (DISVA, UNIVPM) Dr. Serena Radini (SIMAU, UNIVPM) Dr. Alessia Foglia (SIMAU, UNIVPM) Prof. Stefania Gorbi (DISVA, UNIVPM) Prof. Anna Laura Eusebi (SIMAU, UNIVPM) Prof. Francesco Fatone (SIMAU, UNIVPM)

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# Microplastics in urban water cycle: Origins – Pathways – Fate Università Politecnica delle Marche



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The origin of microplastics in the environment and in water





world production of plastics (packaging) > 320 million tons

60-80% of global waste is plastic (Derraik, 2002)

5-13 million tons of plastic waste enter the oceans each year (World Economic Forum, 2016).

Decomposition through chemical-physical mechanisms, such as photo-oxidative processes and mechanical forces

These processes break down larger plastic residues into smaller particles, forming microplastics (smaller than 5 mm in size) and nanoplastics (between 1 and 100 nm in size).

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The origin of microplastics in the environment and in water



Approximately 42,000 tonnes of microplastics end up in the environment every year, when products containing them are Furthermore, used. the of release accidentally formed microplastics (when larger pieces of plastic wear into European surface out) estimated waters is to be around 176 000 tonnes per year.

Source: https://echa.europa.eu/it/hot-topics/microplastics





#### The origin of microplastics in the environment and in water

Figure 2.3 Sources of primary microplastic release to the environment, global, estimated yearly releases, per cent



Vehicle tire abrasion is the predominant source of microplastic release into the environment, followed by road markings and the so-called "city dust", linked to built areas.

Source: UNEP (2018)

In addition to this: the abrasion of shoe soles; peeling and flaking paints and coatings; textile microfibres released through washing; road and sewage run-off; cosmetics





#### Microplastics transport



#### WATER TRANSPORT

- civil waste water
- industrial waste water
- urban runoff water
- agricultural runoff water

domestic microplastics: synthetic textile fibers lost during washing, wear products of plastic materials, seals, paints and microbeads

industrial microplastics: cement pastes, drilling fluids, rust removers and paints

Pipes subjected to wearing

Sewer overflows (mixed sewage systems in the presence of intense atmospheric precipitation)

#### **ATMOSPHERIC TRANSPORT**

Atmospheric deposition: transport mechanisms and tendency of the smallest particles to be transported due to the effect of the wind. This would explain the discovery of microplastics even in places where human activity is reduced or absent.

#### LAND TRANSPORT

- road marking paints
- tire wearing
- city dust: result of the abrasion of various plastic objects common in urban areas (e.g. shoe soles, synthetic turf, ...)





**Microplastics in the internal waters** 



Microplastics originating from industrial and urban activities can be conveyed into the sewage system, arriving at wastewater treatment plants. Even though these facilities can remove even more than 90% of MPs from wastewater, due to the large volumes treated, millions of microplastics are discharged into the environment every day.

Inland waters certainly represent one of the ideal means for their diffusion, as they are able to collect and transport contaminants from numerous environmental sources, even over long distances. From here, microplastics can reach marine environments (often the final receptors of plastic pollution), fragment into smaller particles (even nanoplastics, which require different analytical techniques and toxicological assessments to be detected) or reach drinking water plants.

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# Microplastic determinations: Literature and state of the art, critical aspects and challenges Università Politecnica delle Marche



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Many studies have been conducted on the presence and dispersion of microplastics in the marine environment (since the 1970s) but even freshwater is not immune to this problem.

To date, for inland waters and especially for lake waters, there are gaps in:

- quantity and distribution of microplastics in rivers and lakes
- characterization of the phenomenon
- time trends
- standards and protocols for monitoring, sample processing and analysis
- effects on aquatic organisms and ecosystems
- Information and awareness of the local population



The legislation that establishes indicators and limits to monitor the quality of inland waters (Water Framework Directive 2000/60) does not consider the presence and effects of microplastics on their condition.









#### **Dimensions**







#### **Dimensions**



## **Microplastics as Emerging Contaminants in the Integrated Water Service**



- Microplastics include a wide class of contaminants
- They are characterized by strong heterogeneity
- They do not have the same behavior as other contaminants, such as dissolved or uniformly suspended chemicals
- The heterogeneities are amplified by the wide range of matrix types and samples that are analysed
- There are still many persistent knowledge gaps related to microplastics in water and sludge.
- Challenges in evaluating the full spectrum of characterization of microplastics in complex samples.
- Many microplastics characterization parameters are defined analytically, such as size limits due to sample processing or analytical techniques
- Furthermore, there is no instrument that can quickly measure microplastic content across the full spectrum of types and sizes.
- This introduces challenges associated with effectively defining "microplastics"





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### **Microplastics in the Integrated Water Service**



#### PRODUCTION





SAMPLING AND ANALYSIS



## **Microplastics in the Integrated Water Service**



#### Measure



- Not homogenously distributed
- Variable dimensions
- Variable densities
- Different types with different chemical-physical characteristics (e.g. adhesion)







#### **DRINKING WATER – TREATED WATER:**

- $\rightarrow$  Low concentrations
- $\rightarrow$  Less input variability
- $\rightarrow$  Count of MPs: n. MPs/m3

#### WASTEWATER:

- $\rightarrow$  Higher concentrations
- $\rightarrow$  Greater input variability
- $\rightarrow$  Count of MPs: n. MPs/l or no. MPs/m3

#### SLUDGE:

- $\rightarrow$  Higher concentrations
- → Discontinuity (longer storage/retention times)
- → Count of MPs: n. MPs/d or no. MPs/m3 (some ref also in weight e.g. mg/g)





### **DWTPs**



- INFLUENTIAL CHARACTERIZATION:
  - Uptake
  - Plant influent
- CHEMICAL-PHYSICAL TREATMENTS
  - Clari-flocculation and sedimentation
  - sludge
- OXIDATIVE TREATMENTS
  - Ozonation
- PHYSICAL TREATMENTS
  - Sand filtration
  - Carbon Filtration (GAC or BAC)
  - Backwash water
- DISINFECTION TREATMENTS
  - Chlorination
  - UV disinfection
- EFFLUENT CHARACTERIZATION:
  - System output
  - Distribution





#### **DWTPs**



Be careful to chemical treatments





#### **DWTPs – surface water**







#### **DWTPs** –groundwater







### **DWTPs – treatment units**

| INFLUENT              | UdM       |
|-----------------------|-----------|
| SAMPLING POINT        |           |
| Flowrate              | l/s       |
| CHEMICAL AND PHYSICAL |           |
| CHARACTERISATION      |           |
| рН                    | -         |
| Turbidity             | NTU       |
| Temperature           | °C        |
| Dissolved Oxygen      | mg/L      |
| Conductivity          | microS/cm |
| Others available      |           |

| SAND FILTERS           | UdM    |
|------------------------|--------|
| N° units               | N°     |
| N° working units       | N°     |
| Specific surface       | m2     |
| Volume                 | m3     |
| Contact time           | minuti |
| Velocity               | l/m2/h |
| Flux of backwash air   | l/s    |
| Flux of backwash water | l/s    |
| Backwash frequency     |        |

| GROSS FILTER     | UdM |
|------------------|-----|
| N° units         | N°  |
| N° working units | N°  |
| Pores            | mm  |

| CARBON FILTERS   | UdM    |
|------------------|--------|
| N° units         | N°     |
| N° working units | N°     |
| Carbon typology  |        |
| Volume           | m3     |
| Specific surface | m2     |
| Carbon density   | kg/m3  |
| Contact time     | minuti |
| Velocity         | l/m2/h |

| <b>CLARI-FLOCCULATION</b> | UdM    |
|---------------------------|--------|
| N° units                  | N°     |
| N° working units          | N°     |
| Volume                    | m3     |
| Surface                   | m2     |
| Dosage PAC                | gPAC/h |
| Contact time              | minuti |

| ULTRAFILTRATION        | UdM    |
|------------------------|--------|
| N° units               | N°     |
| N° working units       | N°     |
| Porosità               | micron |
| Materiale membrane     | -      |
| Surface                | m2     |
| Flow J                 | l/m2/h |
| Operating pressure     | bar    |
| Contact time           | minuti |
| Flux of backwash water | I/S    |
| Backwash frequency     |        |
| Frequency of chemical  |        |
| backwash               |        |





#### **DWTPs – treatment units**

| OZONATION        | UdM              |
|------------------|------------------|
| N° units         | N°               |
| N° working units | N°               |
| Volume           | m3               |
| Dosage           | Nm3/h air        |
|                  | yield gO3/m3 air |
|                  | gO3/h            |
| Contact time     | sec              |
| Residual Ozone   | mgO3/L           |

| CHLORINATION | UdM    |
|--------------|--------|
| Contact time | minuti |
| Dosage       | g Cl/h |
| Cl residual  | mg/l   |

| CHEMICAL     | UdM |
|--------------|-----|
| DISINFECTION |     |
| Reagent      | -   |
| Purity       | %   |
| Contact time | Min |
| Dosage       | g/h |

| STORAGE          | UdM |
|------------------|-----|
| N° units         | N°  |
| N° working units | N°  |
| Volume           | M3  |
| Storage time     | h   |

| EFFLUENT     | UdM       |
|--------------|-----------|
| рН           | -         |
| Conductivity | microS/cm |
| Cl residual  | mg/L      |
| Turbidity    | mg/L      |
| Others       |           |





### **WWTPs**





### Sampling points – WWTPs



BE CAREFUL TO THE EVENTUAL POINTS OF INTERNAL PRODUCTION

- INFLUENTIAL CHARACTERIZATION
- PRE-TREATMENTS
  - Screening
  - Sand and oil removal
- PRIMARY TREATMENTS
  - Primary sedimentation
  - Primary sludge
  - Chemical sludge
- SECONDARY TREATMENTS
  - Biological treatment
  - secondary sedimentation
  - Phosphorus removal
  - Organic sludge
  - Chemical sludge
- TERTIARY TREATMENTS
  - Filtration
  - UV disinfection
  - Chemical disinfection
- EFFLUENT CHARACTERIZATION





#### WWTPs – treatment units

| INFLUENT          | UdM       |
|-------------------|-----------|
| Flowrate          | m3/d      |
| рН                | -         |
| Temperature       | °C        |
| Conductivity      | microS/cm |
| TSS               | mg/l      |
| COD               | mg/l      |
| BOD <sub>5</sub>  | mg/l      |
| Ntot              | mg/l      |
| NH4               | mg/l      |
| TKN               | mg/l      |
| Ptot              | mg/l      |
| P-PO <sub>4</sub> | mg/l      |

| SCREENING        | UdM      |
|------------------|----------|
| N° units         | N°       |
| N° working units | N°       |
| Area             | m2       |
| Mesh size        | mm       |
| Produced waste   | ton/anno |

| SAND REMOVAL     | UdM      |
|------------------|----------|
| N° units         | N°       |
| N° working units | N°       |
| Area             | m2       |
| Volume           | m3       |
| HRT              | min      |
| Produced sand    | ton/anno |

#### PRIMARY SEDIMENTATION UdM

| N° units                | N°       |
|-------------------------|----------|
| N° working units        | N°       |
| Area                    | m2       |
| Volume                  | m3       |
| Produced sludge         | kg/d     |
| Sludge characterisation | TS%      |
|                         | TVS/TS   |
|                         | N%TS     |
|                         | P%TS     |
| Characterisation of the | mgTSS/I  |
| effluent from primary   | mgCOD/I  |
| seamentation            | mgBOD/I  |
|                         | mgNtot/l |
|                         | mgNH4/I  |
|                         | mgPtot/I |
|                         |          |





WWTPs – treatment units

| <b>BIOLOGIC TREATMENT</b> | UdM  |
|---------------------------|------|
| Type of process           |      |
| N° units                  | N°   |
| N° working units          | N°   |
| Recycle flowrate          | m3/d |
| Aerated mix flowrate      | m3/d |
| Oxidation volume          | m3   |
| Denitrification volume    | m3   |
| MLSS                      | g/l  |
| MLVSS                     | g/l  |
| Air flowrate              | m3/h |

| SECONDARY SEDIMENTATION               | UdM      |
|---------------------------------------|----------|
| N° units                              | N°       |
| N° working units                      | N°       |
| Area                                  | m2       |
| Volume                                | m3       |
| Extracted secondary sludge (supero)   | kg/d     |
| Sludge characterisation               | TS%      |
|                                       | TVS/TS   |
|                                       | N%TS     |
|                                       | P%TS     |
| Characterisation of the effluent from | mgTSS/I  |
| secondary treatment                   | mgCOD/l  |
|                                       | mgBOD/l  |
|                                       | mgNtot/l |
|                                       | mgNH4/l  |
|                                       | mgPtot/l |
|                                       |          |
| SRT                                   | d        |





#### WWTPs - treatment units

| PHYSICAL TERTIARY       | UdM  |
|-------------------------|------|
| TREATMENTS              |      |
| Type of treatment (e.g. |      |
| granular, cloth,        |      |
| membrane filtration,    |      |
| etc.)                   |      |
| N° units                | N°   |
| N° working units        | N°   |
| Filtration media        | -    |
| (sand/carbon)           |      |
| Porosity                | mm   |
| Area                    | m2   |
| Backwash                | m3/h |

| CHEMICAL OR UV | UdM |
|----------------|-----|
| DISINFECTION   |     |
| Reagent        | -   |
| Purity         | %   |
| Dose           |     |
| Contact time   | min |

| EFFLUENT          | UdM  |
|-------------------|------|
| рН                | -    |
| TSS               | mg/l |
| COD               | mg/l |
| BOD <sub>5</sub>  | mg/l |
| Ntot              | mg/l |
| NH4               | mg/l |
| N-NOx             | mg/l |
| TKN               | mg/l |
| Ptot              | mg/l |
| P-PO <sub>4</sub> | mg/l |
| Others            |      |



### **Microplastics in the Integrated Water Service**

Tracing the sources and transport of microplastics in the environment is a complex task. Little is known about the processes governing the transport of microplastics through freshwater environments and their distribution. However, key factors influencing their transport and distribution include the intrinsic properties of microplastics, i.e. their density, size and shape.

To date, there are insufficient methods to trace the sources and transport of microplastics in the environment and there are no standard sampling methodologies.

The presence of microplastics in freshwater has been investigated in the literature, with studies reporting particle counts ranging from approximately 0 to 10^3 particles/L (Koelmans et al., 2019). The results are notably influenced not only by the environmental and site-specific conditions, but also by the sampling technique and the method of analysis.

**PLEASE NOTE:** The interpretation and comparison of the results of the literature studies must be carried out with great care, since the studies often use **different methods**, sampling different volumes, using filters of different sizes and using different enumeration techniques. This means that some studies do not detect the smallest particle sizes or even include some non-plastic materials such as plastics.

| Location |  | Results reported<br>(particles/L)  | Sieve size<br>(µm) | Study                                      |
|----------|--|--|--------------------|--|
|          | Groundwater, Germany   | Average: <sup>b</sup> $0.7 \times 10^{-3}$<br>Range: <sup>b</sup> $0 - 7 \times 10^{-3}$ | 3                  | Mintenig et al., 2019                      |
|          | Three Gorges Reservoir,<br>China                             | Average: 4.7<br>Range: 1.6—12.6  | 48                 | Di and Wang, 2018                          |
|          | Dongting Lake and Hong<br>Lake, China                        | Averages: 1.2 and 2.3<br>Ranges: 0.9–2.8 and<br>1.3–4.7                                  | 50                 | Wang et al., 2018                          |
|          | Wuhan, China   | Range: 1.6—8.9   | 50                 | Wang et al., 2017                          |
|          | Rhine river, Switzerland,<br>France, Germany,<br>Netherlands | Average: 0.0056  | 300                | Mani et al., 2015                          |
|          | Western Lake Superior,<br>USA                                | Average: 0.00026   | 333                | Hendrickson, Minor,<br>and Schreiner, 2018 |



## Sampling methodologies for wastewater



### Literature research

| SOURCE                                   | PROCESS   | VOLUME  | METHOD   | REFERENCE   |
|--|---|---|--|---|
| Municipal<br>wastewater                  | Grit-grease<br>Primary clarifier  | 60.1 L<br>59.3 L  | <ul> <li><u>Grabbed</u> in glass bottles, both in the morning and in the afternoon</li> <li>Filter through diameter 110 mm, pore size 0.45 mm</li> </ul>   | (Bayo, Olmos, & López-Castellanos,<br>2020)           |
|  | Activated sludge  | 103.4 L<br>143 I  |  |   |
| Municipal<br>wastewater                  | Influent (after 6mm screen)<br>After the primary clarification<br>After the disinfection                        | 4-30 L  | Collected with a 10-L stainless steel bucket attached to a metal wire and poured to a cascade of two test sieves<br>with mesh sizes of <mark>250 μm</mark> and 5.0 mm  | (Lares, Ncibi, Sillanpää, & Sillanpää,<br>2018)       |
| Municipal<br>wastewater                  | Influent<br>After the settler<br>Effluent   | 30 L  | <ul> <li>In the morning (9-11 am)</li> <li>Filtered in loco with a suite of steel sieves with a mesh of 5 mm, 2 mm and 63 μm.</li> </ul>   | (Magni et al., 2019)                                  |
| Municipal<br>wastewater                  | Effluent  | <u>500-21000 L</u>  | <ul> <li>Filtered through a set of Tyler sieves at a flow rate of 12-18 L per minute for a period of 2-24 h</li> <li>A <u>355 μm-mesh sieve</u> was stacked atop a <u>125 μm mesh sieve</u> for the shorter (2 h) sampling times, while the 0.355 mm-mesh sieve was used in isolation for the longer sampling periods</li> </ul> | (Mason et al., 2016)                                  |
| Municipal<br>wastewater                  | Influent<br>Pretreated influent<br>Primary effluent<br>Secondary effluent<br>Final effluent                     | 1-2 L<br>1-6 L<br>10-20 L<br>10-20 L<br>34-38 L                           | Grab samples were collected in plastic containers     Grab samples were collected in plastic containers  | (Michielssen, Michielssen, Ni, &                      |
| Municipal<br>(+industrial)<br>wastewater | Effluent from different configurations of WWTPs   | <u>390-1000 L</u>   | Custom made mobile pumping device with a filter housing containing a 10 mm stainless ste <b>Varia fro</b>  | n 1-2 L to >  |
| Municipal<br>wastewater                  | Influent<br>Grit&grease effluent<br>Primary effluent<br>Final effluent  | 30-50 L   | <ul> <li>n=303</li> <li>First passed through steel sieves (65 μm), then vacuum filtered through Whatman No. 1 qualit filter paper, with a pore size of <u>11 μm</u>.</li> <li>Varia</li> </ul>   | 1000 L  |
| Municipal<br>wastewater                  | Disc filter<br>Rapid sand filter<br>Dissolved ait floatation<br>MBR<br>CAS                                      | Different volumes<br>for different filter<br>size and unit (2-<br>1,000L) | <ul> <li>Custom made filtering device with in-situ fractionation</li> <li>The mesh-sizes of the filters were 300, 100 and 20 μm, giving particle size fractions of &gt;300 μn</li> <li>20-100 μm</li> <li>Additional composite samples for 24 h</li> </ul>   | from 11 μm & Setälä,<br>350 μm                        |
| Municipal<br>wastewater                  | Post primary treatment<br>Post primary and secondary<br>treatment<br>Post primary, tertiary and RO<br>treatment | 200   | <ul> <li>Each sampling event took approximately 1 h with a maximum flow rate of 10 L/min</li> <li>The sampling device consists of four removable stainless-steel mesh screens (plain Dutch weave) with sizes of 500, 190, 100 and 25 μm with a diameter of 12 cm.</li> </ul>   | (Ziajahromi, Neale, Rintoul, &<br>Leusch, 2017)<br>3L |
|  |   |   |  |   |

## Sampling methodologies for wastewater

and 1 liter through the 20 µm

filter.

### Literature research



| SOURCE                                       | PROCESS   | VOLUME   | METHOD   | REFERENCE   |  |
|--|---|--|--|---|--|
| Raw wastewater<br>Treated                    |   | <u>11</u>  | Retsch AS 200 <u>vibratory sieve shaker</u> through 2 mm, 1mm and <u>500 µm sieve meshes</u> . Sodium dodecyl sulfate (SDS) as a surfactant (Simon, van Alst, & Vollertsen, added to a final concentration of 0.15 g/L before sieving to detach adhered MP particles from the larger solids. 200mL of the pre-<br>sieved wastewater was incubated with cellulase enzyme (Aspergillus sp., Sigma-Aldrich, CAS no. 9012-54-8) for 48 h at 40 °C to degrade cellulose fibers deriving mainly from toilet paper. Organic material was oxidized with hydrogen peroxide where iron (II) was added to catalyze the reaction (Fenton reaction). Peroxide was added to a final concentration of 250 g/L and iron (II) sulfate to 2.5 g/L. The pH of the mixture was adjusted to approximately 3 with sodium hydroxide. The oxidized sample was wet-sieved (demineralized water with 0.15 g/L SDS) into two size fractions through an 80 µm sieve mesh. The effluent containing particles <80 µm was collected into a glass beaker. Particles >80 µm were removed from the sieve mesh into filtered demineralized water containing 0.15 g/L SDS by treatment in an Elma S50R ultrasonic bath. Particles from this liquid and the collected effluent were gathered on separate 10 µm steel meshes. Particles were removed from the filters into 25mL HPLC grade ethanol by ultrasonic treatment. The resulting particle-ethanol suspensions of the two size fractions were transferred into glass vials where their final volume was set to 5mL by evaporation with nitrogen gas. The chemical composition of the extracted particles was determined with an FPA-based FT-IR imaging technique. |   |  |
| wastewater                                   |   |  | for 48 h at 40 °C with cellulase enzyme. Samp adjusted to 3 by sodium hydroxide. Size fractior   | es oxidized in 180 g/L hydrogen peroxide catalyzed by 1.8 g/L iron (II) sulfate and pH nation by wet-sieving and transferring the particle-ethanol suspension into glass vials.   |  |
| Municipal                                    | Screening   | 30-50 L  | Steel buckets and sieve  | (Murphy et al., 2016)   |  |
| wastewater<br>effluent                       | Grit and grease removal<br>Settling tank<br>Aeration basin<br>Clarifier                                       |  |  | <ul> <li>Grab samples</li> <li>Variable volumes from 1-2   to &gt; 1000  </li> </ul>  |  |
| Municipal<br>wastewater<br>effluent          |   |  | Fractionated filtering   | <ul> <li>Variable filtration mesh from 11 μm to 350 μm</li> </ul>   |  |
| Municipal<br>wastewater<br>effluent          |   |  | Sieving and filtering method • Methodologies of sample pre-processing "customised"   |   |  |
| Municipal<br>wastewater<br>effluent          |   |  | Custom made pump +stainless<br>steel cartridge filter  | Examples of in-situ filtration with ad-hoc  |  |
| Municipal<br>wastewater<br>effluent          |   | 2 L  | Effluent: grab samples   | instrumentation   |  |
| Wastewater, 24-<br>hour composite<br>samples | Influent wastewater, after<br>mechanical purification and<br>after the process from<br>discharged wastewater. | From 100 ml (incoming<br>wastewater) to 8 litres (purified<br>wastewater). 50 liters of purified<br>wastewater were filtered<br>through 300 and 100 µm filters | Filter device consists of three transparent plast<br>another. Round (diameter 80 mm) filters are<br>together with rubber o-rings. Round filters are<br>on the top of the device, 100 μm filter in the m<br>sampling.   | ic tubes (diameter 60 mm) and screw-on plastic connectors attaching the tubes to one (Talvitie et al., 2017)<br>placed into the filter device between the connectors and tubes are screwed tightly<br>cut from different mesh size plankton nets. The largest mesh size filter 300 μm is placed<br>iddle and 20 μm filter at the bottom. All equipment has to be rinsed thoroughly prior to |  |



eu

## Sampling methodologies for sludge



Literature research

| <u>SOURCE</u>    | PROCESS   | VOLUME      | <u>METHOD</u>  | <b>REFERENCE</b>           |
|------------------|---|-------------|--|----------------------------|
| Sewage<br>sludge | Anaerobic<br>digestion                                      | <u>2 kg</u> | <ul> <li>Composite over one day in<br/>each month</li> <li>Suspended, pre-washed and<br/>then filtered through 5 mm<br/>stainless steel</li> </ul> | (Xu et al., 2020)          |
| Sewage<br>sludge | Activated<br>sludge<br>MBR sludge<br>Anaerobic<br>digestion | 150-200 mL  | Poured in glass flasks with metal<br>funnel, kept in dark  | (Lares et al.,<br>2018)    |
| Sewage<br>sludge | Drained   | 500 g       | Taken by shovel, stored in dark  | (Mintenig et al.,<br>2017) |
| Sewage<br>sludge | Anaerobic<br>digestion                                      | <u>30 g</u> | <ul><li>Three replicates</li><li>Pellets of TD sludge were</li></ul>   | (Mahon et al.,<br>2017)    |
|                  | Thermal<br>drying   |             | placed in water for 1 week<br>to induce softening,<br>transferred to a water bath  |                            |
|                  | Lime<br>stabilization                                       |             | (30 °C) for 24 h, and placed in an shaker for 12 h.  |                            |

• Instantaneous samples

• Variable quantities from 30 g to 2 kg

• Discontinuous information on filtering mode and pre-processing



#### Sampling Volumes

| Author                                | Treatment unit | Volume sampled                         | n°MPs/L                                |
|---------------------------------------|----------------|--|--|
| (Mang at al. 2020)                    | Influent       | 1L x 3 samples                         | 6614 ± 1132                            |
| (Wang et al., 2020)                   | Effluent       | 1L x 3 samples                         | 930 ± 71                               |
|                                       | Influent       | 2L x 3 times/day                       | 23±2                                   |
| (Divekenský et al. 2020)              | Effluent       | 2L x 3 times/day                       | 14±1                                   |
| (PIVOROIISKY EL dl., 2020)            | Influent       | 2L x 3 times/day                       | 1296±35                                |
|                                       | Effluent       | 2L x 3 times/day                       | 151 ± 4                                |
|                                       | Influent       | 1L x 3 samples/day x 3 times x 3 days  | 1473 ± 34                              |
|                                       | Effluent       | 1L x 3 samples/ day x 3 times x 3 days | 443 ± 10                               |
| (Divekensky et al. 2018)              | Influent       | 1L x 3 samples/ day x 3 times x 3 days | 1812 ± 35                              |
| (PIVOROTISKY Et al., 2018)            | Effluent       | 1L x 3 samples/ day x 3 times x 3 days | 338 ± 76                               |
|                                       | Influent       | 1L x 3 samples/day x 3 times x 3 days  | 3605 ± 497                             |
|                                       | Effluent       | 1L x 3 samples/day x 3 times x 3 days  | 628 ± 28                               |
|                                       | Influent       | 300-1000 L                             | 0.003                                  |
|                                       | Distribution   | 1200-2500 L                            | <0.001                                 |
|                                       | Influent       | 300-1000 L                             | 0.007                                  |
| (Mintonia et al. 2010)                | Effluent       | 1200-2500 L                            | <0.001                                 |
| (Millitenig et al., 2019)             | Distribution   | 1200-2500 L                            | 0.003                                  |
|                                       | Influent       | 300-1000 L                             | 0.001                                  |
|                                       | Effluent       | 1200-2500 L                            | 0.002                                  |
|                                       | Influent       | 300-1000 L                             | 0.001                                  |
|                                       | Influent       | 10 L *2 duplicates                     | 42 ± 18 particles/L                    |
| (Cherniak et al., 2022)               | Effluent       | 10 L *2 duplicates                     | 20 ± 8 particles/L                     |
|                                       | Distribution   | 10 L *2 duplicates                     | 20.5 ± 7.6 particles/L                 |
| (lung of al. 2022)                    | Influent       | 10,100 L *12 monthly complex           | 2.2 ± 1.3                              |
| (Julig et al., 2022)                  | Effluent       | 10-100 L 12 monthly samples            | 0.02 ± 0.02                            |
| Shi at al 2021                        | Influent       | nd                                     | 6614 ± 1132                            |
| 5111 et al., 2021                     | Effluent       | 11.u.                                  | 930 ± 71                               |
| $(V_{\text{upp}} \text{ at al} 2022)$ | Influent       | nd                                     | 17.88                                  |
| (Tuall et al., 2022)                  | Effluent       | n.u.                                   | 2.75                                   |
| (Johnson at al. 2020)                 | Influent       | nd                                     | 21.09 ± 20.49                          |
|                                       | Effluent       | n.u.                                   | 0.001-0.024                            |
| (loclic ot al 2017)                   | Influent       | n.d.                                   | 1385 (dry season); 1796.6 (wet season) |
|                                       | Effluent       | n.d.                                   | 448.7 (dry season); 769.4 (wet season) |

- Information not homogeneous and not always available on the volumes actually sampled
- Extreme variability from 1L to
  - 2500L



#### **Dimension of seaving mesh**

Comparison of MPs numbers in the drinking water treatment process in this study and previous studies. (Unit: particle/L)

| Research             |                    | Raw             |                               |                  |                            | Samples                          |   |                 |  |                               |   |       |
|----------------------|--------------------|-----------------|-------------------------------|------------------|----------------------------|----------------------------------|---|-----------------|--|-------------------------------|---|-------|
|                      |                    | Raw<br>water    | Pre-ozonation                 | Sedime           | entation                   | Sand 1                           | filtration  | Treated water   | Methodology  | Limit of<br>size<br>detection |   |       |
| This study           |                    | 2.2 ±<br>1.3    | 2.3 ± 0.8                     | 1.4 ±            | : 0.15                     | 0.2 ± 0.15                       |   | $0.02 \pm 0.02$ | Automated via<br>micro-FTIR imaging,<br>(Hyperion 3000, Bruker)          | > 10 µm                       | • | Diffe |
| Wa                   |                    | Raw<br>water    | Coagulation/<br>sedimentation | Deep bed         | filtration                 | tion Ozonation + *GAC filtration |   | Treated water   |  |                               |   |       |
| ng,<br>202<br>0      | Downst<br>ream     | 1296            | 497                           | 24               | 243                        |                                  | 149   |                 | Quantification: SEM<br>Qualification: Raman<br>(ThermoFisher)            | >1 µm                         |   |       |
|                      | Upstrea<br>m       | 23              |                               |                  |                            |                                  |   |                 |  |                               |   |       |
| Les<br>lie           |                    | Raw<br>water    | Screen-outlet                 | Clarifie         | er-outlet                  | Filtration                       |   | Treated water   |  |                               |   |       |
| et<br>al.,           | Dry<br>season      | 1385            | 1298.5                        | 82               | 3.2                        | 5                                | 536.7     448.7     >300 μm: ATR-FTIR<br>hand picking<br>< 300 μm: Confocal |                 | >300 µm: ATR-FTIR<br>hand picking<br>< 300 µm: Confocal                  | 6.5–500 μm                    |   |       |
| 7                    | Rainy<br>season    | 1796.6          | 1669.2                        | 12               | 2.9                        | 844.6                            |   | 769.4           | Raman + Nile red   |                               |   |       |
| Sh                   | iet al             | Raw<br>water    | Sedimentation                 | Sand fi          | ltration                   | Ozonation                        | GAC<br>filtration   | Treated water   | Quantification: 25% of   |                               |   |       |
| 2021                 |                    | 6614 ±<br>1132  | ~3000                         | ~2               | 000                        | ~2000                            | <1000   | 930 ± 71        | Qualification: micro-<br>Raman (ThermoFisher)                            | 1–100 μm                      |   |       |
| Yuan et al.,<br>2022 |                    | Raw<br>water    | Pre-disinfection              | Floccula<br>tion | Pulse<br>clarificat<br>ion | Sand filtration                  |   | Treated water   | Quantification:<br>Fluorescence microscope<br>+ Nile red staining (Zeiss | 1–100 µm                      |   |       |
|                      |                    | 17.88           | 17.53                         | 17.11            | 6.99                       | 11.17                            |   | 2.75            | AXIO)<br>Qualification: ATR-FTIR   |                               |   |       |
| Joh<br>al.           | nson et<br>., 2020 | 21.09±<br>20.49 | -                             | -                | -                          |                                  | -   | 0.001-0.024     | Automated via micro-<br>FTIR imaging                                     | >25 µm                        |   |       |

Different sizes



\*Granular activated carbon. J.-W. Jung et al. 2022

#### Dimension of seaving mesh and separation method



| Reference           | Sampling method   | Downstream sample treatment   |
|---------------------|---|---|
| Z. Wang, 2019       | Water samples were collected in <b>1L</b><br>brown glass bottles (pre-cleaned)<br>from the raw water and effluents<br>from each treatment process.  | Filtration through a series of <b>5 μm</b> membrane filters<br>(PTFE) followed by a pore size of 0.22 μm.   |
| M. Pivokonský; 2020 | Two liters of water was filled into<br>borosilicate glass bottles (pre-<br>cleaned). The samples were then<br>stored in the dark at 4 ° C. Any<br>contact of the samples with plastic<br>materials was avoided during<br>sampling campaign.   |   |
| M. Pivokonsky, 2018 | A raw water sample and a treated<br>water sample ( <b>1L of both</b> ) were taker<br>in autoclavable (pre-cleaned)<br>borosilicate glass bottles. The<br>samples were stored at 4 ° C before<br>analysis. Any contact of the samples<br>with plastic materials was avoided<br>during sampling campaign. | The pretreated samples were passed through a series of<br><b>5 μm</b> polytetrafluoroethylene (PTFE) membrane filters<br>and subsequently <b>0.2 μm</b> pore size.  |
| Mintenig S.M., 2018 |   | 3μm cartridge filters were used. The residual raw water<br>and drinking water were removed from the filter units<br>using filtered compressed air (0.2 μm). The retentate was<br>collected on 3 μm (47 mm diameter) stainless steel filters |

Expensive and complex separation and characterization methods (even functionally at the minimum level of size detectability)



#### **Characterisation methods**

Comparison of MPs numbers in the drinking water treatment process in this study and previous studies. (Unit: particle/L)

| Research             |                   | Raw<br>water    | Pre-ozonation                 | Sedime           | ntation                    | Sand f     | iltration                   | Treated water   | Samples<br>Methodology   | Limit of<br>size<br>detection |
|----------------------|-------------------|-----------------|-------------------------------|------------------|----------------------------|------------|-----------------------------|-----------------|--|-------------------------------|
| This study           |                   | 2.2 ±<br>1.3    | 2.3 ± 0.8                     | 1.4 ±            | 0.15                       | 0.2 ± 0.15 |                             | $0.02 \pm 0.02$ | Automated via<br>micro-FTIR imaging,<br>(Hyperion 3000, Bruker)          | > 10 µm                       |
| Wa                   |                   | Raw<br>water    | Coagulation/<br>sedimentation | Deep bed         | Deep bed filtration C      |            | Ozonation + *GAC filtration |                 |  |                               |
| ng,<br>202<br>0      | Downst<br>ream    | 1296            | 497                           | 243              |                            | 1          | 149                         | 151             | Quantification: SEM<br>Qualification: Raman<br>(ThermoFisher)            | >1 µm                         |
|                      | Upstrea<br>m      | 23              |                               |                  |                            |            |                             | 14              | (11011011010)  |                               |
| Les<br>lie           |                   | Raw<br>water    | Screen-outlet                 | Clarifier-outlet |                            | Filt       | ration                      | Treated water   |  |                               |
| et<br>al.,<br>201    | Dry<br>season     | 1385            | 1298.5                        | 82               | 823.2                      |            | 536.7                       |                 | >300 µm: ATR-FTIR<br>hand picking<br>< 300 µm: Confocal                  | 6.5–500 μm                    |
| 7                    | Rainy<br>season   | 1796.6          | 1669.2                        | 12               | 2.9                        | 84         | 44.6                        | 769.4           | Raman + Nile red   |                               |
| Sh                   | i at al           | Raw<br>water    | Sedimentation                 | Sand fi          | ltration                   | Ozonation  | GAC<br>filtration           | Treated water   | Quantification: 25% of   |                               |
| 2021                 |                   | 6614 ±<br>1132  | ~3000                         | ~20              | 000                        | ~2000      | <1000                       | 930 ± 71        | Qualification: micro-<br>Raman (ThermoFisher)                            | 1–100 μm                      |
| Yuan et al.,<br>2022 |                   | Raw<br>water    | Pre-disinfection              | Floccula<br>tion | Pulse<br>clarificat<br>ion | Sand f     | liltration                  | Treated water   | Quantification:<br>Fluorescence microscope<br>+ Nile red staining (Zeiss | 1–100 µm                      |
|                      |                   | 17.88           | 17.53                         | 17.11            | 6.99                       | 1          | 1.17                        | 2.75            | AXIO)<br>Qualification: ATR-FTIR   |                               |
| Joh<br>al.           | nson et<br>, 2020 | 21.09±<br>20.49 | -                             | -                | -                          |            | -                           | 0.001-0.024     | Automated via micro-<br>FTIR imaging                                     | >25 µm                        |

Different characterisation methods



\*Granular activated carbon. J.-W. Jung et al. 2022

# Sampling methodologies for drinking water



#### Literature research

| SAMPLING POINTS   | VOLUME             | TYPE of SAMPLE        | METHODS OF SAMPLING AND PROCESSING  | FREQUENCY or n°  | <u>Ref.</u>                                |
|---|--------------------|-----------------------|---|--|--|
|   |                    |                       |   | of samples   |  |
| Raw and treated<br>drinking water (after<br>each process) | <u>11</u>          | <u>Grab samples</u>   | Pre-processing: Digestion with 30% hydrogen peroxide (H <sub>2</sub> O2) for 24 h. Filtration through a series of 5 μm (PTFE) membrane filters followed by a 0.22 μm pore sizes. The purpose of this two-filtration was to descend mesh size to pass the entire sample through the filter without clogging. These filters were used for scanning electron microscope (SEM) analysis of retained particles. For each sample, a volume of 250 ml was separately filtered for quantitative and qualitative analysis of particles. The filters after drying in an oven at 30°C for 30 min were stored in covered glass petri dishes for subsequent analysis. DXR2 micro-Raman imaging microscope system (Thermo Fisher Scientific, USA) was employed (532 nm laser, laser spot size around 0.5 μm, Raman shift 50–3550 cm_1, spectral resolution of 5 cm <sup>-1</sup> ) for qualitative analysis of particles.   | 3 times /winter  | (Wang, Lin, & Chen,<br>2020)               |
| Raw and treated<br>drinking water                         | 27L each<br>sample | Average daily samples | Pre-processing: Wet peroxide oxidation was conducted to remove organic material, Filtration through a series of 5 $\mu$ m (PTFE) membrane filters followed by a 0.22 $\mu$ m pore sizes. The purpose of this two-filtration was to descend mesh size to pass the entire sample through the filter without clogging. These filters were used for scanning electron microscope (SEM) analysis of retained particles. For each sample, a volume of 250 ml was separately filtered for quantitative and qualitative analysis of particles. The filters after drying in an oven at 30°C for 30 min were stored in covered glass petri dishes for subsequent analysis   | 3 times within a<br>24-hour period<br>(every 8 h) and<br>repeated three<br>times in winter<br>period | (Pivokonsky et al., 2018)                  |
| Raw and treated drinking water                            | 9-27 L             |                       | Scanning electron microscopy analysis for particle counts; both micro-Raman spectroscopy and µ-FT-IR were used for identification of particles with size of 1e10mm and>10mm   |  | (Eerkes-Medrano,<br>Leslie, & Quinn, 2019) |
| Raw and treated<br>drinking water                         | 1000 L             | Grab samples          | Samples directly sieved, tap water require no digestion.  |  | (Koelmans et al., 2019)                    |
| Raw and treated<br>drinking water                         | <u>300-2500 L</u>  | <u>Grab samples</u>   | Sampling: <u>3µm stainless steel cartridge filters</u> 4 7/8", Wolftechnik, Germany<br>Pre-processing: Residual raw water and drinking water was removed from t e filter units by using filtered (0.2 µm) compressed air. Then, the units<br>were filled again with diluted hydrochloric acid (Carl Roth GmbH & Co. KG, Germany, 0.2 µm filtered, pH=2) to dissolve calcium carbonate and iron<br>precipitates. After 24 h the filter units were emptied, the cartridge filters removed from the units and rinsed with Milli-Q and ethanol (30%, Carl<br>Roth GmbH & Co. KG, Germany, filtered over 0.2 µm). The retentate was collected on 3 µm stainless steel filters (47mm in diameter) that were<br>subsequently transferred into glass bottles and covered with 30 mL hydrogen peroxide (35%, Carl Roth GmbH & Co. KG, Germany). The bottles<br>were closed using aluminium foil and incubated for 24 h at 40 °C. Finally, each sample was enriched onto a 0.2 µm aluminium oxide filter (Anodisc<br>25 mm, Whatman, U.K.) by using an in-house fabricated filter-funnel with an inner diameter of 11 mm. The filters were dried at 40 °C in half<br>closed glass petri dishes for subsequent analysis. | 24 samples   | (Mintenig et al., 2017)                    |

- Grab samples
- Variables volumes from 1 L to 2500 L
  - Lower filtration mesh (3μm)
- Different sampling and pre-processing methods



### Sampling methodologies for combined sewer overflows Literature research



| SAMPLING POINTS   | VOLUME  | TYPE OF SAMPLE     | METHODS OF SAMPLING AND DETENCTION   | FREQUENCY or num <sup>®</sup> of samples | Ref.  |
|-------------------|---|--------------------|--|--|---|
| stormwater runoff | <u>1L</u> per each sample   | <u>Grab sample</u> | Iron bucket that was rinsed three times with the runoff before taking the samples. The samples were a combination of sediment and water due to the strength of the runoff streams in the streets or storm drains. These samples were collected at the beginning of the rain event and at approx 10 min and 30 min after the first samples were collected.  | 94 samples                               | (Piñon-Colin, Rodriguez-<br>Jimenez, Rogel-<br>Hernandez, Alvarez-<br>Andrade, & Wakida,<br>2020) |
| Stormwater pond   | up to several thousand<br>liters for the bigger mesh<br>size, <u>10-70 liters with a</u><br><u>mesh size of 20 μm</u> . | <u>Grab sample</u> | Sampling method consisted of a gasoline pump, hoses, filter holder and filter. Two types of filters were tested; plankton net (mesh size 300 $\mu$ m) manually cut into circles and prefabricated polycarbonate filters (mesh size 10 $\mu$ m). A mechanical volumeter was attached to the outlet hose in order to measure the volume of water filtered. The filter holder consisted of stainless-steel pipes, gaskets, and a clamp. The inner diameter of the stainless-steel pipes was in this case 2 inches. The inlet and outlet hoses chosen had inner diameters of 1,5 and 1 inch. The inlet hose was of sturdier material, not to deflate due to the suction pressure of the pump. Polyester plankton nets (Sefar Petex), where cut into circles to fit the filter holder. Two mesh sizes were used. The mesh size of 300 $\mu$ m was used to allow for comparison of results with the majority of studies conducted thus far. Quantification performed manually by counting MPs using a microscope. Some of the detected MPs were analyzed with FTIR spectroscopy. |  | Coalition Clean Baltic,<br>2017   |

- Existing literature reports the microplastics determined in rainwater.
- The presence and fate of microplastics in CSOs have not been fully investigated to date.
  - Samples were taken discontinuously, in variable volumes (1-70 L).



### **Related projects**



The topic of microplastics in water is of great interest at European level and a wide range of projects are developing innovative solutions for their management. The first step is often to choose a simple, inexpensive and exploitable sampling method, which allows comparison and sharing of results. Some activities foresee the development of a sampling method: projects, such as CLAIM, TextileMission, EMISTOP, ENSURE and RUSEKU; studies, such as those for the HELCOM BASE Project and the Norwegian Institute for Water Research; and guidelines, such as those of CCB.

| Project-Activity Title  | Description  | Links  | Interest for Microplastics  |
|---|--|--|---|
| CLAIM - Cleaning Litter by<br>leveloping and Applying<br>nnovative Methods in european<br>eas   | Development of innovative cleaning technologies and approaches,<br>targeting the prevention and in situ management of micro and<br>macroplastics in the Mediterranean and Baltic Sea.  | https://www.claim-<br>h2020project.eu/   | Pre-filtering system to retain larger plastics, while simultaneously taking two samplers; Photocatalytic nanocoating device fo cleaning microplastics in wastewater treatment plants to obtain the degradation of low-density polyethylene (LDPE) microplastic by visible light-induced heterogeneous photocatalysis activated by Zinc oxide nanorods.  |
| miStop - Identification of<br>ndustrial Plastic Emissions by<br>Aeans of Innovative Detection<br>Aethods and Technology<br>Development to Prevent the<br>nput into the Environment via<br>he Wastewater Pathway | Detect emissions of plastics into wastewater from relevant industries.<br>Select wastewater treatment technologies that will optimise reduction of<br>emissions of plastics for the respective value chain. The project will<br>evaluate existing technologies for separating particles, investigate<br>deposition rates, and develop approaches for technical optimisation.<br>Optimisation also includes technological developments such as<br>flocculants. Researchers will conduct tests in both laboratory and pilot<br>project settings – selected large scale technical wastewater treatment<br>plants will be investigated at industrial facilities. | http://www.emistop.de/pa<br>ge8.html<br>https://bmbf-<br>plastik.de/en/joint-<br>project/emistop | Standardisation of sampling and sample preparation for industrial wastewater (in alignment with Plastik-Net and other join research projects). Quantitative and qualitative measurement of plastic concentrations in industrial wastewater using Ramar spectroscopy and dynamic differential calorimetry. Extended data collection at the sampled industrial wastewater treatment plants for the derivation of the plastic loads and testing of a correlation between the measurement results and routine wate chemical analyses. Development of magnetic plastic particles in the micrometre range with the physical properties of relevant types of plastic. Development of a tracer test with magnetic plastic particles. Assessment of technologies for particle separation with regard to the retention of (micro-) plastic particles. Optimisation of particle separation technologies. Development of flocculants for the targeted improvement of the retention of individual types of plastics and their mixtures |
| TextileMission – Microplastics of<br>Textile Origin - A Holistic View:<br>Optimized Processes and<br>Materials, Material Flows and<br>Environmental Behavior.   | Synthetic fiber particles with a diameter of less than 5 millimetres are<br>only partially filtered out by modern wastewater treatment plants. The<br>partners of the joint resreach project TextileMission have taken on the<br>task of reducing this environmental impact.   | https://textilemission.bsi-<br>sport.de/en/  | WP 4 Polyester fibres: At the TU Dresden, analysis and sample preparation method is established to quantify microparticles from wastewater streams and fractionate them according to size. Investigation of the retention capacity of textile (fluorescence labelled) microparticles in the different stages of a laboratory wastewater treatment plant and identification of efficient retention possibilities. Material flows are analysed including a first estimate of the Germany-wide textile microplastic emission from wastewater into water bodies /soils. WP 5: Consideration of other environmental issues related to the project, stakeholder involvement and communication at the end of the project. Focus on the retention of textile microfibre particles through various purification stages in wastewater treatment plants.   |
| NSURE Development of New<br>Plastics for a Clean Environment<br>by Determining of Relevant Entry<br>Points.   | Holistic approach to reduce plastic in the environment as well as the related negative consequences, including improving methods of analyzing the environmental impact of plastics.  | https://www.ensure-<br>project.de<br>https://bmbf-<br>plastik.de/en/joint-<br>project/ensure     | In the "Traceability" module, undesirable plastic inputs in prioritised sectors (soils, wastewater treatment plants, composting plants and biogas plants) are detected and identified. In a first step, sampling strategies are developed so that in a second step representative investigations can be carried out to determine the current states of plastics in fermentation, compost and wastewater treatment plants.   |
| RUSEKU – Precise Detection of<br>Aicroplastics in Water   | Plastics in the Environment: Sources - Sinks – Solutions. Development of representative test methods that can accurately and quickly determine the microplastic content over various parts of the water cycle. The focus is on sampling methods in urban wastewater systems and watercourses.  | https://netzwerke.bam.de/i<br>useku<br>https://bmbf-<br>plastik.de/en/joint-<br>project/ruseku   | WP 2 Development of sampling methods; WP 3 Simulations software that simulates geometrically complex, application-oriented cases will be used to derive sampling strategy; WP 4 Sampling of real environmental compartment. quantification of the microplastic volume and transport in the real, urban wastewater system for the wastewater and precipitation water, domestic wastewater (partial flows grey and black water), industrial wastewater, and mixed wastewater. Further work will focus on sample preparation and preservation to evaluate the comparability of different sampling strategies. The investigations focus on the quantities and the importance of microplastic volumes in the individual entry points of the urban wastewater system into the water bodies.   |
| Aapping microplastics in sludge -<br>Research Report  | Characterization of microplastics in sewage sludge from Norwegian<br>domestic wastewater treatment plants applying different wastewater<br>and sludge treatment technologies.  | https://niva.brage.unit.no/r<br>iva-<br>xmlui/handle/11250/24935<br>27                           | Fenton's reagent was used to remove organic matter and density separations were employed to extract microplastics from sludge samples. Plastics were found in all ten sludge samples investigated from eight WWTPs. The overall average plastic abundance was 6077 particles kg-1 (d.w.) (1701 – 19 837) or 1 176 889 particles m-3 (470 270 – 3 394 274).  |
| IELCOM BASE Project -<br>mplementation of the Baltic Sea<br>Action Plan in Russia   | Preliminary study on synthetic microfibers and particles at a municipal wastewater treatment plant   | https://helcom.fi/helcom-<br>at-work/projects/base/  | Study the amount of microplastic litter arriving at the Central Wastewater Treatment Plant (WWTP) of St. Petersburg and the effect of the purification process. Helsinki Region Environmental Services Authority HSY developed a microplastic sampling method targeted at wastewaters.  |

### **Related projects**



Projects such as RUSEKU and SubµTrack and the HELCOM BASE study are focusing on MP detection in sewage treatment plants, and the REPLAWA, PLASTRAT, TextileMission and ENSURE projects are analyzing the treatment efficiency of different processes.

| Project-Activity Title   | Description   | Links  | Interest for Microplastics  |
|--|---|--|---|
| RUSEKU – Precise Detection<br>of Microplastics in Water  | Plastics in the Environment: Sources - Sinks – Solutions.<br>Development of representative test methods that can<br>accurately and quickly determine the microplastic content over<br>various parts of the water cycle. The focus is on sampling<br>methods in urban wastewater systems and watercourses.   | https://netzwerke.bam.de<br>/ruseku<br>https://bmbf-<br>plastik.de/en/joint-<br>project/ruseku                             | WP 2 Development of sampling methods; WP 3 Simulations software that simulates geometrically complex, application-oriented cases will be<br>used to derive sampling strategy; WP 4 Sampling of real environmental compartment. quantification of the microplastic volume and transport in<br>the real, urban wastewater system for the wastewater and precipitation water, domestic wastewater (partial flows grey and black water)<br>industrial wastewater, and mixed wastewater. Further work will focus on sample preparation and preservation to evaluate the comparability or<br>different sampling strategies. The investigations focus on the quantities and the importance of microplastic volumes in the individual entry points<br>of the urban wastewater system into the water bodies.   |
| SubµTrack  | Tracking of (Sub)Microplastics of Different Identities -<br>Innovative Analysis Tools for Toxicological and Process-<br>engineering Evaluation. Development of new methods of<br>analysis and evaluation, which will allow for assessment and<br>toxicological investigations of plastic particles of different sizes.  | https://www.wasser.tum.d<br>e/en/<br>submuetrack/startseite/<br>https://bmbf-<br>plastik.de/en/joint-<br>project/submtrack | WP 4 Investigations on entry points and process-related evaluation. Real scenarios are first simulated with reference particles in laboratory wastewater treatment plants at the partner LfU to analyse the fate of the particles within the system. Parallel to this, investigations will be carried out at various measuring points in real wastewater treatment plants.  |
| HELCOM BASE Project -<br>Implementation of the Baltic<br>Sea Action Plan in Russia   | Preliminary study on synthetic microfibers and particles at a municipal wastewater treatment plant  | https://helcom.fi/helcom-<br>at-work/projects/base/  | Study the amount of microplastic litter arriving at the Central Wastewater Treatment Plant (WWTP) of St. Petersburg and the effect of the purification process. Helsinki Region Environmental Services Authority HSY developed a microplastic sampling method targeted at wastewaters.  |
| ENSURE Development of New<br>Plastics for a Clean<br>Environment by Determining<br>of Relevant Entry Points.   | Holistic approach to reduce plastic in the environment as well<br>as the related negative consequences, including improving<br>methods of analyzing the environmental impact of plastics.   | https://www.ensure-<br>project.de<br>https://bmbf-<br>plastik.de/en/joint-<br>project/ensure                               | In the "Traceability" module, undesirable plastic inputs in prioritised sectors (soils, wastewater treatment plants, composting plants and biogas<br>plants) are detected and identified. In a first step, sampling strategies are developed so that in a second step representative investigations can be<br>carried out to determine the current states of plastics in fermentation, compost and wastewater treatment plants.   |
| PLASTRAT - Strategies for<br>Reducing the Entry of Urban<br>Plastics into Limnic Systems   | Development of solution strategies for the sustainable<br>limitation of plastic residues propagation in the aquatic<br>environment. Emphasis will be put on the analysis and<br>evaluation of degradation levels of various types of plastic as<br>well as leaching, adsorption, and desorption in different<br>wastewater treatment stages. The focus will also be on the<br>quantification and technical reduction potential (e.g. use of<br>membrane technology) of plastic emissions in the field of urban<br>water management including sewage sludge treatment under<br>consideration of sampling, sampling preparation, and analysis<br>methods. | http://www.plastrat.de/pr<br>oject/<br>https://bmbf-<br>plastik.de/en/joint-<br>project/plastrat                           | WP 2: Degradation and material dynamics. The release of pollutants (oligomers, additives and their transformation products) is analysed depending on the type of polymer and the degree of degradation. Furthermore, differences in the adsorption/desorption of environmenta chemicals are investigated for different types of polymers and the role of wastewater treatment plants regarding the pollutant load or microplastics (enrichment or depletion). WP 3: Entry points and elimination. Different entry points of microplastics into limnic systems are assessed, focusing on the analysis and evaluation of the wastewater management system including measures for microplastic retention (e.g membrane technology) that already exist or have been modified or developed in the course of the project. Core areas comprise rain and mixed water discharge, the assessment and evaluation of the entire wastewater treatment plant system, and an analysis of sewage sludge, digestate and compost as possible microplastic sinks. Emphasis is also put on the development and selection of suitable processing and analytical methods. |
| REPLAWA Reduction of the<br>Input of Plastics via<br>Wastewater into the Aquatic<br>Environment.   | The project will investigate and quantitatively assess entry<br>points into water bodies through treatment plants, storm water<br>drainage, and combined sewer overflows as well as swales at<br>treatment facilities and in sewage sludge.   | <u>www.replawa.de</u>  | The project will test and rate various practical methods of reducing and eliminating plastic emissions into waterways during wastewater treatment. Based on the results of these investigations and of assessments regarding international regulatory approaches in this field, the project will derive strategies for reducing plastic release from wastewater treatment into waterways.   |
| TextileMission –<br>Microplastics of Textile Origin<br>- A Holistic View: Optimized<br>Processes and Materials,<br>Material Flows and<br>Environmental Behavior. | Synthetic fiber particles with a diameter of less than 5<br>millimetres are only partially filtered out by modern<br>wastewater treatment plants. The partners of the joint resreach<br>project TextileMission have taken on the task of reducing this<br>environmental impact.   | <u>https://textilemission.bsi-</u><br><u>sport.de/en/</u>  | WP 4 Polyester fibres: At the TU Dresden, analysis and sample preparation method is established to quantify microparticles from wastewater<br>streams and fractionate them according to size. Investigation of the retention capacity of textile (fluorescence-labelled) microparticles in the<br>different stages of a laboratory wastewater treatment plant and identification of efficient retention possibilities. Material flows are analysed<br>including a first estimate of the Germany-wide textile microplastic emission from wastewater into water bodies /soils. WP 5: Consideration or<br>other environmental issues related to the project, stakeholder involvement and communication at the end of the project. Focus on the retention<br>of textile microfibre particles through various purification stages in wastewater treatment plants.  |



Concerning the management of civil society organizations, projects such as INTCHATCH and InRePlast and CCB Guidelines provide useful information on the removal of PM in wetlands, rainwater ponds and drainage systems.

| Project-Activity Title  | Description   | Links  | Interest for Microplastics  |
|---|---|--|---|
| NTCATHC   | Monitoring and management of surface water quality;<br>developing efficient, user-friendly water monitoring<br>strategies and systems based on innovative technologies<br>that will provide real time data for important parameters,<br>moving towards SMART Rivers.  | https://www.intcatch.eu/   | Manage water pollution in surface runoffs and CSO:<br>Expert Team provides customers with the most effective and low-cost solutions of Combined Sewer Outflow (CSO) and surface runoff<br>treatment systems<br>A wide range of completely automatic and fully adaptive treatment systems are offered depending on customer's needs including<br>combination of coarse screening system, rotating dynamic filter, quartzite filter, GAC adsorption system and UV disinfection  |
| InRePlast –<br>Environmental Policy<br>Instruments to Reduce<br>Plastic Pollution of<br>Inland Waters through<br>Drainage Systems | How and which plastics end up in wastewater and how<br>these inputs can be reduced with the help of<br>environmental legislation is the focus of the joint research<br>project InRePlast. Based on an analysis of sources, entry<br>points and polluters, the researchers are developing and<br>testing measures for behavioural changes. | www.inreplast.de<br>https://bmbf-<br>plastik.de/en/joint-<br>project/inreplast   | <ul> <li>WP 1: Inventory of the entry points of plastics into the drainage systems. Determine the status quo of plastic discharges into the drainage systems in four selected model municipalities, which represent different settlement structures. For this purpose, the type and quantity of plastic products as well as their sources from households, industry, traffic and other activities related to wastewater collection and drainage of paved areas will be determined.</li> <li>WP 4: Material flow models of plastic inputs into canalisation systems and wastewater treatment plants. Material flow models for plastic discharges of macro- and microplastics from the identified sources into the drainage systems will be developed for the four model communities. Based on the model results, the sources and product types are analysed and evaluated with regard to their relevance for the plastic inputs. The data from the municipalities is then extrapolated to the federal level in a general model.</li> </ul> |
| Guidance on concrete<br>ways to reduce<br>microplastic inputs<br>from municipal<br>stormwater and<br>wastewater discharges        | concrete ways to reduce micropalstics from stormwater<br>and wastewater and simple methodology to monitor<br>riverine inputs of micropalstics.  | https://www.ccb.se/document<br>s/Postkod2017/CCB%20-<br>%20Guidance%20on%20concre<br>te%20ways%20to%20reduce%<br>20microplastics%20in%20stor<br>mwater%20and%20sewage.pd | Constructed free water surface wetlands can be efficient in reducing microplastics from effluents of WWTPs to the water bodies.HighMPconcentrationsfoundinurbanstormwater.Stormwaterpondsusedasendofpipesolutionsshowgoodremovalefficiencyformicroplastics.Methodsforsamplingandanalyzingmicroplasticcontentsinwater.Abundance of microplastics smaller than 300 µm in stormwater and sewage water.usedstormwater.usedusedusedusedusedusedAbundance of microplastics smaller than 300 µm in stormwater and sewage water.usedusedusedusedusedusedusedusedAbundance of microplastics smaller than 300 µm in stormwater and sewage water.usedus  |





New treatment technologies are being developed in the SMART-Plant, CLAIM, Plastfri Roskilde Fjord and SimConDrill projects. New commercial products are also under development, such as Wasser 3.0 PE-X<sup>®</sup> technology.

| Project-Activity Title  | Description  | Links  | Interest for Microplastics   |
|---|--|--|--|
| SMARTPLANT<br>Scale-up of low-carbon<br>footprint MAterial<br>Recovery Techniques in<br>existing wastewater<br>treatment PLANTs | SMART-Plant will scale-up in real environment eco-<br>innovative and energy-efficient solutions to renovate<br>existing wastewater treatment plants and close the<br>circular value chain by applying low-carbon techniques to<br>recover materials that are otherwise lost. | https://www.smart-plant.eu/  | Salsnes Filter AS is a technology provider and a partner of SMART-Plant. Salsness filters have been tested for the treatment of different water fluxes, including the effluent from WWTPs. Salsnes has developed a unique fine mesh sieve system for treatment of municipal and industrial wastewater, a mechanical wastewater treatment system, with integrated thickening and sludge dewatering. The patented filter technology is a very compact unit for mechanical separation of suspended solids from wastewater. With the integrated sludge dewatering unit, Salsnes Filter wastewater treatment processing plants meet the highest standards and the specifications of the European Commission for reduction of waste effluents (primary treatment). Salsnes Filter has significant experience from extensive national and international R&D activities, including FP6 and FP7 projects. |
| Plastfri Roskilde Fjord<br>project  | Investigating, mapping and identifying plastic pollution<br>effects and sources, and finding concrete solutions and<br>actions to prevent plastic pollution  | https://plasticchange.dk/viden<br>scenter/plastfri-roskilde-fjord-<br>plastikforurening-i-danmark/ | Alfa Laval supplied a pilot membrane filtration system that was used to determine the amount and type of microplastics in the main wastewater treatment plant, which releases treated water into Roskilde Fjord.   |
| CLAIM - Cleaning Litter<br>by developing and<br>Applying Innovative<br>Methods in european<br>seas                              | Development of innovative cleaning technologies and<br>approaches, targeting the prevention and in situ<br>management of micro and macroplastics in the<br>Mediterranean and Baltic Sea.   | https://www.claim-<br>h2020project.eu/   | <ul> <li>Pre-filtering system to retain larger plastics, while simultaneously taking two samplers;</li> <li>Photocatalytic nanocoating device for cleaning microplastics in wastewater treatment plants to obtain the degradation of low-density polyethylene (LDPE) microplastic, by visible light-induced heterogeneous photocatalysis activated by Zinc oxide nanorods.</li> </ul>  |
| SimConDrill - Innovative<br>filter modules for the<br>separation of<br>microplastics from<br>wastewater                         | Development of a filter that is ready for serial production,<br>which enables the filtration of particles down to 0.01mm<br>(this equals the thickness of household aluminium foil)<br>based on the patented cyclone filter.   | https://www.simcondrill.com/   | New water filter removes microplastics with laser-drilled tiny holes. A group of five partners from industry and research now wants to develop a new filter that uses laser-drilled holes to efficiently filter out particles as small as 10 micrometres even in large amounts of water.   |
| Wasser 3.0 PE-X®  | Simple, reproducible and cost-efficient processes with no<br>negative environmental effects for the elimination of<br>microplastics from sewage water  | https://wasserdreinull.com/  | With Wasser 3.0 PE X it has been developed the first method to remove microplastics from the water without any additional complicated filter technology. By applying a non-toxic hybrid silica gel, table tennis ball sized balls float on the water surface and are easily removed from there.  |



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#### Thank you for the attention!



BENEFICIARIO COORDINATORE



UNIVERSITÀ Politecnica Delle Marche





BENEFICIARI ASSOCIATI











PROGETTO COFINANZIATO DA



**Blue Lakes** No microplastics, just waves.

# Sampling methodologies Università Politecnica delle Marche



















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## **Sampling methods**



Most of the research on microplastics in water has been focused on seas and oceans. As a result, sampling techniques and protocols have been developed primarily for marine systems.

To date, there is no standardized methodology for sampling microplastics in inland waters or treatment plants. The development of a uniform protocol is necessary for the correct interpretation and comparison of the results. The main variables to consider are:

- SAMPLING POINTS
- SAMPLING VOLUMES
- FILTRATION MESH
- TYPE OF SAMPLE AGGREGATION (e.g. weighted average)
- SAMPLING PERIODS
- SAMPLING FREQUENCIES



#### Sampling in surface waters: Monitoring Protocol developed by ENEA



### Sampling in surface waters: Monitoring Protocol developed by ENEA

.akes



agenzia regionale per la protezione ambientale

#### Sampling in surface waters: Monitoring Protocol developed by ENEA



www.lifetituelation.or./ http://wikiuelation.or.

CORSO DI FORMAZIONE

#### MONITORAGGIO DELLE MICROPLASTICHE: IL PROTOCOLLO BLUE LAKES

ARPA UMBRIA, in collaborazione con ENEA organizza un corso di formazione di tre giorni sui monitoraggio delle microplastiche lacustri finalizzato alla condivisione del Protocollo Standard Blue Lakes, il corso è rivolto ai tecnici e personale ARPA/APPA, La durata è di tre giorni, le lezioni frontali e le attività di laboratorio si svolgono presso i Laboratori ARPA di Terni, mentre le attività di campionamento sono effettuate sul lago Piediluco, area pilota del progetto Blue Lakes.



#### PROGRAMMA PROVVISORIO DEL CORSO

26-09-2022 ore 15:00-19:00 (in sede) 03-10-2022 ore 15:00-19:00 (in sede)

- Registratione
- Introduzione al corso
- Il progetto Blue Lakes: Il monitoraggio delle microplastiche



#### Sede del Como ARPA Umbria Vis Carlo Alberto Dalla Chiesa, 32, 05100 Terni TR

#### Contatti

Responsabili scientifici e organizzativi Maria Sighicalit: tal. 05 30486348 call 340 2936190 maria sighicali @anaa.it Patrizia Menegoni: tel 06 30484278 patrizia menegoni@enea.lt Valentina Della Bella: tel. 0744 4796737; cell. 335 1800862; v.dellabella@arpa.umbrla.it Paolo Straniert: 075 5 1596218: p.stranieri@arpa.umbria.lt Valentina Stufara: tel. 075 51596649 mail: v.stufara@arga.umbria.tt

#### Come raggiungere la sede del Corao Dalla stazione ferroviaria di Terni: Bus 1, 9, 0616, 0618, in subsc

Reccordo Terral - Orte (\$5675): Uscha Terral Nord



#### Informationi albergo convenzionato

**Classic Hotel Tulipano** 

Via Carlo Alberto Dalla Chiesa, 24 Loc. Borgo Rivo Terri 05300 tel: +39 (0) 744 306024 fac: +39 (0) 744 300628 e-mail: info@classichotelterni.com





#### Attività di campionamento:

- uscita in barca per prelievo di campioni di acqua superficiale e colonna
- lungo le splagge per prelievo sabbie







28-09-2022 ore 9:00 -16:00 ( in sede) 05-10-2022 ore 9:00 -16:00 ( in sade)



- Anailsi dei campioni prelevati
  - (sorting a caratterizzazione)
- Discussione risultati











## **Sampling methodologies**

ISO/DIS 24187 Principles for the analysis of microplastics present in the environment

Status : Under development



<u>Key principles</u> for the investigation of microplastics in the environment and for the subsequent development of specific procedures for sampling, processing and analysis.

<u>Minimum requirements</u> for particle size classification, the use of certain sampling equipment, sample preparation and the determination of representative sample quantities.

 $\rightarrow$  The particle size to be considered is strictly related to the detection method.

 $\rightarrow$  This standard does not include requirements for monitoring actions





## Water and Waste Environmental Engineering

### **Sampling methodologies**

ISO/DIS 24187 Principles for the analysis of microplastics present in the environment

| DRAFT INTERNATIONAL STANDARD |
|------------------------------|
| ISO/DIS 24187                |

| ISO/TC 61/SC 14                     |  |
|-------------------------------------|--|
| Voting begins on: <b>2021-12-08</b> |  |

Secretariat: DIN

Voting terminates on: **2022-03-02** 

Principles for the analysis of plastics and microplastics present in the environment

To be considered: different physical and chemical properties, such as shape, size (range), type of polymer(s), presence of additives, presence of fillers, state of degradation.



## **Sampling methodologies**



### ISO/DIS 24187 Principles for the analysis of microplastics present in the environment



- Avoid cross-contamination → blank
- Evaluate if sterilization step is necessary in the processing phase
- Record relevant information on measurement and analysis conditions


# ISO/DIS 24187

Principles for the analysis of microplastics present in the environment

Subdivision into dimensional ranges

| classification   |        | large mi-<br>croplastics |                   | microplastics   |                       |                       |                       |                       |  |  |  |
|--|--------|--------------------------|-------------------|-----------------|-----------------------|-----------------------|-----------------------|-----------------------|--|--|--|
| particle size<br>classes   | μm     | 5 000 to 1 000           | < 1 000 to<br>500 | < 500 to<br>100 | < 100 to<br>50        | < 50 to 10            | < 10 to 5             | < 5 to 1              |  |  |  |
| average parti-<br>cle size   | μm     | 3 000                    | 750               | 300             | 75                    | 30                    | 7,5                   | 3                     |  |  |  |
| mass <sup>a</sup>  | mg     | 14                       | 0,22              | 0,014           | $2,2\times 10^{-4}$   | $1,4\times 10^{-5}$   | $2,2 \times 10^{-7}$  | $1,4 \times 10^{-8}$  |  |  |  |
| number of<br>particles in<br>14,13 mg  | number | 1                        | 64                | 1 000           | 6,4 × 10 <sup>4</sup> | 1,0 × 10 <sup>6</sup> | 6,4 × 10 <sup>7</sup> | 1,0 × 10 <sup>9</sup> |  |  |  |
| <sup>4</sup> Mass here is determined from the average particle size assuming spherical particle with a density of 1. |        |                          |                   |                 |                       |                       |                       |                       |  |  |  |

## • Choice of the method of analysis

- Fourier Transform Infrared Spectroscopy (FTIR),
- Attenuated Total Reflection Fourier Transform Infrared Spectroscopy (ATR-FTIR),
- Focal Plan Array Detector Fourier Transform Infrared Spectroscopy (FPA-FTIR),
- Quantum cascade laser induced infrared spectroscopy (QCL-IR),
- Near or Short-Wave Infrared Spectroscopy (NIR, SWIR).

The analysis of very small particles (<5 μm) is complex and partly limited for real samples.









ISO/DIS 24187 Principles for the analysis of microplastics present in the environment

SAMPLING VOLUME

The sampling volume depends on the size range of the microplastics under investigation and the expected number of particles or mass. It is assumed that the smaller the diameter of the particle is, the greater its presence is.

The smaller is the expected particle quantity, the more sample must be taken in order to examine a representative number of particles.

Very large representative sample volumes need to be taken in almost solid-free water bodies.





## ISO/DIS 24187 Principles for the analysis of microplastics present in the environment

#### FILTRATION MESH

Subdivision into dimensional classes based on the filtration ranges.

In case of high concentrations of solids in the water sample and large sample volumes, a fractional filtration helps to reduce the filter cake formation and the consequent blockage or partial blockage of the filters.

The volume of water sampled and the volume of water actually filtered must always be reported in the documentation of sampling methods.

 $\rightarrow$  To ensure the quality of the filtration process, recovery tests are recommended.





ISO/DIS 24187 Principles for the analysis of microplastics present in the environment

FILTRATION MATERIALS

#### FILTRATION EQUIPMENT

References to filtration methods non-specifics for microplastics for the different environmental matrices (marine waters, surface waters, groundwater)

#### SLUDGE

In sewage sludge analysis, traditional sampling practices are not easily transferable to the field of MPs. The size and number of plastic particles, in particular, play an important role in representative sampling.





There is a huge differentiation in the volume of water and sludge samples taken from influent and/or effluent as well as from different treatment plant processes.

Sampling can be done in several ways, mainly including:

- Collection in containers and subsequent filtration
- Collection with autosampler and subsequent filtration
- Pumping and filtration
- Composite filtration devices

**PROS:** easier from an operative point of view **CONTRA:** limited volumes

**PROS:** higher volumes **CONTRA:** not common, developed for specific projects

Due to the relatively low concentrations of microplastics and their non-uniform temporal and spatial distributions in waters, the representativeness of the sample should be considered when interpreting the data and defining the sampling methodology.

Research has highlighted the need to analyze microplastic pollution in treatment plants over a longer period of time to reveal the temporal variation of microplastic concentrations.





## Minimum sampling volumes proposed in BLUE LAKES

| Sector                  | Plant                         | Unit                                | Type of sample             | Min.<br>volume | Min. number<br>of samples | Notes  |
|-------------------------|-------------------------------|-------------------------------------|----------------------------|----------------|---------------------------|--|
|                         |                               | Influent                            | Min 1-2 h average sampling | 1000           | 3*                        | *Min. number of sampling campaigns defined to detect seasonal variability.                       |
|                         |                               | Effluent from<br>each operativeunit | Min 1-2 h average sampling | 1000           | 3*                        |  |
| Duinking                | Drinking water                |                                     |                            |                |                           |  |
| Drinking<br>water       |                               | Final Effluent                      | Min 1-2 h average sampling | 1000 l         | 3*                        |  |
| supply                  |                               | Sludge**                            | Grab                       | 51             | 3*                        | **Sludge is considered as liquid at maximum TS% of about 5%TS.                                   |
|                         | Distribution                  | Final<br>Distribution***            | Min 1-2 h average sampling | 1000           | 3*                        | ***Min. Number of Sampling points has to be set accordingto the distribution network complexity. |
| Sewage<br>system        | CombinedSewer<br>Overflow     | CSO                                 | Grab or Average sampling   | 50  ****       | 3*                        | ****Min volume could be very variable depending on the quantity overflowed.                      |
|                         | Wastewater<br>Treatment Plant | Influent                            | Average sampling           | 30-300  *****  | 3*                        | *****Min. volume could be very variable depending on watercharacteristic.                        |
| Wastewater<br>Treatment | Wastewater<br>Treatment Plant | Effluent from each operativeunit    | Average sampling           | 30-300   ***** | 3*                        |  |
|                         | Wastewater<br>Treatment Plant | Final Effluent                      | Average sampling           | 30-300   ***** | 3*                        |  |
|                         | Wastewater<br>Treatment Plant | Sludge**                            | Grab                       | 51             | 3*                        |  |





#### Scheme for sampling activity

| SAMPLING POINT | DATE | WEATHER | METHOD | START OF<br>SAMPLING | DURATI<br>ON | END OF SAMPLING | VOLUME | CHEMICAL AND<br>PHYSICAL<br>CHARACTERISATION<br>(es. TSS) | NOTES |
|----------------|------|---------|--------|----------------------|--------------|-----------------|--------|---|-------|
|                |      |         |        |                      |              |                 |        |   |       |
|                |      |         |        |                      |              |                 |        |   |       |
|                |      |         |        |                      |              |                 |        |   |       |

Write down any particular condition (e.g. unit malfunctions, events, anomalies during sampling, ...)

Possibly match the sampling with the routine characterizations of the plant





#### **Sampling points**



Carbon filters





**Optimisation of sampling methods** 





Istantaneous sampling and subsequent filtration



Limited quantities of volumes (about 5-20 L)



For **sludge samples**, which are generally taken discontinuously, in situ collection and subsequent filtration in the laboratory on batteries of sieves are generally used.

Prior to filtration, sludge samples are stored at -20°C if not processed immediately.





## ATTENTION TO THE CONCEPT OF SAMPLING YIELD

Recovery capacity of standard MPs/ introduced MPs %

Also by Size Class







#### Sieving

The sieve batteries consist of steel sieves with mesh sizes of 5 mm, 2 mm and 50  $\mu$ m (ISO 3310-1:2000). They are used in case of instantaneous sampling, as this procedure is feasible only for limited quantities of volume (e.g. 25 litres).





A pumping system may be required to convey the water flow to the sieve battery. The pumping system must be made with nonplastic components, to avoid contamination. In particular, the pump is generally made of steel and the connections are made of copper or brass.





## Sieving

- 1. Adjust the water flow and record it manually.
- 2. Calculate the time required to filter the desired volume in situ
- 3. Filter the sample through the battery of steel sieves with 5 mm, 2 mm and 50  $\mu m$  meshes (ISO 3310-1:2000)
- 4. BLANK: To control any external environmental contamination, place a jar filled with deionized water up to <sup>3</sup>/<sub>4</sub> of the volume. Leave the can open during the entire filtration time through the sieves and during material recovery operations. Close the jar with the lid and then with the parafilm.
- 5. After filtration, remove the 5 mm mesh sieve: this sieve is used in sets to exclude any plastic particles larger than 5 mm that may be present. Material retained on the 5mm sieve should not be recovered.
- 6. Rinse the particles on 2 mm and 50  $\mu m$  sieves in glass jars with water
- 7. Use spray bottles and/or hand pressure pump with deionized water to collect material retained on sieves. The recovered material must be stored in glass jars closed with lids and then with parafilm.
- 8. Identify the sample by writing directly on the can or on white tape/packet attached to the container. The stored sample will then be processed later in the laboratory.











#### Sieving

Pittura et al., 2021 "Microplastics in real wastewater treatment schemes: Comparative assessment and relevant inhibition effects on anaerobic processes" Chemosphere, 262, 128415

## Wastewater processing for MPs analysis







#### **Closed filtering systems**

Closed filter systems are preferably used when there is the possibility of connecting the filter system directly to the system pipes.

Direct filtration allows to obtain composite samples during the filtration time and can process larger volumes (about 1000 L).

The filtering device consists of a metal container inside which a cylindrical filter with a 50  $\mu$ m mesh is housed. The filter cartridge is made of micro-expanded stainless steel sheet.

The connections between the cartridge filter and the system piping are made with copper pipes to avoid any plastic contamination.







| Operating pressure            | 30 barg            |
|-------------------------------|--------------------|
| Filtration degrees            | From 50 µm onwards |
| Design pressure               | 45 barg            |
| Maximum operating temperature | 70 °C              |
| Inlet/Outlet flanges          | Ø 1" 1/2           |
| Discharge                     | Ø 3/8              |
| Drain valve                   | Manual ball        |





#### **Closed filtering systems**

- 1. Connect the filter to the system sampling tap using suitable copper pipes and brass fittings.
- 2. Adjust the water flow and record it manually.
- 3. Calculate the time required to filter the desired volume in situ, through a 50  $\mu$ m stainless steel filter.











**Closed filtering systems** 

- 4. Once filtration is complete, place the filter in a steel pan or glass beaker and rinse the filter with deionized water to recover any particles retained on the filter. If necessary, use pressure washers and/or manual pressure pumps that can facilitate/optimize the operation.
- 5. Place an empty jar filled with deionized water and leave open during the wash step for each sample. This will be used as a control to account for any contamination from the air.
- 6. Once the filter is completely cleaned, pour the washed water into a glass jar and rinse the bowl in the jar to recover all particles. Close the jar with its lid and then using the parafilm.
- 7. Name the samples and each control jar, then take them to the laboratory for processing.













**Closed filtering systems** 

Drinking water treatment plants

SAMPLING A VOLUME OF ~1000 L AND FILTERING WITH STEEL CARTRIDGE FILTER DEVICE (50 micron)





 Polymers identification by µFT-IR in ATR mode.

CHARACTERIZATION WITH IR RECOVER OF MATERIALS AND DIGESTION WITH H2O2 OF ORGANIC MATTER

6. Visual examination and collection of fibers and particles.

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Blue
Lakes



### **Closed filtering systems**



González Camejo, J., Morales, A., Peña-Lamas, J., Lafita, C., Enguídanos, S., Seco, A., Martí, N., 2022. Feasibility of rapid gravity filtration and membrane ultrafiltration for the removal of microplastics and microlitter in sewage and wastewater from plastic industry. J. Water Process Eng. 03853. (submitted)

Be careful if they form layer a control necessary to stop filtration

- Sampling device for in situ filtration with a wide range of sizes simultaneously during sampling.
- Ability to continuously filter a large volume of wastewater.
- Divide the sample into several fractions, so that each of them contains solids of a certain size range.
- Filters and/or sieves of different pore sizes.
- Four removable stainless steel links with sizes of 500, 190, 100 and 25 μm.
- In this way it is possible to separate particles with diameters between >500, 190-500, 100-190 and 25-100 μm.
- The links have a diameter of 20 cm and are placed on bars stacked on top of each other.
- The stack of rods is placed inside a rigid polypropylene case with a base and lid that serve as the body of the sampling device.
- The cap and base are tight to the casing to prevent any leaks that could occur at the edges of the displays.
- Flow meter at the sampler head, to know the volume of the sampled wastewater at any time





QA/QC: Prevention and control of external contamination from plastic material and fibres

Main measures adopted during all phases: sampling, recovery, processing and characterization

- use of non-plastic material (stainless steel, glass, aluminum, copper)
- if plastic materials are used (gaskets, washers...) the material must be characterized to eventually exclude it from the results.
- Closed sampling system vs system exposed to the atmosphere.







 Execution of blank samples for each sample collected: a beaker containing deionized water is left open on the workbench for the entire time necessary to recover the sample and subsequently processed according to the same procedures as the water or sludge sample.



#### **Automatic sampler**

The automatic sampling system, currently under development by the WWEELab (Marche Polytechnic University), consists of a timed sampling pump, a cartridge filtration system (50 micron), an electromagnetic flow meter and PLC data logging and control. This allows you to acquire a composite sample (24 hours) and filter larger quantities of water (up to 300-1000 L for wastewater and up to 5000 L for potable water). Flow rate measurements will allow you to know exactly how much volume is being filtered and to stop the pump when the flow rate drops below a certain value due to clogging problems.









#### **Automatic sampler**

The device can be set to work with time control or combined time and flow rate.

When set in timed mode, the pump is activated only at certain time intervals, with filtering cycles and pause cycles. In this way it is possible to perform average samples.

When the device works in time and flow control, the pump automatically interrupts its filter/pause cycles when the flow reaches a predefined lower threshold, which means that the filter has reached a certain clogging level.









#### **Automatic sampler**



- Automatic recording of sampled volumes
- Possibility to modulate sampling intervals and periods
- Time-based and flow-based regulation
- Possibility of filtering large volumes (≈ a few m3)
- Representativeness of plant variations
- Ease of use by operators





**Automatic sampler** 

#### NEW PROTOTYPE OF AUTOMATIC SAMPLER TESTED IN A FULL-SCALE WWTP

- Flowrates meter with data recording
- Pressure meter
- Subsequent filters of 50  $\mu m$  and 25  $\mu m$



🖌 50 μm filter



 $25\,\mu m$  filter



Filtered volumes registration





Tests to identify the minimum representative volume for the quantification of microplastics in purification plants



By increasing the sampling volume, the concentrations of microplastics decrease, until they reach a stable level:

- 100-400 L of filtered influent
- 1000-2000 L of filtered effluent



Maximum volume filtered at different solid concentrations





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#### Thank you for the attention!



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# **Extraction and characterization methodologies**

# Samples processing for microplastics analysis in Integrated Water Service matrices

Università Politecnica delle Marche



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## **Extraction and characterization of MPs from environmental matrices**



#### **COMPLEX MATRICES**

Inland water, wastewater, sludge, treated water : mix of organic matter and mineral

components, often in aggregation.

+

#### **HETEROGENEOUS CLASS OF CONTAMINANTS**

Microplastics (MPs) are a complex group of synthetic solid particles varying for sizes (5mm-1µm), shapes, colors, density, surface charge and chemical compositions.





From Monteiro & da Costa, 2021

SAMPLES ARE PROCESSED TO REDUCE MASS (OR VOLUME) AND ISOLATE MPS FROM OTHER ORGANIC AND INORGANIC PARTICLES.

PROPERLY ISOLATING MPS IS THE KEY TO OBTAIN HIGH EXTRACTION EFFICIENCIES, GUARANTEE PARTICLE PRESERVATION AND GENERATE ACCURATE DATA.



# DIGESTION **DENSITY-BASED SEPARATION** SIEVING/FILTRATION

- The 3 procedures are usually applied in combination using different methods which vary in complexity, time and cost of materials.
- The choice of method often depends on the type of sample (water vs sludge/; organic matter and mineral content).
- The extraction efficiency is influenced by: type of matrix, MPs characteristics, applied method.
- In turn, MPs characteristics (shape, color, surface texture, chemical composition) can be altered by the extraction method applied.



Silvia S. Monteiro\*, João Pinto da Costa



#### DIGESTION

- Used to eliminate organic material and to remove biofilm from the surface of MPs.
- Applied to waste/treated water and sludge samples as first processing, due to the high concentration of organic matter in these matrices: pretreatment to filtration and density-based separation procedures.
- There are several methods of digestion, used singly or in combination. The digestion efficiency and the effect on MPs depend on the type of agent used and the concentration, the incubation time and the working temperature.
- Acid digestion (HNO3 or HCl): the most effective but also the most destructive method, it causes the degradation of various plastic polymers especially at high acid concentrations and at high temperatures. Method not recommended as it may lead to underestimation of MPs.
- Alkaline digestion (KOH): valid alternative, it is a simple, inexpensive method, relatively low chemical risk and few effects on plastic polymers. However, it has low efficiency in degrading cellulose and chitin, which are common components of soil and sludge. Most commonly used method for processing samples of animal origin.
- Oxidative digestion (H<sub>2</sub>O<sub>2</sub>): most popular method. Little effect on the integrity of the MPs if used at less than 20%: at higher concentrations, changes in transparency and shrinkage of the dimensions of the MPs and interferences in the chemical characterization phase can be observed. Long digestion times at room temperature which can be reduced if carried out at room temperature (less than 40°C).
- H<sub>2</sub>O<sub>2</sub> +Fe<sup>2+</sup> (catalyst): faster reaction, low cost of reagents, high degradation efficiency of lignin, cellulose, EPS. However highly reactive (caution!) and can decompose MPs in an acidic environment.
- Enzymatic Digestion (e.g. Proteinase-K): simple, low-risk method with high recovery rates and no adverse effects on plastics. However, method onerous and often requires long incubation periods. Applicable to small volumes (or quantities) of samples.

#### **DENSITY-BASED SEPARATION**

- Applied to separate MPs from non-organic material.
- The process is based on the buoyancy properties of MPs in a denser saline solution: the sample is mixed with the saline solution and left to decant to collect the supernatant while heavier non-plastic debris settles on the bottom. The extraction efficiency increases by repeating the procedure.
- Duration of mixing and settling time can vary considerably depending on the volume and type of sample, from minutes to several hours or days.

....

• There is a range of salts that varies in density, cost and toxicity.

#### Comparison of the main salts used for density-based separation (Frias et al., 2018)

| Salt                     | Abbr.             | Toxicity<br>(Health hazard) | Price<br>(€/500g,<br>listino <i>Merck</i> ) |
|--------------------------|-------------------|-----------------------------|---|
|                          |                   |                             |   |
| Cloruro di sodio         | NaCl*             | Low                         | 30  |
| Sodio tungstato diidrato | STD               | Low                         | 303   |
| Bromuro di sodio         | NaBr              | Low                         | 80  |
| Ioduro di sodio          | Nal               | Moderate                    | 345   |
| Cloruro di zinco         | ZnCl <sub>2</sub> | High                        | 90  |
| Bromuro di zinco         | ZnBr <sub>2</sub> | High                        | 170   |
| Sodio politungstato      | SPT               | Low                         | 1300  |

**\*NaCl** the most common used, inexpensive, readily available and ecofriendly, recommended for monitoring plans with high number of samples. May have a low recovery rate for MPs with similar or slightly higher densities.



Buoyancy of plastic polymers in saline solutions based on their respective densities (g cm-3)

| Polymer                       | Abbr. | Polymer density | <b>NaCl*</b><br>(1.2) | STD<br>(1.4) | NaBr<br>(1.4) | Nal<br>(1.4-1.8) | ZnCl <sub>2</sub><br>(1.6-1.8) | ZnBr <sub>2</sub><br>(1.1.7) | SPT<br>(2.94-3) |
|-------------------------------|-------|-----------------|-----------------------|--------------|---------------|------------------|--------------------------------|------------------------------|-----------------|
| Polystyrene                   | PS    | 0.01 - 1.06     | +                     | +            | +             | +                | +                              | +                            | +               |
| Polypropylene                 | PP    | 0.85 – 0.92     | +                     | +            | +             | +                | +                              | +                            | +               |
| Low-density<br>polyethylene   | LDPE  | 0.89 – 0.93     | +                     | +            | +             | +                | +                              | +                            | +               |
| Ethylene vinyl acetate        | EVA   | 0.93 - 0.95     | +                     | +            | +             | +                | +                              | +                            | +               |
| High-density<br>polyethylene  | HDPE  | 0.94 – 0.98     | +                     | +            | +             | +                | +                              | +                            | +               |
| Polyamide                     | PA    | 1.12 – 1.15     | +                     | +            | +             | +                | +                              | +                            | +               |
| Nylon 6,6                     | PA 66 | 1.13 – 1.15     | +                     | +            | +             | +                | +                              | +                            | +               |
| Poly methyl<br>methacrylate   | PMMA  | 1.16 – 1.20     | +-                    | +            | +             | +                | +                              | +                            | +               |
| Polycarbonate                 | PC    | 1.20 – 1.22     | +-                    | +            | +             | +                | +                              | +                            | +               |
| Polyurethane                  | PU    | 1.20 – 1.26     | +-                    | +            | +             | +                | +                              | +                            | +               |
| Polyethylene<br>terephthalate | PET   | 1.38 – 1.41     | -                     | +-           | +             | +                | +                              | +                            | +               |
| Polyvinyl chloride            | PVC   | 1.38 – 1.41     | -                     | +-           | +-            | +                | +                              | +                            | +               |
| Polytetrafluoroethylene       | PTFE  | 2.10 - 2.30     | -                     | -            | -             | -                | -                              | -                            | +               |





#### SIEVING/FILTRATION

- **SIEVING** : applied as a first phase to remove coarse material (5mm mesh), reduce the volume (or mass) of the sample (if not done in the sampling phase), make a first selection of MPs on a dimensional basis.
  - > Use of a battery of sieves with mesh in the 5mm-20 $\mu$ m range.
  - > The material retained by the <5mm mesh sieves is recovered and subjected to processing for the extraction of the MPs.
  - The portion of sample that passes from the last sieve of the battery can be filtered under vacuum on membranes of lower porosity to recover MPs smaller than 20μm.
- **FILTRATION UNDER VACCUM**: direct filtration of the sample without a pre-treatment (digestion and/or density-based separation) is not recommended, it would require long times and a high workload in the characterization phase of MPs. Applied as the last stage of the extraction process.
  - $\blacktriangleright$  Porosity of membrane filters varies between 0.1 and 20  $\mu$ m.
  - Filters are available in different materials: polytetrafluoroethylene, polycarbonate, nylon, glass fibre, cellulose (nitrocellulose, cellulose acetate or mixed cellulose), stainless steel, aluminum oxide.
  - Type of filter is chosen on the basis of availability, porosity, structure and suitability for the analytical techniques used for the subsequent characterization of the retained particles.









#### **ALTERNATIVE METHODS**

Physical separation of particles according to their lipophilic properties (oil extraction methods)...

A novel, density-independent and FTIR-compatible approach for the rapid extraction of microplastics from aquatic sediments †

#### Ellika M. Crichton, i Marie Noël, Esther A. Gies ab and Peter S. Ross \*a

|          | Science of the Total Environment 733 (2020) 139338  |                   |
|----------|---|-------------------|
|          | Contents lists available at ScienceDirect           |                   |
|          | Science of the Total Environment                    | Lotal Environment |
| ELSEVIER | journal homepage: www.elsevier.com/locate/scitotenv |                   |
|          |   |                   |

Olive oil-based method for the extraction, quantification and identification of microplastics in soil and compost samples



Costanza Scopetani<sup>a,\*</sup>, David Chelazzi<sup>b</sup>, Juha Mikola<sup>a</sup>, Ville Leiniö<sup>c</sup>, Reijo Heikkinen<sup>d</sup>, Alessandra Cincinelli<sup>b,e</sup>, Jukka Pellinen<sup>a</sup>

GRAPHICAL ABSTRACT

#### HIGHLIGHTS

- A methodology for microplastic extraction from solid samples is suggested.
- · Olive oil was selected to extract microplastics from soil and compost samples.
- The developed method is able to extract high density polymers.
- High recovery rate of 90%  $\pm$  2% to 97%  $\pm$ 5% were achieved.
- · Olive oil seems to have high affinity for a wide set of polymers.



# Contents lists available at ScienceDirect



Marine Pollution Bulletin

journal homepage: www.elsevier.com/locate/marpolbul

Retention of microplastics in a major secondary wastewater treatment plant in Vancouver, Canada



Esther A. Gies<sup>a,b</sup>, Jessica L. LeNoble<sup>a</sup>, Marie Noël<sup>a</sup>, Anahita Etemadifar<sup>a</sup>, Farida Bishay<sup>c</sup>, Eric R. Hall<sup>b</sup>, Peter S. Ross<sup>a,\*</sup>

#### ... or surface charge (electrostatic separation).



**Environmental Pollution** Volume 234, March 2018, Pages 20-28



A new approach in separating microplastics from environmental samples based on their electrostatic behavior 🕁

Stefanie Felsing 😕 🖾, Christian Kochleus, Sebastian Buchinger, Nicole Brennholt, Friederike Stock , Georg Reifferscheid 😤 🖾

#### Marine Pollution Bulletin 133 (2018) 553-561

#### QUALITY ASSURANCE AND QUALITY CONTROL (QA/QC)

DURING THE PROCESSING (AND SAMPLING) OF ENVIRONMENTAL MATRICES IT IS ESSENTIAL TO DEVELOP AND APPLY PROCEDURES THAT ENSURE THE ACCURACY AND REPRODUCIBILITY OF DATA.

QA/QC procedures include:

- 1. implementation of contamination mitigation measures: particularly relevant for microfibres.
- 2. determination of the recovery rates of extraction methods: they are influenced by various variables, including the reagents used, the characteristics of MPs (polymer, size, shape) and the environmental matrix.
- 3. evaluation of effects of the extraction methods on the properties of MPs: alteration of size, shape, surface texture, color and characteristics of the polymer.



Particle confirmation %

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#### QA/QC (1): MITIGATION MEASURES ON CONTAMINATION by EXTERNAL MPs

- Avoid using plastic devices and materials, replacing them with glass or metal. If not possible, it is necessary to characterize these materials and compare them with MPs extracted from samples: if they correspond, remove from results.
- Clean materials and instruments with ultrapure/deionized water and/or ethanol, filter solutions, especially saline solutions, (0.22 or 0.45 µm pore size), keep samples covered as much as possible (petri dish lids, aluminum foil).
- The problem of fibers:
- the use of cotton lab coats may contain the release of synthetic textile fibers. However, even natural fabrics can release fibers: the use of lab-coats, gloves and brightly colored paper can help identify accidental contamination.
- it is suggested to process the samples under a laminar flow hood: chemical hoods are poorly efficient in controlling contamination as the unfiltered air from the laboratory is sucked into the hood and then expelled outside. The alternative is to work in rooms with controlled airflow and access (minimum circulation of personnel).
- To check background contamination :
- > Airborne blanks: Beakers with water or wet filter in a Petri dish left open on the workbench during sample processing.
- Procedural blanks: pure reagents (or water) treated with the same procedures and for the same times as the environmental samples.

They can be performed separately or in a single solution (a single blank to control airborne deposition and procedures).













#### QA/QC (2 and 3): RECOVERY RATE OF EXTRACTION METHOD AND IMPACT ON PARTICLES

- The effectiveness of a procedure for extracting MPs from a matrix (recovery rate and impact on particles) can be tested using **recovery experiments** (spiking experiments): addition to the sample of a known number of MPs, of which size, shape, color are also known and polymer («positive control»).
- It is suggested to prepare a heterogeneous mixture of MPs, since the extraction efficiency depends on the method used but also on the characteristics of the particles: commercial MPs (primary origin) or hand-made MPs obtained from commonly used plastic objects can be used (secondary origin).
- **Extraction yield**: measured by calculating the percentage of added MPs recovered at the end of the sample processing, also verifying possible variations in the physical and chemical characteristics of the added MPs.
- Known organic and inorganic material could also be added to the samples to evaluate the incidence of false positives.






#### QA/QC: VALIDATION OF MPs EXTRACTION FROM SEWAGE SLUDGE

#### Digestion with 15% H<sub>2</sub>O<sub>2</sub> solution + density-based separation with NaBr

- 4 types of sludge (primary, active, post-dehydration, granular) + 1 blank sample
- 12 MPs added to each kind of sludge (2 particles of 6 different typologies):
- 1. Polyisoprene ( $\delta$ =0.93-0.98g/cm3). Size: 1-2mm. Hand-made particles from stationery elastics (secondary origin).
- 2. Nylon (PA) ( $\delta$ =1.13-1.15g/cm3). Size: 1-0.5mm. Hand-made particles from fishing lines (secondary origin).
- 3. Polyethylene terephthalate (PET) (δ=1.31-1.43g/cm3). Size: 1.5-2mm. Hand-made particles from plastic bottles (secondary origin).
- 4. Polypropylene (PP) (δ=0.82-0.90g/cm3). Size: 1.5-0.8mm. Commercial particles (primary origin).
- 5. Polyethylene (PE) ( $\delta$ =0.92-0.97g/cm3). Size: 1-0.5mm. Commercial particles (primary origin).
- 6. Polystyrene (PS) ( $\delta$ =1.05-1.06g/cm3). Size: 1-0.5mm. Commercial particles (primary origin).

#### Chemosphere 262 (2021) 128415



Microplastics in real wastewater treatment schemes: Comparative assessment and relevant inhibition effects on anaerobic processes



MPs were photographed, measured and characterized (FT-IR spectroscopy) before and after processing: no appreciable modifications on size, color, polymer IR spectra.

> EXTRACTION YIELD: Primary sludge: 95% Activated sludge: 92% Dewatered sludge: 96% Granular sludge: 98% Blank sample: 100%

# **Analytical Techniques for MPs Characterization**



- PHYSICAL AND CHEMICAL CHARACTERIZATION: once the extraction phase is complete, particles are characterized in terms of size, shape, surface texture, color and, above all, polymer to identify the actual number of items of synthetic nature (i.e. MPs).
- The various techniques available can be distinguished on the basis of their ability to determine some of the properties of microplastics.
- Several techniques can be used in sequence or in association to compensate for the analytical limitations of one or the other.
- The choice of techniques depends on the objective of the study and on the available instruments (often the dimension of microplastics to be analyzed is the driving factor).
- Depending on the objective of the analysis, it may be sufficient to apply a (pre)screening method with relatively simple and inexpensive techniques which may provide limited information but does not require sophisticated instrumentation: cost- and time- effective routine analysis.

# Physical characterization: Microscopy techniques (OM and SEM)

- Physical characterization through microscopy techniques is primarily used to identify and classify microplastics preserved on a filter or in petri dishes or jars.
- OPTICAL MICROSCOPY (OM) is suitable to visually examine particles of submillimeter size retaining the 3D shape and color of suspected MPs but does not provide information on the chemical composition.
- Visual guidelines, physical and tactile guidelines can help the operator in the identification of suspected MPs:

bright and unnaturally colored particles, fragments with sharp geometrical shapes, shiny surfaces, and featureless fibers with a consistent width, the particle holding its shape or stretched when poked and resistance to easy breakage (*Primpke et al., 2020*).

- Advantage of OM: relatively cheap and easy approach.
- **Disadvantage** of OM: it requires considerable time and resources in terms of researchers involved in counting hundreds of particles. It gets better with experience.
- Once particles were identified, they are measured using an image analysis software and categorized by shape, color, and size classes.











**FRAGMENT**: hard particle, thick, with sharp cutting edges and an irregular shape (*Lusher et al., 2017*).



GLITTER: iridescent disc with a hexagonal shape(Yurtsever et al., 2019).



**SPHERE**: particle with every point on the surface having the same distance from the center. It can also be present as a hemisphere, probably due to breakage during production, use or presence in the environment (*Hartmann et al., 2019*).



**FIBER:** filiform structure with irregular diameter and frayed ends which can take trilobate, ribbon or L-shaped shapes (*Cesa et al., 2017*).



FILM: flat and flexible particle, with an irregular shape(Hartmannet al., 2019).



**FOAM**: flexible and elastic particle, it shapes and softens to the touch (spongy), of different thicknesses and with an irregular outline(*Rochman et al., 2019*).



**PELLET**: similar to the sphere but tends to be larger and ovoid in shape, usually between 3 and 5 mm (*Rochman et al., 2019*).



**LINE**: particle with regular diameter along its entire length and without frayed ends relative to the fibers (*Magni et al., 2019*).



### **MPs classification by color**





- Recording of the maximum particle length.
- Identification of size ranges within which to categorize MPs: the upper limit is set at 5 mm (maximum size of MPs by definition), the lower limit is dictated by sampling methodologies and analytical techniques for characterization.
- Development of increasingly performing analytical techniques in the identification of the smallest MPs: the sizes reported in the literature are becoming more diversified incorporating a wider range of size classes.
- There is a lack of harmonization in the definition of size classes: a major obstacle to comparing studies.

| classification           |    | large mi-<br>croplastics | microplastics     |                 |             |            |           |          |  |
|--------------------------|----|--------------------------|-------------------|-----------------|-------------|------------|-----------|----------|--|
| particle size<br>classes | μm | 5 000 to 1 000           | < 1 000 to<br>500 | < 500 to<br>100 | < 100 to 50 | < 50 to 10 | < 10 to 5 | < 5 to 1 |  |

Classificazione proposta da: ISO/DIS 24187

Principles for the analysis of microplastics present in the environment

# Physical characterization: Microscopy techniques (OM and SEM)

- Scanning electron microscopy (SEM) evaluation of the surface characteristics of the particles (texture), especially the smallest ones (even nanometric)
- **SEM-EDX**: Discrimination of surface structures of plastics and other materials can be integrated with an energy-dispersive X-ray probe to provide further information on the elemental composition of organic and inorganic species, particularly useful for environmental samples
- **Disadvantage** of SEM-EDX: expensive and requires substantial time and effort for sample preparation and examination, which limits the number of samples that can be handled in routinary analyses.



From Prajapati et al., 2021. et al., 2017. ESPR



From Wang et al., 2017. STOTEN



# **Physical characterization: Light-Scattering Technique (DLS)**

- Multiple methods apply the scattering of laser light on particles to obtain information on physical properties like particle size and particle-size distribution in suspensions and emulsions.
- Dynamic light scattering (DLS), the most widely used, measures particle sizes in the range from 1 nm to 3 mm based on the fluctuation of intensity of a laser beam that passes the suspension:
   the particles are illuminated with a monochromatic and coherent light source (laser) and the intensity variations of the

scattered light are measured as a function of time: at the same temperature and viscosity, the 'small' particles move rapidly – creating variations scattering intensity – while 'large' particles move more slowly – creating slow intensity variations.

• **Disadavantage**: does NOT allow to distinguish MPs from other particles: MPs can be measured only if previously isolated in a rigorous manner from all other organic/inorganic particles.





# Chemical characterization: Spectroscopy Methods (FTIR e Raman)

- They are the most common approaches in the chemical identification of microplastics: based on the energy absorption by characteristic functional groups of polymer particles, resulting in a vibrational spectrum which is unique for every polymer type.
- Nondestructive techniques: they allow multiple analyses on the same sample and can be coupled with other methodologies to obtain additional and complementary information on the composition of plastic polymers.
- In coupling the spectrometer (FTIR or Raman) to a microscope, small microplastics are measurable through the "micro"-spectroscopy (μ-FTIR and μ-Raman): μ-Raman spectroscopy can characterize microplastic samples higher than 1 μm, while μ-FTIR spectroscopy could identify microparticles higher than 10–20 μm
- Point-wise measurements: Require manual isolation of selected potential MPs by visual examination. Time-consuming.
- Focal plane array (FPA)-based FTIR imaging: allows for detailed and unbiased high-throughput analysis of total MPs on a sample filter. This technique enables the simultaneous recording of several thousand spectra within an area with a single measurement and thus the generation of chemical images. **Disadvantages:** extended processing time to map an entire filter (9 h) to scan one filter paper, refractive errors during measurement of irregularly shaped MPs, lack of information on associated organic additives to MPs and overlap of polymer bands given by organic and inorganic contaminations that can disturb identification of particles.



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macroFT-IR e  $\mu$ FTIR



µRaman



# Chemical characterization: Spectroscopy Methods (FTIR e Raman)

• Interpretation of data is facilitated by the existence of dedicated software which significantly reduces the execution times of the analyses.

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• Chemical identification of the particles is achieved by comparing the spectrum of the sample under investigation with spectra of known polymers by matching them to spectral libraries using database comparison algorithms.



# Chemical characterization: Spectroscopy Methods (FTIR e Raman)

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BEWARE OF FALSE POSITIVES...never trust reference libraries completely...



It is important to implement commercial libraries that often derive from the characterization of pure polymers, with spectra acquired from environmental sample and of non-synthetic materials....



# **Chemical characterization: Thermoanalytical Methods (Py-GC-MS)**

- **Pyrolytic gas chromatography in combination with mass spectrometry** (Py-GC-MS) can be used to assess the chemical composition of potential microplastic particles by analyzing their thermal degradation products.
- In following a pyrolytic process, decomposition products characteristic of each polymer are trapped on a solidphase adsorbent and thermally desorbed. Volatile compounds are then separated by gas chromatography and identified by mass spectrometry
- The pyrolysis of plastic polymers results in characteristic pyrograms, which facilitate the polymer identification by comparing combustion products with reference pyrograms of known virgin-polymer samples.
- Advantages:
- individual sorting of particles is not needed
- contrarily to Raman or FTIR technique, which only investigates the surface of a particle, Py-GC-MS allows the analysis of the whole particle, enabling to simultaneously identify polymer types and associated organic plastic additives.
- Disadvantages:
- It allows to chemically identify particles but not to quantify or classify them according to shape.
- Destructive analysis: it does not allow the sample to be reused to submit it to other types of analysis.
- The amount of sample that can be analyzed (e.g., 0.35–7 mg).











# Risk considerations associated with Microplastics in water and in environment Università Politecnica delle Marche



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### **Microplastics in the Integrated Water Service**





Microplastics are present in the environment and in water

Humans could be exposed to microplastics through ingestion of contaminated food and water, inhalation of air, and skin contact.

The potential toxicological effects of microplastics are still unknown

Microplastics are classified as emerging contaminants (CECs)



### **Microplastics in the Integrated Water Service**





#### > ACCUMULATION THROUGH THE FOOD CHAIN

The effect on human health is still unknown, but plastics often contain additives, such as stabilizers or flame retardants, and other potentially toxic chemicals that can be harmful to the animal or human who ingests them.

https://www.europarl.europa.eu/news/en/headlines/society/20181116ST O19217/microplastics-sources-effects-and-solutions



### **Microplastics in the Integrated Water Service**





# Water and Waste Environmental Engineering

#### IMPACT ON THE ENVIRONMENT DUE TO THE PRODUCTION

The 2016 Ellen MacArthur Foundation report finds that 390 million tons of carbon were emitted as part of plastic production (and consumption) in 2012, or about 1% of the carbon balance, and that this share should grow up to 15% by 2050 as a result of the expected increase in demand.

#### IMPACT ON THE HEALTH DUE TO THE RELEASE

There are concerns that some additives used in plastics, such as bisphenol A (BPA) or some phthalates used in polyvinyl chloride (PVC), may have adverse effects on human health and the environment, mainly due to the release of these substances from wastes into the environment and subsequently transmitted to wildlife, with uncertainties about long-term exposure and cumulative effects. Additionally, persistent organic pollutants can attach to plastics in water and enter the food chain via marine life.





### **Conventional, non-conventional and emerging pollutants**









#### • European Directive 2008/105/CE

Defines environmental quality standards (EQS) of priority list substances

EQS: the concentration of a particular pollutant or group of pollutants in water, sediment and biota which must not be exceeded, in order to protect human and environmental health.

#### • European Directive 2009/90/CE

Defines the monitoring and analysis technical specifications (measurement uncertainty, limits of quantification, ISO17025 obligation)

• New European Directive 2013/39/CE



### **Microplastics in national and European legislation**





- News Directive 2013/39/EC
  - EQS for 12 new priority substances (Bifenox, Terbutrin, Cybutrin, Cypermethrin, Dichlorvos, Aclonifen, Hexabromo-cyclodecane, Quinoxifen, Dicofol, PFOS, Heptachlor/Eptachlor epoxide, "Dioxins")
  - EQS modified for some existing substances
  - EQS biota new priority substances and existing substances
  - Inland water EQS based on the bioavailable fraction for nickel and lead (Annex II)
  - Specific provisions for UBPT (ubiquitous, persistent, bioaccumulation and toxicity) - (art 8 bis) - Reduction of monitoring and Separate Classification
  - New priority hazardous substances (Annex I)
- Provisions for drugs (strategy and monitoring art. 8)





Emerging contaminants

Substances that are not regulated
..... are not monitored
..... there is no data available
.....there are no data to propose prioritization
..so there is no data to assess the risk

 CHECKLIST for substances for which monitoring data needs to be collected



### **New Drinking Water Directive**



DIRECTIVE (EU) 2020/2184 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 16 December 2020 concerning the quality of water intended for human consumption

L 435/1



To respond to growing public concern about the human health effects of emerging compounds, such as endocrine disruptors, pharmaceuticals and microplastics, present in water intended for human consumption and to address the issue of new emerging compounds in the supply chain, a checklist mechanism should be introduced in this Directive. The checklist mechanism will make it possible to respond dynamically and flexibly to growing concerns. It will also make it possible to follow up on new knowledge on the importance of these emerging compounds to human health and on the most appropriate monitoring methods and approaches.





DIRECTIVE (EU) 2020/2184 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 16 December 2020 concerning the quality of water intended for human consumption

A type of approach was introduced in the Directive, based on the drawing up a "checklist" of substances (for which limits or threshold values have not been defined) with the aim of approaching the problem in a flexible and dynamic way.



The Directive subordinates the inclusion of the parameter into the checklist to the definition of a methodology for measuring microplastics that the European Commission must issue by 12 January 2024.



The transmission of a report (and subsequent possible updates) by the Commission to the European Parliament and the Council (by 12 January 2029) on the potential threats due to microplastics for sources of water intended for human consumption, as well as on the related potential health risks.





DIRECTIVE (EU) 2020/2184 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 16 December 2020 concerning the quality of water intended for human consumption

In order to adapt this Directive to technical and scientific progress, the power should be delegated to the Commission to [...] adopt a methodology for measuring microplastics.

By 12 January 2024, the Commission adopt delegated acts in accordance with Article 21 to supplement this Directive by adopting a methodology for measuring microplastics to include them into the checklist referred to in paragraph 8 of this Article once the conditions referred to in this paragraph are met.

The Commission submit to the European Parliament and the Council by 12 January 2029 and subsequently, if appropriate, a report on the potential threats to sources of water intended for human consumption due to microplastics, pharmaceuticals and, if necessary, other pollutants causing new concern, as well as its potential health risks.



# Water and Waste Environmental Engineering

### **New Drinking Water Directive**

DIRECTIVE (EU) 2020/2184 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 16 December 2020 concerning the quality of water intended for human consumption

In the case surface waters are used as water intended for human consumption, Member States should be pay particular attention to **microplastics** and endocrine distrupting compounds in their **risk assessment**, and, where relevant, they should also require water suppliers to **monitor and, if necessary, treat** these and other parameters included in the checklist **if they are considered to be a potential danger to human health**.

Based on basin risk assessment for abstraction points, management measure should be adopted to prevent or control the identified risks in order to safeguard the quality of water intended for human consumption.







DIRECTIVE (EU) 2020/2184 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 16 December 2020 concerning the quality of water intended for human consumption

Better knowledge of relevant information and increased **transparency** should aim at strengthening **citizens' trust** in water supply and water services and should lead to an **increase in the use of tap water** as water intended for human consumption, which could contribute to the **reduction of plastic waste and use and greenhouse gas emissions** and have **a positive impact on climate change mitigation and the environment** as a whole.

The Commission adopts implementing acts to **establish and update a checklist for substances or compounds of public or scientific health concern ("checklist")**, such as pharmaceuticals, interfering compounds endocrines and microplastics.



### Water safety plan – general scheme







### Water safety plan – risk analysis

| Risk = Probability * Severity  |   |   |  |
|--|---|---|--|
|  |   | Severity of the consequences  |  |
| Probability  | s<br>d<br>h<br>t<br>l<br>'OMS c                                 | severity or intensity of the impact that the<br>danger occurrence may have, primarily for<br>human health, but also for the quality of<br>the service in terms of sanitation quality of<br>the water supplied, organoleptic<br>characteristics, quantity supplied, continuity |  |
| hazard or dangerous event can Gravità delle conseguenze  | 0   | of supply , etc   |  |
| conceivably occur, particularly<br>considering hazards that have<br>occurred in the past and their<br>probability of recurrence over | Molto grave<br>(effetti gravi<br>/catastrofici<br>sulla salute) |   |  |
| time; it must also predict the probability of risks and events that did not occur.   | 5   |   |  |
| Improbabile<br>(es. 1 volta all'anno) 2 4 6 8  | 10  |   |  |
| Moderatamente<br>probabile36912(es. 1 volta al mese)   | 15  |   |  |
| Probabile<br>(es. 1 volta a 4 8 12 16<br>settimana)  | 20  |   |  |
| Quasi certo<br>(es. 1 volta al giorno)5101520  | 25  | www.lifebluelakes.e   |  |
| Legenda del rischio  |   |   |  |
| VVV==         Grado         <6         6-9         10-15         >15   |   |   |  |

No microplastics, just waves.

## **Microplastics in national and European legislation**



From drinking water to reuse water





### Wastewater reuse



Guidelines to support the application of Regulation (EU) 2020/741 laying down minimum requirements for water reuse

| 5.8.2022 | IT                | Gazzetta ufficiale dell'Unione europea  | C 298/1 |
|----------|-------------------|---|---------|
|          |                   |   |         |
|          |                   | II  |         |
|          |                   | (Comunicazioni)   |         |
|          | COMUNICAZ         | ZIONI PROVENIENTI DALLE ISTITUZIONI, DAGLI ORGAN<br>DAGLI ORGANISMI DELL'UNIONE EUROPEA                     | NI E    |
|          |                   | COMMISSIONE EUROPEA   |         |
|          |                   | COMUNICAZIONE DELLA COMMISSIONE   |         |
|          | Orientamenti a so | stegno dell'applicazione del regolamento (UE) 2020/741 recante presc<br>minime per il riutilizzo dell'acqua | rizioni |
|          |                   | (2022/C 298/01)   |         |

Some pollutants which are not yet regulated and cannot be found in the directives and regulations (e.g. **microplastics** or some **compounds of emerging interest**) could be **added to the hazard list, if the risk to human and animal health or environment is supported by scientific evidence and it is demonstrated that these contaminants come from the water reuse system** and not from other sources. Risk assessment could also **identify the source** of these contaminants, e.g. for the presence of particular industries, and plan any preventive measures.



### Wastewater reuse



#### ANNEX II to REGULATION (EU) 2020/741 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 25 May 2020 laying down minimum requirements for water reuse

#### MAIN ELEMENTS OF RISK MANAGEMENT

- Description of the whole water reuse system
- Identification of the **bodies** involved, roles and responsibilities
- List of possible hazards and hazardous events
- Identification of populations at risk and routes of exposure
- Risk assessment for the environment and for human and animal health

#### ADDITIONAL REQUIREMENTS

- → Heavy metals
- Pesticides
- → Disinfection **by-products**
- → Drug
- → Other substances of growing concern (micropollutants,

microplastics, ...)

 Resistance to antimicrobial agents

#### **PREVENTION MEASURES**

- → Access control
- Additional measures of disinfection or elimination of pollutants
- → Irrigation technologies
- → Specific requirements for sprinkler irrigation Specific requirements for agricultural fields
- → Suppression of pathogens before harvesting
- → Minimum safety **distances**
- → Signage
- Quality control systems and procedures, water monitoring, maintenance programs
- → Environmental **monitoring systems**
- Management of incidents and emergency situations
- **Coordination** between the different actors

RISK ANALYSIS







|                          | OPERATIONAL ASPECTS  | REGULATORY ASPECTS   |  |  |
|--------------------------|--|--|--|--|
| STATE OF<br>ART          | Microplastics present in the environment and in water.             | Microplastics included in the "checklists"<br>and their presence must be evaluated in the<br>risk analysis |  |  |
| MATERIALS AND<br>METHODS | Microplastics sampling campaign in drinking water treatment plant. | The EU must propose a standardized methodology for monitoring microplastics in water                       |  |  |
| RESULTS                  | Results of the sampling surveys. Monitoring protocol.              | Analysis and report of the European<br>Commission  |  |  |
|                          | Monitoring of microplastics in pur                                 | ification plants and drinking water.   |  |  |

Analysis of the possible risks caused by the presence of microplastics in water.

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The <u>report of the European Environment Agency</u>, published in mid-March 2022, highlights the environmental impacts due to the dispersion of microfibres from textile products.

#### **ETC/CE** Report 1/2022: Microplastic pollution from textile consumption in Europe

**Every year 6-15 million tons of plastic,** which represent 2-4% of world production, enter the **environment** 

- > Primary microplastics
- Secondary microplastics





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European Environment Agency





# **Microplastics as emerging contaminants**

### ETC/CE Report 1/2022: Microplastic pollution from textile consumption in Europe

environmental and health impacts associated with microplastics pollution

- INGESTED BY LIVING ORGANISMS (from plankton to fish to mammals)
- INHALED IN THE INTERNAL ENVIRONMENT
- ➢ INHALED IN THE EXTERNAL ENVIRONMENT
- INGEST IN FOOD
- > ACCUMULATION THROUGH THE FOOD CHAIN
- Potentially toxic effects of the substances contained (additives, monomers, catalysts and production reaction by-products)
- High levels of exposure to microplastics induce inflammatory reactions and toxicity, and microplastics can be vectors for the spread of pathogens and microbes







### **Microplastics and risk analysis**





- significant uncertainties about the quality and extent of data on human exposure to microplastics in drinking water
  - difficulty in estimating a cause-effect relationship for microplastics

current knowledge on toxicological effects requires the acquisition of more solid scientific evidence







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Martellone et al., "Microplastiche nelle acque potabile, ISS.






#### INDIRECT TOXICITY

Surface of microplastics suitable for the absorption of persistent organic contaminants such as polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs) or pesticides and to favor the growth of bacterial biofilms, which could also contribute to the phenomenon of antibiotic resistance. Additives, such as phthalates and colorants (e.g. titanium dioxide - TiO2, cadmium pigments - Cd) to improve the properties of plastic materials.

Release into waters following degradation of plastics.

The toxicological profile of absorbable substances, additives and pathogens linked to the development of biofilms is quite well known, but **at the moment the WHO suggests a low risk of toxicity from these contaminants linked to microplastics.** 









## Presence of microplastics and nanoplastics in food, with particular focus on seafood

https://www.efsa.europa.eu/it/efsajournal/pub/4501

Microplastics found in fish mostly in the stomach and intestines

Consumers less exposed if eliminated

Greater exposure for crustaceans and bivalve molluscs, (oysters and mussels) for which the digestive tract is consumed

• Microplastics also found in other foods (honey, beer, table salt)

**POTENTIAL HAZARD**: Concentrations of polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) and residues of compounds used in packaging such as bisphenol A (BPA), which can accumulate in microplastics.

Some studies indicate that microplastics, after consumption in food, can transfer to tissues.

EVALUATE THE AVERAGE INTAKE

EFSA has estimated that one portion of mussels (225g) could contain seven micrograms of microplastic. Even if that amount of material contained the highest ever measured concentrations of PCBs or BPA, for example, it would contribute little to overall exposure to these substances: it would increase PCB exposure by less than 0.01% or exposure to BPA of less than 2%.



# WHO 2019: Microplastics in drinking water

Microplastics are not individual chemicals or well-characterized substances but are particles that vary in **shape, size and composition** 

Challenges to traditional human health risk assessment approaches



- No human or epidemiological studies on ingested microplastics were identified.
- Data from laboratory animal studies are scarce and inadequate to inform human health risk assessment of ingestion of microplastics.
- The current database of information on the toxicity (and absorption) of plastic particles is limited to a few studies using PET, PS or PE, and there are questions about the reliability of some of these studies.







Potential hazards associated with particles

Absorption studies of microplastics and nanoplastics FAO 2017 and EFSA 2016

### Absorption kinetics in the gastrointestinal tract of microplastics

- Most ingested microplastics (>90%) are probably not absorbed
- Microplastics >150 µm are probably not absorbed
- Limited absorption of smaller microplastics (≤0.3%).
- It is possible that uptake and distribution may be more significant for nanoplastics than microplastics (up to 7% for nanoplastics <0.1 μm</li>
- Limited data  $\rightarrow$  further studies are needed







## WHO 2019: Microplastics in drinking water

Potential hazards associated with particles

### MONOMERS ADDITIVES SUBSTANCES ABSORBED

| PARAMETER                    | ΙΝΤΑΚΕ           | RATIONAL                                   |
|------------------------------|------------------|--|
| Shape                        | Spherical        | Greater specific surface area and volume   |
| Dimensions                   | 150 µm           | More frequent (Mintening et al., 2019)     |
| Density                      | 2.3 g/cm3        | Highest Density Detected (Polyester)       |
| Number of particles in water | 10.4 particles/L | Conservative estimate from literature data |

CONSERVATIVE SCENARIO: Estimated daily intakes 0.1 - 2 µg/day

| Chemical*                       | opper bound<br>concentration in<br>microplastic (µg/g) | Maximum<br>daily intake<br>(ng/kg bw/day) <sup>b</sup> | Point of departure<br>(µg/kg bw/day) | Margin of exposure<br>(MOE) | Adequacy of MUE                   | Conclusion        |
|---------------------------------|--|--|--------------------------------------|-----------------------------|-----------------------------------|-------------------|
| Bisphenol A                     | 0.7297   | 0.001  | 609                                  | 5.9 × 10 <sup>8</sup>       | MOE of at least 100               | No safety concern |
| Cadmium                         | 3390   | 5.0  | 0.8                                  | $1.7 \times 10^{2}$         | MOE of at least 10 <sup>c</sup>   | No safety concern |
| Chlordane                       | 0.0144   | 0.00002  | 50                                   | 2.5×10°                     | MOE of at least 100               | No safety concern |
| Di(2-ethylhexyl)phthalate       | 0.0699   | 0.0001   | 2500                                 | 2.5×10 <sup>10</sup>        | MOE of at least 100               | No safety concern |
| Dichlorodiphenyltrichloroethane | 7.1  | 0.0001   | 1000                                 | 1.0 × 10 <sup>8</sup>       | MOE of at least 100               | No safety concern |
| Hexachlorobenzene               | 0.0587   | 0.00002  | 50                                   | 6.0 × 10 <sup>8</sup>       | MOE of at least 100               | No safety concern |
| Polyaromatic hydrocarbons       | 119  | 0.06   | 100                                  | 6.0 × 10 <sup>5</sup>       | MOE of at least 10 000            | No safety concern |
| PBDEs                           | 9.9  | 0.01   | 100                                  | 7.2×10 <sup>6</sup>         | MOE of at least 100               | No safety concern |
| PCBs                            | 18.7   | 0.03   | 5                                    | 1.9×10 <sup>5</sup>         | MOE of at least 1000 <sup>d</sup> | No safety concern |

From the available data, no significant risks are identified  $\rightarrow$  To be investigated on a case-by-case basis based on the individual active compounds







# WHO 2019: Microplastics in drinking water

Potential hazards associated with particles

limited data on the distribution of biofilms (both in suspension and in pipes  $\rightarrow$  Different effects to be explored) associated with microplastics in drinking water

no evidence suggesting a risk to human health from biofilms associated with microplastics in drinking water.

Some microplastics can detach from **materials within water treatment and distribution systems**  $\rightarrow$  very small fraction of the surface area and biofilms generated on the starting materials.

Additionally, in many countries, plastics and materials used in drinking water systems are subject to standards, including **testing to demonstrate that they do not support microbial growth** (WHO, 2014).











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### Thank you for the attention!



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# Examples and case studies Università Politecnica delle Marche



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## **Microplastics in the Integrated Water Service**



Plants receive the microplastics contained in the influent from a wide variety of potential sources:

- Textile laundry fibres
- Plastics in personal care products
- Fragmentation of larger plastic objects
- Plastics in industrial waste
- Tire and road wear particles
- Runoff waters

Wastewater treatment plants are often regarded as a *source of microplastics* for the environment.

However, most of the microplastics released do not originate from internal plant processes. For this reason, the most appropriate definition is a *release path* 



Routes of entry for microplastics leading to drinking water sources. Eerkes-Medrano 2019

The reduction of microplastics in the effluent depends to some extent on the treatment processes employed.

Removal efficiencies of up to 99% have been reported in the literature.

Despite the low concentrations of microplastics in the effluent, the large volumes treated by the plants result in substantial **cumulative releases over time.** 

Mass load = Concentration \* Volume

During the treatment processes, the microplastics are transferred to the sludge which, based on the type of final delivery, could give rise to release phenomena based on the final delivery point.





#### From measures to mass balances



N.B. Transform hours into minutes!



#### From measures to mass balances

| Sampling point | Volume (l) | n. Microplastic Particles<br>(MPPs) in the sample | n. Natural Microfibres<br>(MNFs)    | n. Plastic Microfibres<br>(MPFs) | n. Microplastics (MPs) | MPS concentration<br>[MPs/m3] |      |
|----------------|------------|---|-------------------------------------|----------------------------------|------------------------|-------------------------------|------|
| Influent       | 972        | 10  | 2                                   | 4                                | 14                     | 14.4                          |      |
| Out ozone      | 1037       | 6   | 3                                   | 2                                | 8                      | 7.7                           |      |
| Out filters    | 1000       | 3   | X                                   | 0                                | 3                      | 3.0                           |      |
| Effluent       | 970        | 1   | 2                                   | 1                                | 2                      | 2.1                           |      |
| Distribution   | 1000       | 3   | 1                                   | 0                                | 3                      | 3.0                           |      |
| Count I        | Microplo   | astics (MPs) =                                    | Plastic Micro                       | particles(MPI                    | Ps) + Plastic N        | Aicrofibres (.                | MPFs |
|                | Conce      | entration MPs                                     | $\left(\frac{MPs}{m3}\right) = n.M$ | Ps counted/V                     | Volume (l)/10          | $00 \ (\frac{l}{m3})$         |      |



■ film ■ fragment ■ line ■ fiber

#### From measures to mass balances

| SHAPE          |         |                |                           |                            |                       |              |
|----------------|---------|----------------|---------------------------|----------------------------|-----------------------|--------------|
| Sampling point | n. MPs  | film           | fragment                  | line                       |                       | fiber        |
| Influent       | 14      |                | 8                         | 2                          |                       | 4            |
| Out ozone      | 8       | 1              | 5                         |                            |                       | 2            |
| Out filters    | 3       |                | 3                         |                            |                       |              |
| Effluent       | 2       |                | 1                         |                            |                       | 1            |
| Distribution   | 3       |                | 3                         |                            |                       |              |
|                |         |                |                           |                            |                       |              |
| DIMENSION      |         |                |                           |                            |                       |              |
| Sampling point | nn. MPs | 5 -1 mm        | 1-0.5 mm                  | 0.5-0.1 mm                 | C                     | ).1-0.02 mm  |
| Influent       | 14      | 5              | 5                         | 4                          |                       |              |
| Out ozone      | 8       | 2              | 5                         | 1                          |                       |              |
| Out filters    | 3       |                | 1                         | 2                          |                       |              |
| Effluent       | 2       |                |                           | 2                          |                       |              |
| Distribution   | 3       |                | 2                         | 1                          |                       |              |
|                |         |                |                           |                            |                       |              |
| TYPOLOGY       |         |                |                           |                            |                       |              |
| Sampling point | n. MPs  | polyester poly | ester resin polypropylene | polyvinylidene<br>fluoride | polyvinyl<br>chloride | polyethylene |
| Influent       | 14      | 5              | 5                         | 4                          |                       |              |
| Out ozone      | 8       | 1              | 1                         | 3                          | 2                     | 1            |
| Out filters    | 3       | 1              | 2                         |                            |                       |              |
| Effluent       | 2       |                | 1                         |                            |                       | 1            |
| Distribution   | 3       | 2              | 1                         |                            |                       |              |

Influent Out ozonation Out filtration Effluent Distribution



#### From measures to mass balances



#### EVALUATE THE CONCENTRATION VARIATIONS IN THE DIFFERENT TREATMENT UNITS





From measures to mass balances

Es: Drinking water treatment plant with Qin = 100 l/sec

 $Flowrate \ (m3/d) = Flowrate \ \left(\frac{l}{s}\right) * \frac{1m3}{1000 \, l} * \frac{60 \, sec}{1 \, min} * \frac{60 \, min}{1 \, h} * \frac{24 \, h}{1 \, d}$ 

| Sampling point | Concentration<br>MPs/m3 | Load<br>MPs/d |
|----------------|-------------------------|---------------|
| Influent       | 14.4                    | 124416        |
| Out ozone      | 7.7                     | 66528         |
| Out filters    | 3.0                     | 25920         |
| Effluent       | 2.1                     | 18144         |
| Distribution   | 3.0                     | 25920         |



Pittura et al., 2021 "Microplastics in real wastewater treatment schemes: Comparative assessment and relevant inhibition effects on anaerobic processes" Chemosphere, 262, 128415 Chemosphere 262 (2021) 128415



Microplastics in real wastewater treatment schemes: Comparative assessment and relevant inhibition effects on anaerobic processes



Lucia Pittura <sup>a</sup>, Alessia Foglia <sup>b, \*\*</sup>, Çağrı Akyol <sup>b, \*</sup>, Giulia Cipolletta <sup>b</sup>, Maura Benedetti <sup>a</sup>, Francesco Regoli <sup>a</sup>, Anna Laura Eusebi <sup>b</sup>, Simona Sabbatini <sup>b</sup>, Linda Y. Tseng <sup>c</sup>, Evina Katsou <sup>d</sup>, Stefania Gorbi <sup>a</sup>, Francesco Fatone <sup>b</sup>

<sup>a</sup> Department of Life and Environmental Sciences, Marche Polytechnic University, 60131, Ancona, Italy

<sup>b</sup> Department of Science and Engineering of Materials, Environment and Urban Planning-SIMAU, Marche Polytechnic University, 60131, Ancona, Italy

<sup>c</sup> Department of Environmental Studies and Physics, Colgate University, 13346 New York, United States

<sup>d</sup> Department of Civil Engineering and Environmental Engineering, Institute of Environment, Health and Societies, Brunel University London, Middlesex, UB8 3PH Uxbridge, United Kingdom

IMPIANTO DI TRATTAMENTO IN PIENA SCALA





Presence and removal of microplastics in conventional large-scale and innovative pilot-scale treatment schemes of a municipal wastewater treatment plant





ACTIVATED SLUDGE TAS SIMPLETION SCREEN and GRE PRE-TREATED P CENTRIFUGU DEWAT SLUD **MPs sampling** MPs Effect on the processes N2 GAS SPARGING Biogas MPs processing **MPs characterization Visual sorting** 

Conventional Full-scale WWTP and pilot scale AnMBR



## Wastewater processing for MPs analysis



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Presence and removal of microplastics in conventional large-scale and innovative pilot-scale treatment schemes of a municipal wastewater treatment plant

|   | Analyzed samples  | MPs concentration                        |     |               |
|---|---|--|-----|---------------|
| Full scale WWTP<br>based on<br>conventional<br>active sludge<br>process (CAS) | Influent wastewater                                       | 3.1 MPs/L                                | ן ן |               |
|   | Wastewater after primary settling                         | 1.9 MPs/L                                |     |               |
|   | Wastewater after biological treatment (aerobic condition) | 0.8 MPs/L                                |     | <b>8</b><br>C |
|   | Wastewater in the final effluent                          | 0.5 MPs/L                                |     |               |
|   | Secondary sewage sludge                                   | 5.3 MPs/g (79% particles and 21% fibers) |     |               |
| Innovative<br>AnMBR system in<br>the Pilot Hall                               | Wastewater after UASB reactor                             | 1.7 MPs/L                                |     | 9             |
|   | Wastewater after ultrafiltration unit                     | 0.2 MPs/L                                |     | 11            |
|   | UASB sludge   | 1 MPs/L (10% particles and 90% fibers)   |     |               |

ALONG THE TREATMENT LINE, MPS DECREASE AND ARE RETAINED IN SLUDGE >> ATTENTION TO SLUDGE DISPOSAL/ VALORIZATION!

#### **86%** REMOVAL FROM COVENTIONAL WATER LINE

**94%** REMOVAL FROM INNOVATIVE WATER LINE

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## *Efficiency in restraining microplastics: conventional vs innovative system*

| Treatment Steps                      | n°MPs/day | MP<br>remova | 9s<br>nl (%) |
|--------------------------------------|-----------|--------------|--------------|
| INFLUENT                             | 7.73E+07  |              |              |
| EFFLUENT AFTER PRIMARY SETTLING      | 4.14E+07  | 45           |              |
| EFFLUENT AFTER BIOLOGICAL TREATMENT  | 1.56E+07  | 62 79 82     |              |
| EFFLUENT AFTER CHEMICAL DISINFECTION | 1.33E+07  | 15           |              |
| INFLUENT                             | 7.73E+07  |              |              |
| EFFLUENT AFTER UASB                  | 3.47E+07  | 55           | 95           |
| EFFLUENT AFTER AnMBR                 | 3.91E+06  | 40           |              |

SYSTEM REMOVAL EFFICIENCY UASB ANMBR: 94% > SYSTEM REMOVAL EFFICIENCY CAS: 86%



#### **Microplastics characterisation**





SHAPE

Polycaprolactone Fluorosilicone Polvester based copolymer □ Styrene-butadiene-styrene copolymer Styrene-allyl alchol copolymer Ethylene-propylene-EVA copolymer Styrene-ethylene vinyl acetate copolymer Ethylene-vinyl acetate copolymer Polyvinyl stearate Acrylic paint Polyurethane-acrylic resin Styrene-butyl methacrylate copolymer Polyacrylamide Paraffin wax Alkyd resin Styrenic resin Polycarbonate ■ Silicone Polystyrene



Polyester based elastomerPolystyrene based copolymer

- PVC PVOH PE copolymer
- Styrene-ethylene-butylene copolymer
- Ethylene-propylene-copolymer
- Polyethylene-ethylene-vinyl acetate blend
- Polyvinyl acetate
- Acrylic rubber
- Alkyl acrylate-copolymer
- Isoprene-polyacrylic copolymer
- Ethylene-ethyl acrylate copolymer
- PolyacrylatePolyester epoxide resin
- Epoxy resin
- PolytetrafluoroethylenePolyamide
- Polyurethane
  Polyesters

Polyethylene





DIMENSIONS

#### THE MOST FREQUENT MPS ARE POLYETHYLENE AND POLYPROPYLENE

ALONG THE SUPPLY CHAIN THE CONCENTRATIONS DECREASE AND THEY ARE FOUND IN THE SLUDGE >> ATTENTION TO SLUDGE DISPOSAL / VALORIZATION!



Legends: PRE-TREAT IN (pre-treated effluent), I EFF (primary effluent), II EFF (secondary effluent), FINAL EFF (final effluent), AerEXC SLUD (excess sludge), WAS (waste activated sludge), DEWAT SLUD (dewatered sludge).

Polypropylene



#### Analysis of inhibitory effects of MPs on anaerobic biomass

#### LAB TESTS

|                           | BIOMASSS  | <b>BIOLOGICAL PROCESS</b> | BATCH TESTS  | MONITORING PARAMETER |                 |
|---------------------------|-----------|---------------------------|--------------|----------------------|-----------------|
| AEROBIC ACTIVATED SLUDGE  | suspended | aerobic                   | F/M=0.4-0.7  | SOUR                 | mg/gVSS/h       |
| ANAEROBIC GRANULAR SLUDGE | suspended | anaerobic                 | COD/VSS=0.25 | SGP                  | g biogas/gVSS/d |
| ANAEROBIC SLUDGE          | suspended | anaerobic                 | COD/VSS=0.25 | SGP                  | g biogas/gVSS/d |

#### Anaerobic granular sludge



#### Anaerobic flocculant sludge







(A) Schematic diagram of the mass balance of the spiked PP-MPs in the pilot-scale UASB reactor



Analysis of inhibitory effects of MPs on anaerobic biomass







(B) Variation in CH<sub>4</sub> production rate of the pilot-scale UASB reactor upon the PP-MPs spiking. 1. and 2. phases refer to the spiking of  $15\pm3$  PP-MPs gTS<sup>-1</sup> and  $50\pm5$  PP-MPs gTS<sup>-1</sup>, respectively.

Water and Waste Environmental Engineering



# *Effect of tertiary disinfection treatments on PE microplastics: dose-dependent effects of oxidants*

- Changes in absorbance at 1470 and 1540 cm-1 related to the NO3 group.
- Treatments with doses of 0.4 mgO3\*h/L/MPs and 2 mLNaOCI\*h/m3/MPs reduce the absorbance ratio by 68% and 64%, respectively.
- The toxicity before and after the oxidative actions is still being evaluated with specific targeted experimental activities.

- Preparatory studies for the definition of the MPs sampling method and of the extraction protocol for the detection and characterization of MPs in drinking water, wastewater and sludge;
- The presence and removal of MPs in WWTPs were evaluated, both for conventional and innovative treatment processes, resulting in a crucial role of ultrafiltration units (94%);
- The effect of MPs on biological processes has been studied, demonstrating an inhibition of methanogenic activity (58%) at a high concentration of MPs.
- The effect of high-dose tertiary treatments on MPs was studied resulting in a dependent effect between dosages and MPs composition by changing the chemical structure of the NO3 group.





# Innovation activities for water infrastructures in the Lake Garda basin (Combined sewer overflows)



HORIZON 2020 INTCATCH (<u>http://www.intcatch.eu/</u>) Integration of Compact Solutions for the treatment of flood overflows

2020

**Compact Solutions in the plants**: (Lazise - Lago di Garda) Solids Removal, Rapid Adsorption, UV Disinfection (Lazise - Lago di Garda, IT)





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# Innovation activities for water infrastructures in the Lake Garda basin (Combined sewer overflows)



#### Villa Bagatta lifting station

- The station pumps approximately 22,000 m3/day
- Solutions for CSO treatment
- The treated CSO is discharged far from the shore, from a sub-lake pipe
- In 2016-2017 only 36 CSO events with an average discharged volume of 1,800 m3 and a duration of 3.35 hours

Parameter analysed: COD, TSS, TP, TN, E. coli and coliforms, PPCPs and emerging contaminants, such as microplastics

## **Combined sewer overflows**

- They contain untreated waste from human and industrial activities, toxic materials and debris, as well as rainwater
- One of the main problems of the countries that have the Combined Sewage System
- Numbers:
- 650,000 CSOs in Europe (EurEau)
- 20% of surface waters are at serious risk of pollution (EU Water Framework Directive)
- 11 CSOs on the eastern shore of Lake Garda







## Innovation activities for water infrastructures in the Lake Garda basin



Critical analysis with evaluation of peak and minimum scenarios, both long and short term (e.g. high vs low tourist season, weekend vs working days) for system performance optimization



## SAMPLING OF EMERGING CONTAMINANTS IN THE PLANT: MPs



Sampling points

- $\rightarrow$  Pre screening: 25 L grab sample
- $\rightarrow$  Post degritting: 25 L grab sample
- $\rightarrow$ Out (post disinfection): 25 L grab sample

Sampling dates : 1/08/2018, 24/08/2018, 28/09/2018





## Innovation activities for water infrastructures in the Lake Garda basin



Critical analysis with evaluation of peak and minimum scenarios, both long and short term (e.g. high vs low tourist season, weekend vs working days) for system performance optimization

## SAMPLING OF EMERGING CONTAMINANTS IN THE PLANT: MPs: 01/08/2018

| WATER STREAM          | N. MPs/m3* | N. MPs/h | N. MPs/d | % rimozione relativa | % rimozione assoluta |
|-----------------------|------------|----------|----------|----------------------|----------------------|
| PRE SCREENING         | 400        | 390933   | 9.38E+06 |                      |                      |
| POST DEGRITTING       | 120        | 117280   | 2.81E+06 | 70                   |                      |
| OUT POST DISINFECTION | 40         | 39093    | 9.38E+05 | 67                   | 90                   |

\*Mainly Polyethylene



## Innovation activities for water infrastructures in the Lake Garda basin



Critical analysis with evaluation of peak and minimum scenarios, both long and short term (e.g. high vs low tourist season, weekend vs working days) for system performance optimization

## SAMPLING OF EMERGING CONTAMINANTS IN THE PLANT: MPs: 24/08/2018

|                       |            |          |          | % RELATIVE | % ABSOLUTE |
|-----------------------|------------|----------|----------|------------|------------|
| WATER STREAM          | N. MPs/m3* | N. MPs/h | N. MPs/d | REMOVAL    | REMOVAL    |
|                       |            |          |          |            |            |
| PRE SCREENING         | 800        | 781866   | 1.88E+07 |            |            |
|                       |            |          |          |            |            |
| POST DEGRITTING       | 80         | 78186    | 1.88E+06 | 90         |            |
|                       |            | 20002    |          | -          |            |
| OUT POST DISINFECTION | 40         | 39093    | 9.38E+05 | 50         | 9          |

\*Mainly polyvinyl stearate and polymer rubber → from tyres, pipes, shoe soles and gaskets





Microplastics in drinking water treatment plants – case study 1

Analysis of MPs  $\geq$  1 µm in raw and treated water from two treatment plants which are both located on the same river, but where the local water quality and applied treatment technology differ. Sampling was conducted three times in one day, taking 2 L of water on each occasion.



Upstream – DWTP Milence Investigating microplastics (MPs) at two drinking water treatment plants Raw Drinking (DWTP) within a river catchment water water Evaluation of number, size, shape, material composition 23 14 MPs L MPs L Coagulation Ozonation Deep Drinking Raw bed + GAC Ellipticawater water Downstream – DWTP Plzeň filtration sedimentation filtration 1296 243 151 497 149 MPs L<sup>1</sup> MPs L MPs L MPs I MPs L M. Pivokonský et al. (2020)



#### **Microplastics in drinking water treatment plants – case study 1**



M. Pivokonský et al. / Science of the Total Environment 741 (2020)

High concentrations found, mainly due to:

- Mesh (1 μm)
- Representativeness of the sample (2-6 L)



#### **Microplastics in drinking water treatment plants – case study 1**



#### CUMULATIVE REMOVAL EFFICIENCY



M. Pivokonský et al. / Science of the Total Environment 741 (2020)

| Treatment                              | Removal efficiency [%] |
|--|------------------------|
| coagulation-flocculation-sedimentation | 62%                    |
| Deep bed filtration                    | 20%                    |
| Granular active carbon filtration      | 6%                     |

Removal efficiencies range from 40% for the first plant (basic configuration) up to 88% for the complex plant



#### Microplastics in drinking water treatment plants – case study 2





#### Microplastics in drinking water treatment plants - case study 3



#### DWTP

Flowrate: 150000 m3/d Captation from surface waters Volumes: from 1L (5–20 µm) to 100–200L (20 µm-5 mm)

Abundance of granular MPs (n/L) in raw water and treated water of the DWTP.

| Category                              | Characters of   | Raw water  | Treated water   | Removal   |
|---------------------------------------|---|--|---|---|
|                                       | MPs   | Average(n/L)   | Average(n/L)  | %   |
| Size<br>Major material<br>composition | 1-5 mm<br>500 μm-1 mm<br>100-500 μm<br>20-100 μm<br>10-20 μm<br>5-10 μm<br>PP <sup>a</sup><br>PE <sup>b</sup><br>PVC <sup>c</sup><br>PA <sup>d</sup><br>VINYL <sup>e</sup><br>VINYCN <sup>f</sup> | $\begin{array}{c} 0.10 \pm 0.09 \\ 0.13 \pm 0.05 \\ 2.88 \pm 1.04 \\ 41.80 \pm 6.6 \\ 933.34 \pm 49.01 \\ 2760.14 \pm 408.27 \\ 917.27 \pm 67.4 \\ 500.58 \pm 32.33 \\ 479.57 \pm 54.22 \\ 243.39 \pm 64.22 \\ 362.24 \pm 70.28 \\ 566.80 \pm 57.89 \end{array}$ | $\begin{array}{c} 0\\ 0.02 \pm 0.01\\ 0.07 \pm 0.06\\ 0.31 \pm 0.1\\ 316.03 \pm 82.84\\ 379.24 \pm 51.25\\ 208.73 \pm 50.49\\ 176.33 \pm 10.46\\ 69.85 \pm 32.29\\ 93.36 \pm 50.53\\ 20.77 \pm 31.49\\ 40.61 \pm 35.27\\ \end{array}$ | 100.00<br>88.10<br>97.74<br>99.21<br>65.82<br>86.24<br>77.12<br>64.70<br>85.15<br>59.25<br>95.05<br>92.39 |
| Total                                 |   | 3738.36 ± 461.29   | 695.66 ± 39.32  | 81.18   |




### **Oxidation Treatment**

- Negative removal efficiencies
- Better removal effect on smaller sizes (5-10 μm) than 10-20 μm. the smallest MPs could remain attached to the biofilm or adhere to the microorganisms

## Physical treatments:

higher removal efficiencies

- BAC (Biological Activated Carbon)
- Sand filtration

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**Microplastics in wastewater treatment plant** 

- 4 WWTPs with different technologies
- Customized in situ filtration device with mesh sizes of 300, 100 and 20 μm.
- No information on sampled volumes



Fig. 1. Concentration of MPs in each size fraction (20–100  $\mu$ m, 100–300  $\mu$ m, >300  $\mu$ m) before and after the treatments. DF10 = discfilter with pore size 10  $\mu$ m, DF20 = discfilter with pore size 20  $\mu$ m, RSF = rapid sand filters, DAF = dissolved air flotation and MBR = membrane bioreactor.

| Treatment method  | Influent      | Effluent         | Removal % |
|-------------------|---------------|------------------|-----------|
| Primary treatment | 686.7 ± 155.0 | 10.9 ± 2.9       | 98        |
| CAS               | 10.9 ± 2.9    | 1.3 ± 0.9        | 88        |
| RSF               | 0.7 ± 0.1     | $0.02 \pm 0.007$ | 97.1      |
| MBR               | 6.9 ± 1.0     | 0.005 ± 0.004    | 99.9      |



■ Fibers ■ Fragments ■ Films ■ Flakes ■ Spherical

Fig. 2. Concentration of MPs in the different MP shape categories before and after the treatments. DF10 = discfilter with pore size 10  $\mu$ m, DF20 = discfilter with pore size 20  $\mu$ m, RSF = rapid sand filters, DAF = dissolved air flotation and MBR = membrane bioreactor.

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#### Talvitie et al., 2017

CONSIDERING PLANT HRT!!

Water and Waste Environmental Engineering

**Microplastics in wastewater treatment plant** 



# Impact of MicroPlastics in AgroSystems and Stream Environments (IMPASSE)



productions



## **Microplastics in wastewater treatment plant**

| WWTP | Influent type    | Treatment | Season | MP<br>retention<br>% | Effluent<br>discharge<br>m <sup>3</sup> day <sup>-1</sup> | MPs emitted on sampling day |
|------|------------------|-----------|--------|----------------------|---|-----------------------------|
| 1    | Urban/industrial | Secondary | Summer | 97.8                 | 24675   | 202,070                     |
|      |                  |           | Autumn | 99.4                 | 19989   | 148,470                     |
| 2    | Urban            | Secondary | Summer | 93.8                 | 520   | 327,030                     |
|      |                  |           | Autumn | 46.5                 | 370   | 1,075,885                   |
| 3    | Urban/industrial | Tertiary  | Summer | 70.0                 | 1837  | 8,179,635                   |
|      |                  |           | Autumn | 96.2                 | 2121  | 5,071,545                   |
| 4    | Urban            | Secondary | Summer | 80.5                 | 1982  | 101,400                     |
|      |                  |           | Autumn | 67.9                 | 2011  | 166,500                     |
| 5    | Urban/industrial | Tertiary  | Summer | 98.7                 | 32077   | 1,110,375                   |
|      |                  |           | Autumn | 86.0                 | 37567   | 5,596,920                   |



|   | Amount of wastewater<br>produced in the | Concentration of MPs in wastewater |                     |  |  |
|---|---|------------------------------------|---------------------|--|--|
|   | catchment in 2017<br>in m <sup>3</sup>  | mg m <sup>-3</sup>                 | MPs m <sup>-3</sup> |  |  |
| Ireated urban and<br>industrial<br>wastewater | 49,225,286                              | 6.47                               | 161                 |  |  |
| Untreated urban<br>wastewater                 | 789,574                                 | 76                                 | 2439                |  |  |
| Industrial effluents<br>unknown<br>treatment  | 2,276,415                               | 5.5                                | 123                 |  |  |
| Total amount                                  | 52,161,976                              |                                    |                     |  |  |

- The capture efficiency of the wastewater treatment plant varies between 46.5% and 99.4%
- Polyethylene, polypropylene and polyester are the predominant polymer types
  - Paint chips in effluent not detected in influent
- Preferential retention of some polymer/particle types



#### **Microplastics in wastewater treatment plant**



Project funded by the Norwegian Environment Agency in 2017

Main objective: to characterise microplastics in sewage sludge from Norwegian domestic wastewater treatment plants which apply different wastewater and sludge treatment technologies.

Mapping microplastics in sludge

Lusher, Amy L.; Hurley, Rachel; Vogelsang, Christian; Nizzetto, Luca; Olsen, Marianne

Abstract

#### Research report

Published version



Vlew/Open 7215-2017.pdf (8.920Mb)

URI http://hdl.handle.net/11250/2493527

The main objective of this project was to characterize microplastics in sewage sludge from Norwegian domestic wastewater treatment plants applying different wastewater and sludge treatment technologies. WWTPs were selected to cover the threemain domestic WWTP categories in Norway and the main applied sludge treatment processes. Fenton's reagent was used to remove organic matter and density separations were employed to extract microplastics from sludge samples. Plastics were found in all ten sludge samples investigated from eight WWTPs. The overall average plastic abundance was 6 077 particles kg-1 (d.w.) (1701 – 19 837) or 1 176 889 particles m-3 (470 270 – 3 394 274). Based on the average microplastic abundance and the present application of sewage sludge in Norway, it was estimated that over 500 billion microplastics are released into the environment via sewage sludge application each year, to agricultural soils, green areas and soil producers. This likely represents a significant source of microplastics to terrestrial and marine systems.

Description Project manager Marianne Olsen

Publisher Norsk institutt for vannforskning

Serles NIVA-rapport;7215 Miljødirektoratet-rapport;907

Copyright Norwegian Institute for Water Research



8 Norwegian sewage plants monitored (35% of population and annual sludge production).

Analysed sludge samples  $\approx 100$  g for MPs < 50  $\mu m$ 

On average, 181 679 012 microplastic particles transferred to the sludge phase each day, corresponding to 1316 MPs/AE/d (median: 383).

→ Corresponding to 6.8 billion MPs/d, if compared to the Norwegian population

IVAR 762 246 MP m<sup>-3</sup> 152 449 184 MP d<sup>-1</sup> 462 MP person<sup>-1</sup> 470 270 MP m

9 123 243 MP d

152 MP person



Microplastics in wastewater treatment plant - sludge



446 billion MPs spread on agricultural soils

27 billion MPs added to green areas

112 bn MPs sent to soil producers

Reproportioning on production in Norway, 584 bn MPs released into the environment through sludge each year





Polyethylene terephthalate





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# Thank you for the attention!



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# **Results and protocol from Blue Lakes** project Università Politecnica delle Marche





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# **Development of the Standard Monitoring Protocol**





# **Definition of a sampling protocol**









# **Definition of a sampling protocol**



**TECHNICAL PROTOCOL FOR TREATMENT PLANT** 

# NO MICROPLASTICS, JUST WAVES.

Deliverable ACTION B3

TECHNICAL REPORT AND OPERATING MANUAL ON THE

IMPROVEMENT OF THE TREATMENT PROCESS

Manuale tecnico-operativo per l'analisi delle microplastiche negli

impianti di trattamento acque

#### 

A cura di:

Dr. Lucia Pittura (DISVA, UNIVPM) Dr. Veronica Vivani (DISVA, UNIVPM) Dr. Serena Radini (SIMAU, UNIVPM) Dr. Alessia Foglia (SIMAU, UNIVPM) Prof. Stefania Gorbi (DISVA, UNIVPM) Prof. Anna Laura Eusebi (SIMAU, UNIVPM) Prof. Francesco Fatone (SIMAU, UNIVPM)

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PROMOTION OF GOOD PRACTICES EXPERIMENTED ON THE PILOT LAKES DURING THE PROJECT ACTIVITIES





# **Definition of a sampling protocol**

# Literature analysis

| SAMPLING | VOLUME   | TYPE of   | METHODS OF SAMPLING AND                        | FREQUENC   | Ref.      |
|----------|----------|-----------|--|------------|-----------|
| POINTS   |          | SAMPLE    | DETENCTION                                     | Y or num°  |           |
|          |          |           |  | of samples |           |
| Raw and  | 1000 L   | Grab      | Samples directly sieved, tap water require     |            | (Koelmans |
| treated  |          | samples   | no digestion.                                  |            | et al.,   |
| drinking |          |           |  |            | 2019)     |
| water    |          |           |  |            |           |
| Raw and  | 300-2500 | Зµт       | Residual raw water and drinking water was      | 24 samples | (Mintenig |
| treated  | L        | stainless | removed from t e filter units by using         |            | et al.,   |
| drinking |          | steel     | filtered (0.2 µm) compressed air. Then, the    |            | 2017)     |
| water    |          | cartridge | units were filled again with diluted           |            |           |
|          |          | filters   | hydrochloric acid (Carl Roth Cirlin & Co.      |            |           |
|          |          | 4 7/8",   | KG, Germany, 0.2 μm fitel d, pH=2) to          |            |           |
|          |          | Wolftech  | dissolve calcium carbonite and iron            |            |           |
|          |          | nik,      | precipitates. After 2, h the filter units were |            |           |
|          |          | Germany   | emptied, the card lige filters removed from    |            |           |
|          |          |           | the units and insed with Milli-Q and           |            |           |
|          |          | •         | ethalol (30%, Carl Roth GmbH & Co. KG,         |            |           |
|          |          |           | a many, filtered over 0.2 µm). The             |            |           |
|          |          |           | etentate was collected on 3 µm stainless       |            |           |
|          |          |           | steel filters (47mm in diameter) that were     |            |           |
|          |          | •         | subsequently transferred into glass bottles    |            |           |
|          |          |           | and covered with 30 mL hydrogen peroxide       |            |           |
|          |          |           | (35%, Carl Roth GmbH & Co. KG, Germany).       |            |           |
|          |          |           | The bottles were closed using aluminium        |            |           |
|          |          |           | foil and incubated for 24 h at 40 °C. Finally, |            |           |
|          |          |           | each sample was enriched onto a 0.2 µm         |            |           |
|          |          |           | aluminium oxide filter (Anodisc 25 mm,         |            |           |
|          |          |           | Whatman, U.K.) by using an in-house            |            |           |
|          |          |           | fabricated filter-funnel with an inner         |            |           |
|          |          |           | diameter of 11 mm. The filters were dried at   |            |           |
|          |          |           | 40 °C in half closed glass petri dishes for    |            |           |
|          |          |           | subsequent analysis.                           |            |           |

## 1. NECESSARY INSPECTION BEFORE SAMPLING CAMPAIGN

- DETAILS ON THE IDENTIFIED SAMPLING POINTS (presence of taps, pressure, flow rate, possibility of pumping..etc)
- 3. DURATION OF SAMPLING FOR EACH PLANT OF ABOUT 1 DAY, TO POSSIBLY COINCIDE WITH ROUTINE COMPANY CHECKS





# Minimum sampling volumes proposed in BLUE LAKES

| Sector                  | Plant                         | Unit                                | Type of sample             | Min.<br>volume | Min. number<br>of samples | Notes  |
|-------------------------|-------------------------------|-------------------------------------|----------------------------|----------------|---------------------------|--|
|                         |                               | Influent                            | Min 1-2 h average sampling | 1000           | 3*                        | *Min. number of sampling campaigns defined to detect seasonal variability.                       |
|                         | Drieliesuster                 | Effluent from<br>each operativeunit | Min 1-2 h average sampling | 1000           | 3*                        |  |
| Duinking                | Drinking water                |                                     |                            |                |                           |  |
| Drinking<br>water       |                               | Final Effluent                      | Min 1-2 h average sampling | 1000 l         | 3*                        |  |
| supply                  |                               | Sludge**                            | Grab                       | 51             | 3*                        | **Sludge is considered as liquid at maximum TS% of about 5%TS.                                   |
|                         | Distribution                  | Final<br>Distribution***            | Min 1-2 h average sampling | 1000           | 3*                        | ***Min. Number of Sampling points has to be set accordingto the distribution network complexity. |
| Sewage<br>system        | CombinedSewer<br>Overflow     | CSO                                 | Grab or Average sampling   | 50  ****       | 3*                        | ****Min volume could be very variable depending on the quantity overflowed.                      |
|                         | Wastewater<br>Treatment Plant | Influent                            | Average sampling           | 30-300  *****  | 3*                        | *****Min. volume could be very variable depending on watercharacteristic.                        |
| Wastewater<br>Treatment | Wastewater<br>Treatment Plant | Effluent from each operativeunit    | Average sampling           | 30-300   ***** | 3*                        |  |
|                         | Wastewater<br>Treatment Plant | Final Effluent                      | Average sampling           | 30-300   ***** | 3*                        |  |
|                         | Wastewater<br>Treatment Plant | Sludge**                            | Grab                       | 51             | 3*                        |  |







N° N°

m3

g/h

-

secondi

N° unità

N° unità in funzione Volume caduna

Tempo di contatto

Dosaggio (ozono, reagenti, ...)

**Controlavaggi e frequenze** 





Blue Lakes

BEFORE THE BEGINNING OF THE SAMPLING CAMPAIGN, A DATA REQUEST QUESTIONNAIRE FOR THE CHARACTERIZATION OF THE FLOWS AND THE OPERATING UNITS, EVEN UNDER THE DIFFERENT OPERATING AND DISTRIBUTION CONDITIONS RELATED TO SEASONALITY WAS SENT

| UNIT                     | UdM            | Medio mese | Medio mese |
|--------------------------|----------------|------------|------------|
|                          |                | estivo     | invernale  |
| Flowrate                 | l/s            |            |            |
| рН                       | -              |            |            |
| Turbidity                | NTU            |            |            |
| Temperature              | °C             |            |            |
| Dixolved Oxygen          | mg/L           |            |            |
| Conductivity             | microS/cm      |            |            |
| N° unità                 | N°             |            |            |
| N° operating units       | N°             |            |            |
| Volume                   | m3             |            |            |
| Dose (ozone, chemicals,) | g/h            |            |            |
| Contact time             | sec            |            |            |
| Backwash                 | n, m3/h, freq. |            |            |
|                          | _              |            |            |



# Scheme for sampling activity

| SAMPLING POINT | DATE | WEATHER | METHOD | START OF<br>SAMPLING | DURATI<br>ON | END OF SAMPLING | VOLUME | CHEMICAL AND<br>PHYSICAL<br>CHARACTERISATION<br>(es. TSS) | NOTES |
|----------------|------|---------|--------|----------------------|--------------|-----------------|--------|---|-------|
|                |      |         |        |                      |              |                 |        |   |       |
|                |      |         |        |                      |              |                 |        |   |       |
|                |      |         |        |                      |              |                 |        |   |       |

Write down any particular condition (e.g. unit malfunctions, events, anomalies during sampling, ...)

Possibly match the sampling with the routine characterizations of the plant



# **Sampling methodologies for water treatment plants**

Tank



# **Sampling points**



Carbon filters



Sampling

# Sampling methodologies for water treatment plants



**Optimisation of sampling methods** 



# Sampling methodologies for water treatment plants



QA/QC: Prevention and control of external contamination from plastic material and fibres

Main measures adopted during all phases: sampling, recovery, processing and characterization

- use of non-plastic material (stainless steel, glass, aluminum, copper)
- if plastic materials are used (gaskets, washers...) the material must be characterized to eventually exclude it from the results.
- Closed sampling system vs system exposed to the atmosphere.







 Execution of blank samples for each sample collected: a beaker containing deionized water is left open on the workbench for the entire time necessary to recover the sample and subsequently processed according to the same procedures as the water or sludge sample.

# **MPs in drinking water treatment plants**



| DWTP B         |
|----------------|
| Influent       |
| Out ozonation  |
| Out filtration |
| Effluent       |
| Distribution   |

















# **MPs in wastewater treatment plants**



#### WWTP D

Influent Out flotation

Effluent

Sludge



# WATER: AUTOMATIC SAMPLER 25 μm and 50 μm



#### **WWTP E**

Influent

Out sedimentation

Out lamellar settler

Effluent

Sludge



# ABOUT 1000 L FILTERED AT EACH SAMPLING POINT

SLUDGE: GRAB SAMPLE 25 L



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# **Results and Protocol from Blue Lake Project**

# Technical-operative manual for microplastics analysis in the Integrated Water Service matrices

Università Politecnica delle Marche





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# Technical-operative manual for sampling and processing of Integrated Water Service matrices for microplastics analysis, edited by UNIVPM.



The document was developed as "Action" of the Life *Blue Lakes* Project by integrating the skills and knowledge of the research groups of Water and Wastewater Environmental Engineering Lab and of Ecotoxicology and Environmental Chemistry Lab belonging, respectively, to the SIMAU and DiSVA Departments of the Polytechnic University of Marche.

It was developed based on the main methods described in the scientific literature and optimized and validated by the two research groups over the years of experience in sampling and processing environmental matrices from water treatment plants for MPs analysis.

The protocol aims to be an operational guide for those who work in the sector by proposing procedures, providing alternatives and suggestions. The intention is also to favor the harmonization of methods to facilitate the comparison of results.



Microplastics in real wastewater treatment schemes: Comparative assessment and relevant inhibition effects on anaerobic processes

Lucia Pittura <sup>a</sup>, Alessia Foglia <sup>b, \*\*</sup>, Çağrı Akyol <sup>b, \*</sup>, Giulia Cipolletta <sup>b</sup>, Maura Benedetti <sup>a</sup>, Francesco Regoli <sup>a</sup>, Anna Laura Eusebi <sup>b</sup>, Simona Sabbatini <sup>b</sup>, Linda Y. Tseng <sup>c</sup>, Evina Katsou <sup>d</sup>, Stefania Gorbi <sup>a</sup>, Francesco Fatone <sup>b</sup>





Science of the Total Environment 652 (2019) 602-610



The fate of microplastics in an Italian Wastewater Treatment Plant

Stefano Magni <sup>a,\*</sup>, Andrea Binelli <sup>a,\*</sup>, Lucia Pittura <sup>b</sup>, Carlo Giacomo Avio <sup>b</sup>, Camilla Della Torre <sup>a</sup>, Camilla Carla Parenti <sup>a</sup>, Stefania Gorbi <sup>b,c</sup>, Francesco Regoli <sup>b,c</sup>



**Deliverable ACTION B3** 

TECHNICAL REPORT AND OPERATING MANUAL ON THE IMPROVEMENT OF THE TREATMENT PROCESS

Manuale tecnico-operativo per l'analisi delle microplastiche negli impianti di trattamento acque

**Microplastics (MPs):** any synthetic solid particle or polymeric matrix, with size ranging from 1  $\mu$ m to 5 mm, consisting of either items that are manufactured to be of microscopic dimensions (primary origin) or that are formed from the weathering and fragmentation of larger plastic waste items (secondary origin) (*Bessa et al., 2019*).



**Definitions** 



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.......................

**Microfibers (MFs):** synthetic or natural materials of thread like structure with a diameter lower than 50 µm, length ranging from 1 µm to 5 mm, and length to diameter ratio greater than 100. Microfibers are released or shed from textiles or related fibre-based products such as clothes; agricultural, industrial, and home textiles; and some textile products, semi-manufactured goods, or accessories used in other fields, during production, use, and end-of-life disposal (Liu et al., 2019).



**Definitions** 

Synthetic textile fibers: created in the laboratory and derived from petroleum (e.g. polyester, polyamide, acrylic).

Natural textile fibers: obtained from materials of a vegetable (e.g. cotton, linen and hemp) or animal (wool, silk and leather) nature.

Artificial textile fibers: obtained from natural raw materials (such as cellulose or animal and vegetable proteins) but are processed in the laboratory using chemical substances (e.g. viscose or rayon).

NATURAL-BASED MICROFIBRES UBIQUITOUS IN THE ENVIRONMENT and MORE FREQUENTLY FOUND THAN SYNTHETICS: EMERGING FOCUS FOR THE SCIENTIFIC COMMUNITY

IF POSSIBLE, IT IS STRONGLY SUGGESTED TO ALSO INDICATE THE QUANTITATIVE DATA RELATING TO THE FRACTION OF MFs OF NATURAL ORIGIN COMPARED TO THOSE OF SYNTHETIC ONE, IN THE MATRICES UNDER STUDY.





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impianti di trattamento acque

#### 



Sludge sample



Blank sample



#### NOTE

**NaBr:** the solution used is recovered and reused after filtration (0.45  $\mu$ m cellulose membrane), the density is possibly adjusted by adding salt.

**NaCl:** recommended for monitoring plans with a large number of samples to contain costs.

# **Extraction of MPs and MFs: sludge samples**

Complex matrix: several pre-treatment steps are required compared to the procedure applied to water samples.

If the sample to be analyzed is a dewatered sludges, the processing starts directly from point 3.

#### 1. SIEVING

Sieving through a 5 mm (to exclude macro-plastics) and 50  $\mu$ m mesh steel sieve (comparison with water samples obtained from 50  $\mu$ m mesh cartridge filters). The sludge remaining on the 50  $\mu$ m sieve is recovered with a steel spoon/spatula and placed in a preferably low glass container with a large surface area to facilitate phases 2 and 3 (Petri dishes or crystallizers are recommended).

#### 2. DIGESTION (15% $H_2O_2$ )

#### DRYING (40°C)

15% H2O2 solution is added in small quantities several times a day until the digestion reaction is no longer observed. At the end of the reaction, the sample is left to dry in an oven. This stage can last up to a few days.

#### 3. GRINDING

DIGESTION ( $15\% H_2O_2$ )

Once the sample is dried, it is gently ground in a mortar to obtain a powder which will be subjected to further digestion (it prevents the re-aggregation of the dried material).

#### 4. DENSITY SEPARATION with NaBr

| Polymer                       | Abbr. | Polymer density | NaCl | STD | NaBr | Nal | ZnCl <sub>2</sub> | ZnBr <sub>2</sub> | SPT |
|-------------------------------|-------|-----------------|------|-----|------|-----|-------------------|-------------------|-----|
| Polystyrene                   | PS    | 0.01 - 1.06     | +    | +   | +    | +   | +                 | +                 | +   |
| Polypropylene                 | PP    | 0.85 – 0.92     | +    | +   | +    | +   | +                 | +                 | +   |
| Low-density polyethylene      | LDPE  | 0.89 – 0.93     | +    | +   | +    | +   | +                 | +                 | +   |
| Ethylene vinyl acetate        | EVA   | 0.93 - 0.95     | +    | +   | +    | +   | +                 | +                 | +   |
| High-density polyethylene     | HDPE  | 0.94 – 0.98     | +    | +   | +    | +   | +                 | +                 | +   |
| Polyamide                     | PA    | 1.12 – 1.15     | +    | +   | +    | +   | +                 | +                 | +   |
| Nylon 6,6                     | PA 66 | 1.13 – 1.15     | +    | +   | +    | +   | +                 | +                 | +   |
| Poly methyl methacrylate      | PMMA  | 1.16 – 1.20     | +    | +   | +    | +   | +                 | +                 | +   |
| Polycarbonate                 | PC    | 1.20 - 1.22     | +-   | +   | +    | +   | +                 | +                 | +   |
| Polyurethane                  | PU    | 1.20 - 1.26     | +-   | +   | +    | +   | +                 | +                 | +   |
| Polyethylene<br>terephthalate | PET   | 1.38 – 1.41     | -    | +-  | +    | +   | +                 | +                 | +   |
| Polyvinyl chloride            | PVC   | 1.38 - 1.41     | -    | +-  | +-   | +   | +                 | +                 | +   |
| Polytetrafluoroethylene       | PTFE  | 2.10 - 2.30     | -    | -   | -    | -   | -                 | -                 | +   |



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#### 

After step 4, the same steps applied to water samples follow.

#### 5. VACCUM FILTRATION





#### 6. DIGESTION



#### 7. DRYING OF FILTER MEMBRANE (40°C)







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#### 

FRGMENT: hard particle, thick, with sharp cutting edges and an irregular shape (Lusher et al., 2017).



GLITTER: iridescent disc with a hexagonal shape (Yurtsever et al., 2019).



**SPHERE**: particle with every point on the surface having the same distance from the center. It can also be present as a hemisphere, probably due to breakage during production, use or presence in the environment (*Hartmann et al.*, 2019).



FIBER: filiform structure with irregular diameter and frayed ends which can take trilobate, ribbon or L-shaped shapes (<u>Cesa</u> et al., 2017).



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#### **Classification based on Color**

 1. Black ■
 5. Red ■

 2. Blue ■
 6. Green ■

 3. White
 7. Multicolour ■

 4. Transparent
 8. Others ■

# Identification of MPs and MFs extracted from water and sludge samples

# Physical characterization: optical microscopy

#### **Classification based on Shape**

FILM: flat and flexible particle, with an irregular shape (Hartmannet al., 2019).



**FOAM**: flexible and elastic particle, it shapes and softens to the touch (spongy), of different thicknesses and with an irregular outline (*Rochman et al., 2019*).



PELLET: similar to the sphere but tends to be larger and ovoid in shape, usually between 3 and 5 mm (*Rochman et al., 2019*).







Visual sorting: the particles are isolated and transferred onto a "homemade" support which will also be used for the chemical characterization in  $\mu$ ATR-FT-IR spectroscopy. It is closed as a "sandwich" waiting to be characterized.

#### **Classification based on Size**

- 4 size classes: 5 -1 mm, 1-0.5 mm, 0.5-0.1 mm, 0.1-0.02 mm.
- Upper size limit fixed according to definition of MPs and MFs
- Lower limit defined by the detection limit of 20  $\mu m$  of the  $\mu \text{FT-IR}$  instrument in use.
- The size classes identified are in line with the classification proposed by the ISO protocol under development.



190/019 2/1197

Table 1 — Particle size classification

|                          |    |                          |                   |                 |             |            | Dia 100/010 2-     | +107     | £ |
|--------------------------|----|--------------------------|-------------------|-----------------|-------------|------------|--------------------|----------|---|
| classification           |    | large mi-<br>croplastics | microplastics     |                 |             | plastics   | in the environment |          |   |
| particle size<br>classes | μm | 5 000 to 1 000           | < 1 000 to<br>500 | < 500 to<br>100 | < 100 to 50 | < 50 to 10 | < 10 to 5          | < 5 to 1 |   |

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#### NO MICROPLASTICS, JUST WAVES

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#### TECHNICAL REPORT AND OPERATING MANUAL ON THE IMPROVEMENT OF THE TREATMENT PROCESS

INFROVENIENT OF THE TREATMENT PROCESS

Manuale tecnico-operativo per l'analisi delle microplastiche negli impianti di trattamento acque

#### 

# Identification of MPs and MFs extracted from water and sludge samples

Chemical characterization: FT-IR spectroscopy (single-point measurements)



It is made using an **interferometer**, which allows the scanning of all the frequencies present in the IR radiation generated by the source. Scanning is possible thanks to a mobile mirror which, by moving, introduces a difference in the optical path, which gives rise to constructive or destructive interference with the ray reflected by a fixed mirror. This results in an **interferogram** showing the intensity representation in the time domain. By applying the **Fourier transform** we obtain the **infrared spectrum**, i.e. the representation of the intensity in the frequency domain expressed as a wave number.





# Infrared (IR) spectrum: the fingerprint of materials

It appears as a series of absorption bands positioned according to the wave number (frequency expressed in cm-1).

The location (frequency) and intensity of individual absorption bands contribute to the overall spectrum, creating the characteristic fingerprint of the molecule.

The parameters that characterize an IR absorption band are:

- the position
- the intensity that expresses the height of the absorption peak (*strong, medium, weak*)
- the shape (*sharp, broad*)



Absorption band location:

- Functional groups area, from 3800 to 1300 cm-1 and includes bands due to both stretching and deformation of functional groups (e.g. N-H, O-H, C-H, C=C, C=O bonds, etc.).
- Fingerprint area, from 1300 to 650 cm-1. Peaks are the result of the combination of various vibrational modes which give rise to a series of absorption bands typical of each single molecule. They make it impossible for different molecules to have the same IR spectrum.

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# Chemical identification of MPs and MFs extracted from water and sludge sample

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Opening of the hand-made support and check to reposition the particles and fibers that may have moved from their original position or may have remained adhered to the internal surface of the slide used in the "sandwich".

# Instrument in use: FT-IR spectroscope with UATR

An FT-IR spectrophotometer equipped with a universal ATR accessory (UATR) is used to characterize all those extracted particles that are visible to the naked eye and that can be transferred to the instrument using tweezer or mounted needle.









Macro ATR





# Chemical identification of MPs and MFs extracted from water and sludge samples

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Opening of the hand-made support and check to reposition the particles and fibers that may have moved from their original position or may have remained adhered to the internal surface of the slide used in the "sandwich".

# Instrument in use: FT-IR spectroscope + microscope with µATR

An FT-IR spectrophotometer coupled to a microscope equipped with the  $\mu$ ATR accessory is used to characterize all those isolated particles that are not visible to the naked eye.














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- Data elaboration and expression: MPs
- Information resulted from analyses are recorded in a paper file and then reported in an Excel file for the elaboration of quantitative and qualitative results on MPs and MFs.
- Only items of synthetic nature and < 5 mm in size are considered as MPs.
- Items extracted from field samples matching for shape, color and polymer with those found in blank samples, are eliminated from the count.
- Data on quantification/concentration of MPs are expressed as number of MPs extracted per liter/m<sup>3</sup> or per gram (g) of sample based on the type of analyzed matrix.
- Data on physical and chemical characterization of MPs are expressed as the percentage contribution (frequencies) of each shape, size class, color and polymer on the total extracted MPs.







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 Besides data on synthetic microfibers (included in MPs elaboration) it is suggested to elaborate and show also data relating to microfibers of natural origin.

Results can be presented as follows:

- Number of total MFs extracted per liter (L) or per m<sup>3</sup> or per gram (g) (depending on the type of analyzed matrix), including both those of synthetic and natural origin.
- Percentage contribution of natural MFs vs synthetic MFs on the total of characterized MFs.
- Number of MFs of natural origin and, separately, of synthetic one extracted per liter (L) or per m<sup>3</sup> or per gram (g).

| Sampling step         | Sampling period | MFs/m <sup>3</sup> | Synthetic MFs (plastic) | Natural MFs |
|-----------------------|-----------------|--------------------|-------------------------|-------------|
|                       | November 2020   | 0.0                | 0                       | 0           |
| Influent              | June 2021       | 2.06               | 0                       | 100%        |
|                       | September 2021  | 5.02               | 0                       | 100%        |
|                       | November 2020   | 2.0                | 0                       | 100%        |
| Out ozonation         | June 2021       | 4.82               | 40%                     | 60%         |
|                       | September 2021  | 0.99               | 0                       | 100%        |
|                       | November 2020   | 3.0                | 33%                     | 67%         |
| Out filtration        | June 2021       | 2.00               | 0                       | 100%        |
|                       | September 2021  | 2.95               | 0                       | 100%        |
|                       | November 2020   | 3.9                | 100%                    | 0%          |
| Effluent              | June 2021       | 3.09               | 33%                     | 67%         |
|                       | September 2021  | 8.86               | 22%                     | 78%         |
|                       | November 2020   | 3.0                | 0                       | 100%        |
| Distribution          | June 2021       | 1.00               | 0                       | 100%        |
|                       | September 2021  | 6.01               | 17%                     | 83%         |
|                       | July 2020       | 26,500             | 40%                     | 60%         |
| Flocculated<br>sludge | November 2020   | 23,250             | 23%                     | 77%         |
| 00000                 | May 2021        | 2,000              | 60%                     | 40%         |

Data elaboration and expression: focus on MFs

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#### Thank you for the attention!



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#### 



#### Pratical activities at the DiSVA Educational Laboratories (afternoon of April 21<sup>st</sup>)

#### **EXTRACTION OF MICROPLASTICS FROM WATER AND SLUDGE SAMPLES**

Demonstration of the processing of a sludge sample: pre-treatments and density gradient separation, parallel run of the blank sample.

Vacuum filtration of pre-digested water samples and blank sample.

#### VISUAL SORTING E PHYSICAL CHARACTERIZATION of POTENTIAL MPs

Observation of membrane filters obtained from the filtration of water samples and classification of the isolated particles on the basis of shape and color.

#### CHEMICAL CHARACTERIZATION of EXTRACTED ITEMS

Polymeric identification of isolated particles by macro-ATR-FTIR spectroscopy.

VISIT TO THE DISVA RESEARCH LABORATORIES (Laboratory of Ecotoxicology and Environmental Chemistry) Characterization of MPs by µATR-FTIR spectroscopy.



# **Benchmark assessment** Università Politecnica delle Marche

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DELLE MARCHE



QUESTIONNAIRE ON THE METHODOLOGIES APPLIED BY DRINKING WATER AND WASTE WATER TREATMENT PLANTS IN ITALY AND GERMANY TO REDUCE MICROPLASTICS (MPs) IN THE ENVIRONMENT

#### 📄 🛛 Questionario LIFE Blue Lakes 🗀 🕁

| () () ()<br>() | Invia |
|----------------|-------|
|----------------|-------|

#### Domande Risposte

| gurazione descritta è tipica per quanti impianti e di che taglia, tra gli impianti gestiti dalla *<br>nda? | a configurazione descritta è tipica per quanti impianti e di che taglia, tra gli impianti gestiti dalla *<br>La azienda?<br>MORE THAN 50 STAKEHOLDERS CONTACTED | LIFE Blue Lakes Questionario         Metodologie applicate dagli impianti di trattamento delle acque potabili e reflue in Italia e in Germania per ridurre le microplastiche (MPs) nell'ambiente         Per quale tipologia di impianto sta compilando il questionario?       *         Impianto di depurazione       Impianto di potabilizzazione | <ul> <li></li></ul> | <b>Biue</b><br>Biue<br>De microplastice, just weives |
|--|---|---|---------------------|--|
|  | MORE THAN 50 STAKEHOLDERS CONTACTED   | La configurazione descritta è tipica per quanti impianti e di che taglia, tra gli impianti gestiti dalla *<br>sua azienda?  |                     | NO MICROPLASTICS, JUST WAVE                          |



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**T T T T** 





In caso di impianto di potabilizzazione, quali sono i diversi contributi dell'acqua influente? Indicare tipologie e percentuali dei diversi contributi







In caso di impianto di depurazione, la rete è mista o separata? 24 risposte



In caso di impianto di depurazione, quali sono i diversi contributi dell'acqua reflua influente (es: acque reflue municipali o industriali)? Indicare tipologie e percentuali dei diversi contributi







State attualmente considerando la presenza ed il destino delle microplastiche come tematica prioritaria nella vostra regione e nell'impianto? 24 risposte

NoSì



Al momento state monitorando e/o misurando microplastiche? Nel caso, con che frequenza e con quali metodi? Potreste descrivere le procedure di campionamento e misurazione applicate? <sup>24 risposte</sup>



Sono applicate al momento nell'impianto trattamenti/metodi specifici per ridurre le microplastiche? Nel caso, che tipo di metodi e/o trattamenti?







Ci sono evidenze (es: da altri progetti o attività svolti nella vostra regione) riguardo la presenza di microplastiche nell'acqua trattata? 24 risposte



In caso di presenza di microplastiche, il vostro impianto è flessibile a miglioramenti per aumentare le efficienze di rimozione? Ci sono unità esistenti...entate o ne dovrebbero essere installate di nuove? 24 risposte



Sono presenti metodologie/trattamenti nell'impianto che, anche se non specificatamente mirati per le microplastiche, potrebbero rimuoverle dall'acqu...no le efficienze di rimozione supposte o misurate? <sup>24 risposte</sup>



Siete disponibili/disposti a organizzare campagne di monitoraggio nel vostro impianto per individuare microplastiche?

No

24 risposte







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| IFE Blue Lakes Survey Index Risposte Index Risposte Index Risposte Used to a supplied by Italian and German treatment plants to reduce microplastics (MPa) in the environment Indirizzo email * Methodologies applied by Italian and German treatment plants to reduce microplastics (MPa) in the environment Indirizzo email * | *       • | <ul> <li>41 Italian water companies and 23 German utilities contacted</li> <li>24 surveys collected from 17 Italian water utilities and 5 questionnaires from plants in Germany</li> </ul> |
| The selected configuration is typical for how many plants and for which treatment capacity? *<br>Testo risposta lunga   |   |  |

- While the most common configurations of water and wastewater treatment plants are not specifically designed for the removal of microplastics, current units can impact their removal from water streams.
- Although most of the interviewees do not yet consider the removal of MPs as a priority, their presence in waters has already been investigated by some activities carried out in the area by research institutions or universities (VALUE CE-IN, ENEA, UNICT project).
- The companies interviewed showed their willingness to organize sampling campaigns to detect microplastics in the plants
- Companies have expressed their interest in having specific free training on the occurrence and removal of microplastics in treatment plants.

# **Microplastics in the Integrated Water Service**



Several aspects require more scientific attention to better understand the presence and fate of microplastics in these systems



Routes of entry for microplastics leading to drinking water sources. Eerkes-Medrano 2019

- Study focused on the fate of microplastics in different plants
- Study focused on the fate of microplastics in different processes and treatment units
- Role of sewage flood events in the release of microplastics
- The fate and implications of microplastic release into receiving water bodies and soil environments.







A.A. Koelmans et al. (2019)

Box and whisker plot showing median and variation in microplastic number concentrations in individual samples taken from different water types. Data relate to individual samples unless only means were reported, in which case the mean value was taken into account n times, with n being the number of samples which the mean was based on. References included: (Estabbanati and Fahrenfeld, 2016; Faure et al., 2015; Fischer et al., 2016; Hoellein et al., 2017; Kosuth et al., 2018; Leslie et al., 2017; Magnusson and Noren, 2014; Mason et al. 2016a, 2018; McCormick et al. 2014, 2016; Mintenig et al., 2019b; Obmann et al., 2018; Pivokonsky et al., 2018; Rodrigues et al., 2018; Schymanski et al., 2018; Simon et al., 2018; Talvitie et al. 2015, 2017a, 2017b; Vollertsen and Hansen, 2017; Wang et al. 2017, 2018; Ziajahromi et al., 2017), with n ½ 27.

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## **Microplastics in the Integrated Water Service**



NB: Pay attention to the number of quantifications available (number of case studies) and the protocols used (both for sampling and for analyses)

Currently, the legislation does not indicate a concentration limit for the presence of microplastics in water, nor sampling and analysis methods.

- Considerable heterogeneity of the concentrations found in the literature (logarithmic scale) between the different categories
- Considerable variability even within the same category (e.g. purification effluent)
- Groundwater is characterized by low concentrations (and low variability)
- Effluents from treatment plants are on average about 3 orders of magnitude lower than influents
- Bottled water has higher average concentrations than tap water



# Literature analysis for drinking water

| Author                     | Treatment unit          | Volume sampled                         | n°MPs/L                                |  |  |
|----------------------------|-------------------------|--|--|--|--|
| (Mang at al. 2020)         | Influent                | 1L x 3 samples                         | 6614 ± 1132                            |  |  |
| (Wallg et al., 2020)       | Effluent 1L x 3 samples |  | 930 ± 71                               |  |  |
|                            | Influent                | 2L x 3 times/day                       | 23±2                                   |  |  |
| (Divokonský ot al. 2020)   | Effluent                | 2L x 3 times/day                       | 14±1                                   |  |  |
| (FIVOROTISKY et al., 2020) | Influent                | 2L x 3 times/day                       | 1296±35                                |  |  |
|                            | Effluent                | 2L x 3 times/day                       | 151 ± 4                                |  |  |
|                            | Influent                | 1L x 3 samples/day x 3 times x 3 days  | 1473 ± 34                              |  |  |
|                            | Effluent                | 1L x 3 samples/ day x 3 times x 3 days | 443 ± 10                               |  |  |
| (Divokonsky at al. 2018)   | Influent                | 1L x 3 samples/ day x 3 times x 3 days | 1812 ± 35                              |  |  |
| (FIVOROTISKY Et al., 2010) | Effluent                | 1L x 3 samples/ day x 3 times x 3 days | 338 ± 76                               |  |  |
|                            | Influent                | 1L x 3 samples/day x 3 times x 3 days  | 3605 ± 497                             |  |  |
|                            | Effluent                | 1L x 3 samples/day x 3 times x 3 days  | 628 ± 28                               |  |  |
|                            | Influent                | 300-1000 L                             | 0.003                                  |  |  |
|                            | Distribution            | 1200-2500 L                            | <0.001                                 |  |  |
|                            | Influent                | 300-1000 L                             | 0.007                                  |  |  |
| (Mintenig et al. 2010)     | Effluent                | 1200-2500 L                            | <0.001                                 |  |  |
| (Wintering et al., 2013)   | Distribution            | 1200-2500 L                            | 0.003                                  |  |  |
|                            | Influent                | 300-1000 L                             | 0.001                                  |  |  |
|                            | Effluent                | 1200-2500 L                            | 0.002                                  |  |  |
|                            | Influent                | 300-1000 L                             | 0.001                                  |  |  |
|                            | Influent                | 10 L *2 duplicates                     | 42 ± 18 particles/L                    |  |  |
| (Cherniak et al., 2022)    | Effluent                | 10 L *2 duplicates                     | 20 ± 8 particles/L                     |  |  |
|                            | Distribution            | 10 L *2 duplicates                     | 20.5 ± 7.6 particles/L                 |  |  |
| (lung of al. 2022)         | Influent                | 10,100 L *12 monthly samples           | 2.2 ± 1.3                              |  |  |
| (Julig et al., 2022)       | Effluent                | 10-100 L 12 monthly samples            | 0.02 ± 0.02                            |  |  |
| Shi at al 2021             | Influent                | nd                                     | 6614 ± 1132                            |  |  |
| 5111 et al., 2021          | Effluent                | 11.0.                                  | 930 ± 71                               |  |  |
| (Yuan et al., 2022)        | Influent                | nd                                     | 17.88                                  |  |  |
|                            | Effluent                | 11. <b>u</b> .                         | 2.75                                   |  |  |
| (Johnson et al. 2020)      | Influent                | nd                                     | 21.09 ± 20.49                          |  |  |
| (Johnson et al., 2020)     | Effluent                | n.u.                                   | 0.001-0.024                            |  |  |
| (leclie et al. 2017)       | Influent                | n.d.                                   | 1385 (dry season); 1796.6 (wet season) |  |  |
|                            | Effluent                | n.d.                                   | 448.7 (dry season); 769.4 (wet season) |  |  |

The analysis of the literature reveals considerable heterogeneity, also due to the different sampling methods and the representativeness of the volumes analyzed.

As the sampling volumes increase, the concentrations are considerably reduced >> greater representativeness of the results, also in consideration of the actual volumes treated in the plant





# Literature analysis for tab water and bottled water



| Author   | Water type   | Lower size bound<br>(µm)   | Particles/L in sample (average)   | Particles/L in blanks<br>(average)                         | Particle size (μm)   | Particle shape                                    | Predominant polymer<br>type  | Few studies  |
|--|--|--|---|--|--|---|--|--|
| (Oßmann et al.,<br>2018)                           | Bottled (mineral water)<br>• Glass<br>• Single use PET<br>• Reusable PET | 1  | 3074–6292<br>2649<br>4889   | 384  | Most particles smaller than 5<br>(>75% in glass<br>and >95% in plastic bottles), | No discussion of shapes                           | PET in plastic bottles, PE,<br>and styrene butadiene<br>copolymer in glass | <ul> <li>Variability of values</li> <li>The size is not</li> </ul>     |
| Schymanski et al.<br>(2018)                        | Bottled<br>• Single use<br>• Returnable<br>• Glass<br>• Beverage carton  | 5–20   | 14<br>118<br>50<br>11   | 14±13  | 40–50% in 5–10<br>range; over 80%<br><20   | No discussion on shape;<br>described as fragments | PET but also PP, PE  | always<br>defined  |
| (Mason et al., 2016)                               | Bottled  | 6.5–100<br>lower bound<br>based on<br>microscope and<br>software | 315   | 23.5   | Not specified  | Not specified                                     | No characterization  | <ul> <li>Not clear if<br/>values include<br/>microfibers of</li> </ul> |
|  |  | ~100   | 10.4  | 4.15   | Not specified  | fibres (13%), films<br>(12%)                      | FF (3470)  | different  |
| Strand et al. (2018)                               | Tap from ground- water sources   | 10–100   | 0.2, 0.8 and 0.0 (LoD<br>= 0.3) **  | Unknown  | Mainly 20–100.   | Fragments   | PET, PP, PS,<br>acrylonitrile butadiene<br>styrene, PUR                    | <ul><li>Origins</li><li>Predominant</li></ul>                          |
|  |  | >100<br>(10 µm sieve size)                                       | 0.312 (LoD = 0.58)**  | 0.26   | Not specified  | Fibres (82%),<br>fragments (14%),<br>films (4%)   | PET, PP, PS  | type of PET  |
| (Mintenig et al., 2019)                            | Tap from ground- water sources   | 20   | 0.0007  | 0.67 particles/L<br>0.3 fibres/L                           | In the range 50–150.<br>Fragments  | Fragments   | Polyester, PVC, PE, PA, epoxy resin  | polymer for  |
| Uhl,<br>Eftekhardadkhah,<br>and Svendsen<br>(2018) | Tap from 24 sources  | 60   | Average not<br>reported since only<br>a single result above<br>LoQ (that result was<br>5.5) | 0.5<br>(LoQ = 4.1 LoD**<br>= 0.9)                          | Not specified  | Not specified                                     | No characterization  | variable for<br>tap water  |
| Kosuth, Mason and<br>Wattenberg (2018)             | Tap from unspecified sources   | 100 lowest<br>reported   | 5.45  | 0.33 (based on 5 particles in<br>30<br>blanks (ea. 500 mL) | Fibre lengths 100–5000   | Mainly fibres (98.3%).                            | No characterization  | •  |

Adattato da WHO, 2019. Microplastics in drinking-water

## Literature analysis for wastewater



| Reference   | Treatment | n°MPs/L     |
|---|-----------|-------------|
| Browne et al. 2011                                    | Effluent  | 1           |
| 0 1 2010  | Effluent  | 0           |
| Carr et al. 2016                                      | Effluent  | 1.36626E-06 |
|   | Influent  | 293         |
| Dris et al. 2015                                      | Effluent  | 35          |
|   | Effluent  | 0.02        |
| Dyachenko et al. 2017                                 | Effluent  | 0.17        |
|   | Influent  | 57.6        |
| Lares et al. 2018                                     | Effluent  | 1           |
|   | Influent  | 489         |
| Leslie et al. 2017                                    | Effluent  | 52          |
|   | Influent  | 15.1        |
| Magnusson and Noren 2014                              | Effluent  | 8.25        |
| Mason et al. 2016                                     | Effluent  | 0.05        |
|   | Influent  | 133         |
|   | Effluent  | 2.6         |
| Michielssen et al. 2016                               | Effluent  | 2.6         |
|   | Effluent  | 0.5         |
| Mintenig et al. 2017                                  | Effluent  | 4.505       |
|   | Influent  | 15.7        |
| Murphy et al. 2016                                    | Effluent  | 0.25        |
|   | Influent  | 7216        |
| Simon et al. 2018                                     | Effluent  | 54          |
|   | Influent  | 430         |
| l'alvitie et al. 2015                                 | Effluent  | 8.6         |
| T   11     2047                                       | Influent  | 645         |
| l'alvitie et al. 2017                                 | Effluent  | 1.4         |
| T   W       2017                                      | Influent  | 6.9         |
| Talvitle et al. 2017b                                 | Effluent  | 0.3         |
| The Device Francisco entrol Destantion According 2017 | Influent  | 5.9         |
| The Danish Environmental Protection Agency, 2017      | Effluent  | 0.016       |
| Vermaire et al. 2017                                  | Effluent  | 0.07        |
|   | Effluent  | 1.5         |
| Ziajahromi et al. 2017                                | Effluent  | 0.48        |
| -   | Effluent  | 0.28        |
| Marri et al. 2010                                     | Influent  | 2.5         |
| iviagni et al., 2018                                  | Effluent  | 0.4         |
|   | Influent  | 24          |
| Gündoğdu et al., 2018                                 | Effluent  | 5.5         |

Also for wastewater treatment plants there is considerable variability in sampling and analysis methods

Although large data variability was reported, MPs concentration ranged between 6 and 7\*10^3 particles/L in influent and 0 and 35 particles/L in effluent.

The concentrations of removed MPs found vary in conventional case studies by 4 orders of magnitude

### **MPs in wastewater treatment plants**



#### **Comparison with literature**

WWTP



WWTP C

WWTP D

Comparison between the concentrations measured with the technicalscientific literature

- The measured values are below those of the literature
- The literature results are extremely variable and influenced by the different sampling, analysis and characterization methods, as well as by site-specific conditions
- Despite the already low concentrations entering the plant, further reductions are found at the end of the treatment chain.



# MPs in drinking water treatment plants



#### **Comparison with literature**





MPs decrement/increment in DWTPs



Comparison between the concentrations measured with the technicalscientific literature

Comparison between the

measured reductions and

the technical-scientific

literature

- The measured values are below those of the literature
- The literature results are extremely variable and influenced by the different sampling, analysis and characterization methods, as well as by site-specific conditions
- Despite the already low concentrations entering the plant, further reductions are found at the end of the treatment chain.



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#### Thank you for the attention!



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