Analysis of microplastics in effluents and digestates samples using FTIR, Raman Spectrophotometer and Pyrolysis GC-MS

Lakshika Rathnayake, Adam Henley, Tim Patterson and Sandra Esteves

Wales Centre of Excellence for Anaerobic Digestion, Sustainable Environment Research Centre, Faculty of Computing, Engineering and Science, University of South Wales, Pontypridd CF37 1DL, UK

Angelo Iannatelli

PI Projects Analytical Services Ltd, Swindon, Wiltshire, SN2 2PN, UK

University of South Wales Prifysgol De Cymru

Introduction

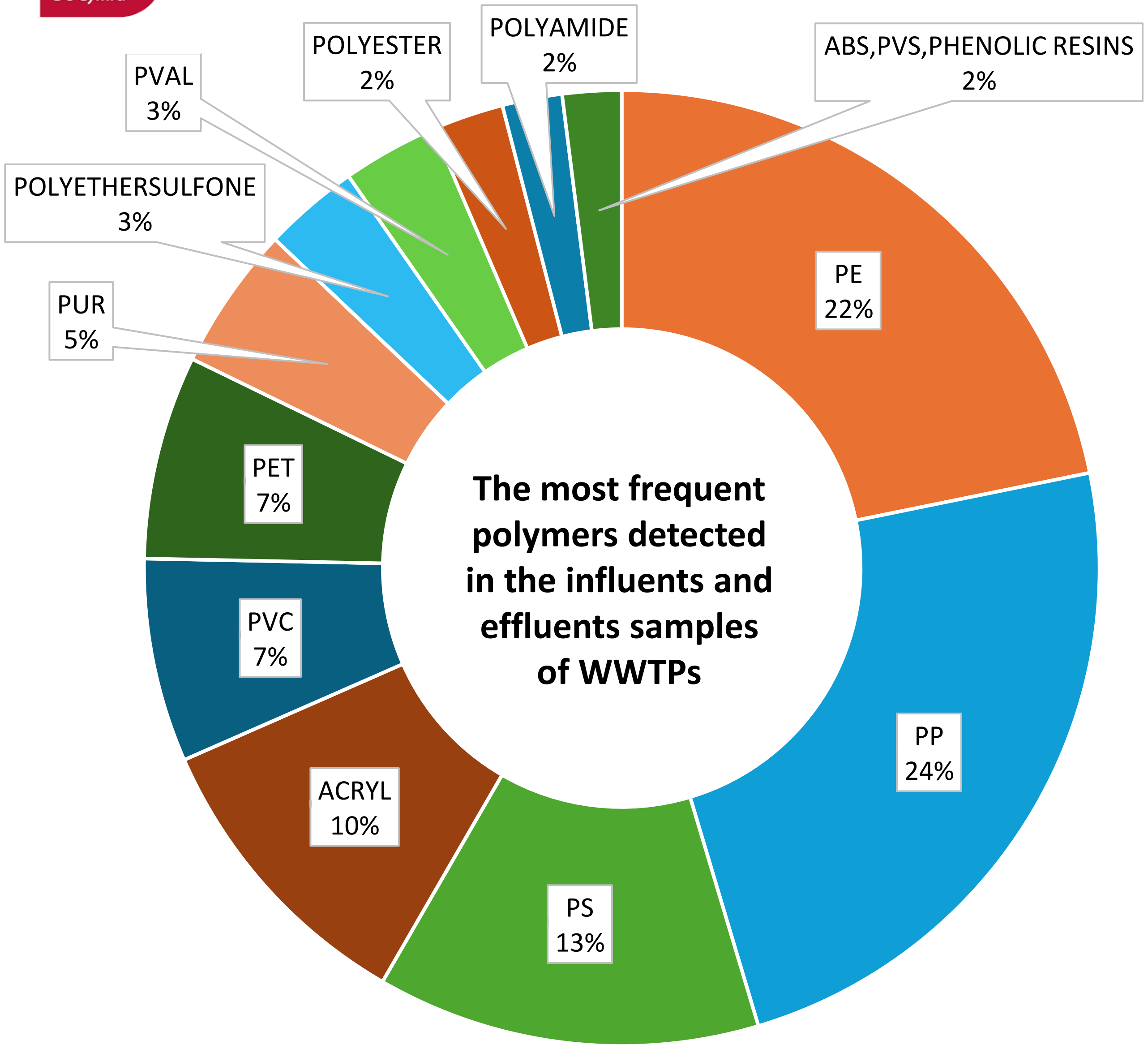
Humans have created 9200 million metric tons of plastic over the past five decades, with 79% of plastic waste ending up in landfills or polluting the environment (Walker and Fequet, 2023)

Once in the environment, the plastic materials can slowly breakdown to generate a large amount of microplastics < 5 mm in size.

Polymers like PP, PE, PPE, PET, PVC, PTFE, polyamide, thermoplastics, synthetic rubbers and PS are some of the major ones that are commonly found in environmental samples.

Microplastics have been visualized by stereomicroscope (using fluorescence tagging) and SEM, whilst polymer identification has been conducted using various techniques including ATR-μFTIR, ATR-FTIR, Raman spectroscopy, and Pyrolysis GC-MS

University of South Wales
Prifysgol De Cymru



The major sources of microplastics entering wastewater treatment plants(WWTPs)

- Synthetic textiles releasing microfibers during washing
- Personal care products with microbeads, household dust
- Industrial discharges, tyre wear particles, road marking paints
- Agricultural activities such as plastic mulching and biosolid
- Construction materials
- Marine debris

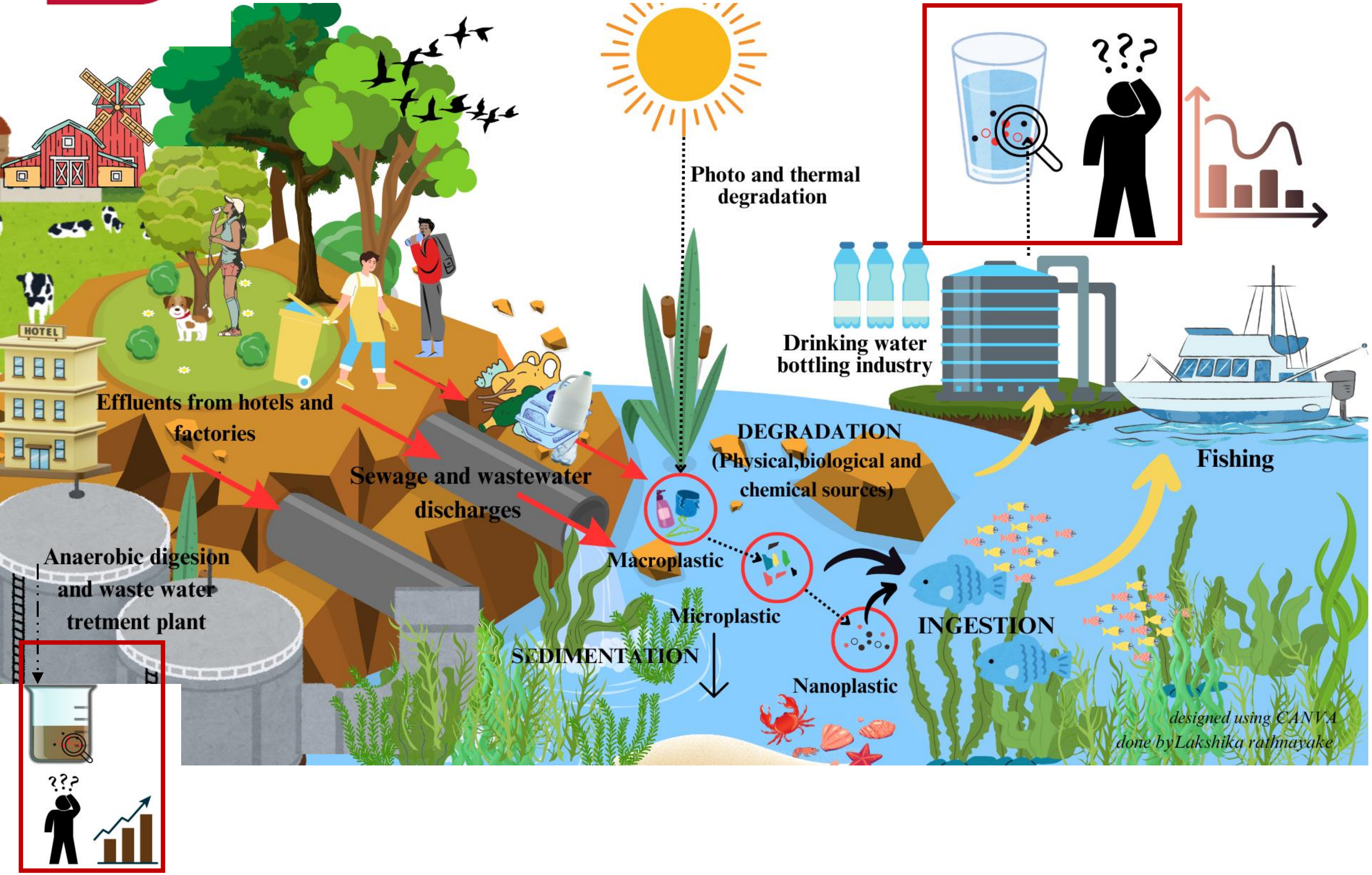
In wastewater treatment plants, microplastics are partially removed through sedimentation and filtration, with some retained in sludge while others escape into effluent and the environment.

Mabadahanye, K., Dalu, M.T. and Dalu, T., 2024. Occurrence and Removal of Microplastics in Wastewater Treatment Plants: Perspectives on Shape, Type, and Density. *Water*, *16*(12), p.1750.

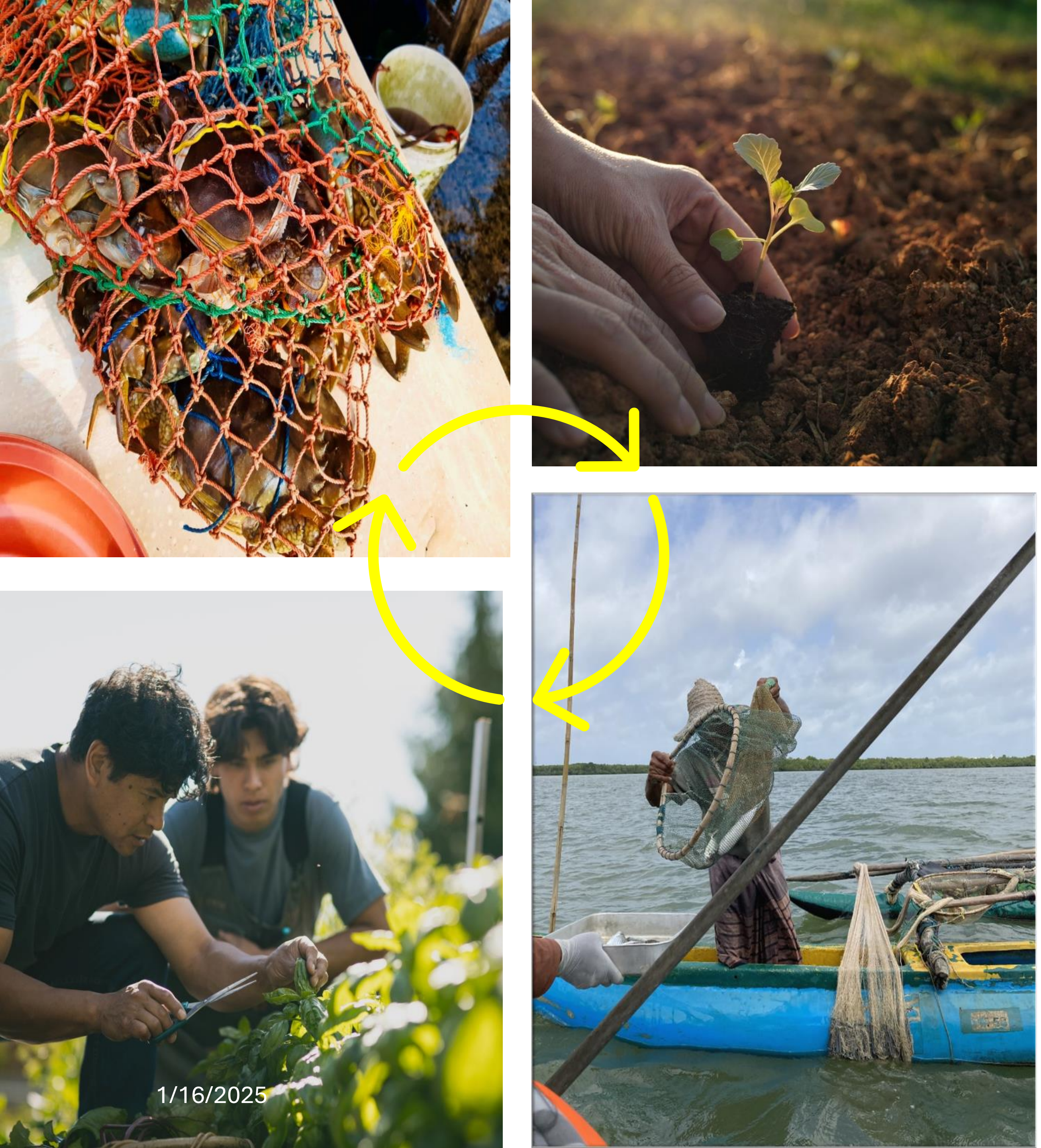
Acarer, S., 2023. Microplastics in wastewater treatment plants: sources, properties, removal efficiency, removal mechanisms, and interactions with pollutants. *Water Science & Technology*, *87*(3), pp.685-710.

University of South Wales
Prifysgol De Cymru

Aim of the study



- To design a methodology to extract microplastics from wastewater, sewage waste and sludge.
- To compare microplastic analysis methods. Ex: FTIR. Raman spectroscopy, Pyr-GCMS.
- To develop and validate microplastic analysis by Pyr-GC MS method



Most of Microplastics retained in sludge.

78.27 ± 9.83%*

They can contaminate soil when used as fertilizer, affecting soil health and entering the food chain.

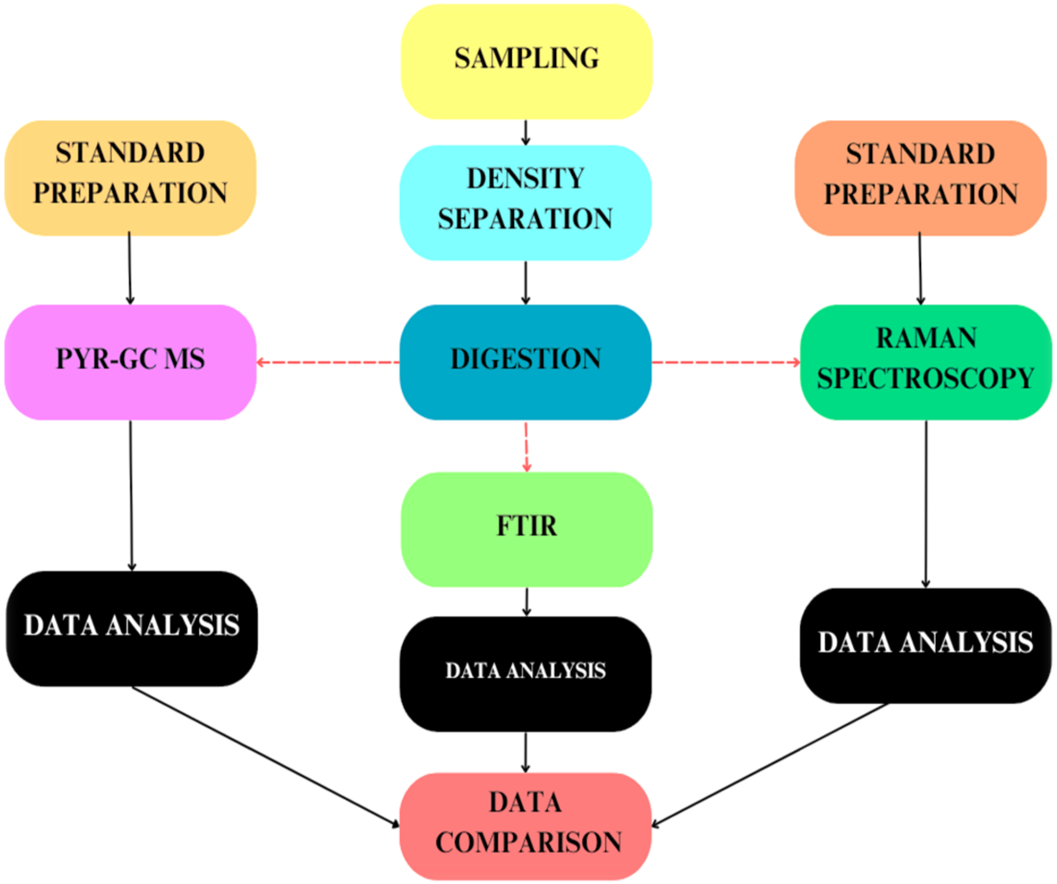
Small amount of Microplastics can remain in effluent after wastewater treatment process.

They can escape into rivers and oceans, harming aquatic ecosystems and accumulating in marine organisms, potentially impacting human health through seafood consumption.

*Li, X., Chen, L., Mei, Q., Dong, B., Dai, X., Ding, G. and Zeng, E.Y., 2018. Microplastics in sewage sludge from the wastewater treatment plants in China. *Water research*, *142*, pp.75-85.

University of South Wales
Prifysgol De Cymru

Methodology



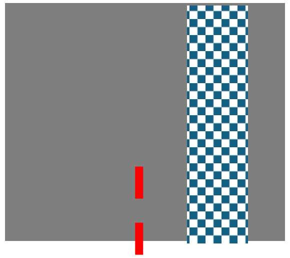
Microplastic analysis techniques

Most common techniques of Microplastic analysis and their characteristics

Optical microscope	Scanning electron microscope (SEM)	Raman spectroscopy	FTIR	Pyr-GC MS
Visual Identification	Polymer and Visual identification	Polymer identification	Polymer identification	Polymer identification (Emerging technique)
Visual counting colour, size and morphology	Colour and the detailed information about the shape, morphology, and structure	colour, shape, morphology, chemical composition and structure	colour, shape, morphology, chemical composition and structure	polymer type and detailed structural information about macromolecules of polymers
Fluorescence tagging by Nile Red	Identify inorganic plastic additives and gives a clear image of the size and topography of the particle.	The interaction of a polymer and the consequent changes of photons in monochromatic light has the ability to provide structural information.	Infrared spectrum of emission or absorption spectra facilitates the determination of the structure of polymer.	The decomposition of the polymer provide a specific signature to a specific polymers and determines the chemical identity by mass spectrometry (MS)

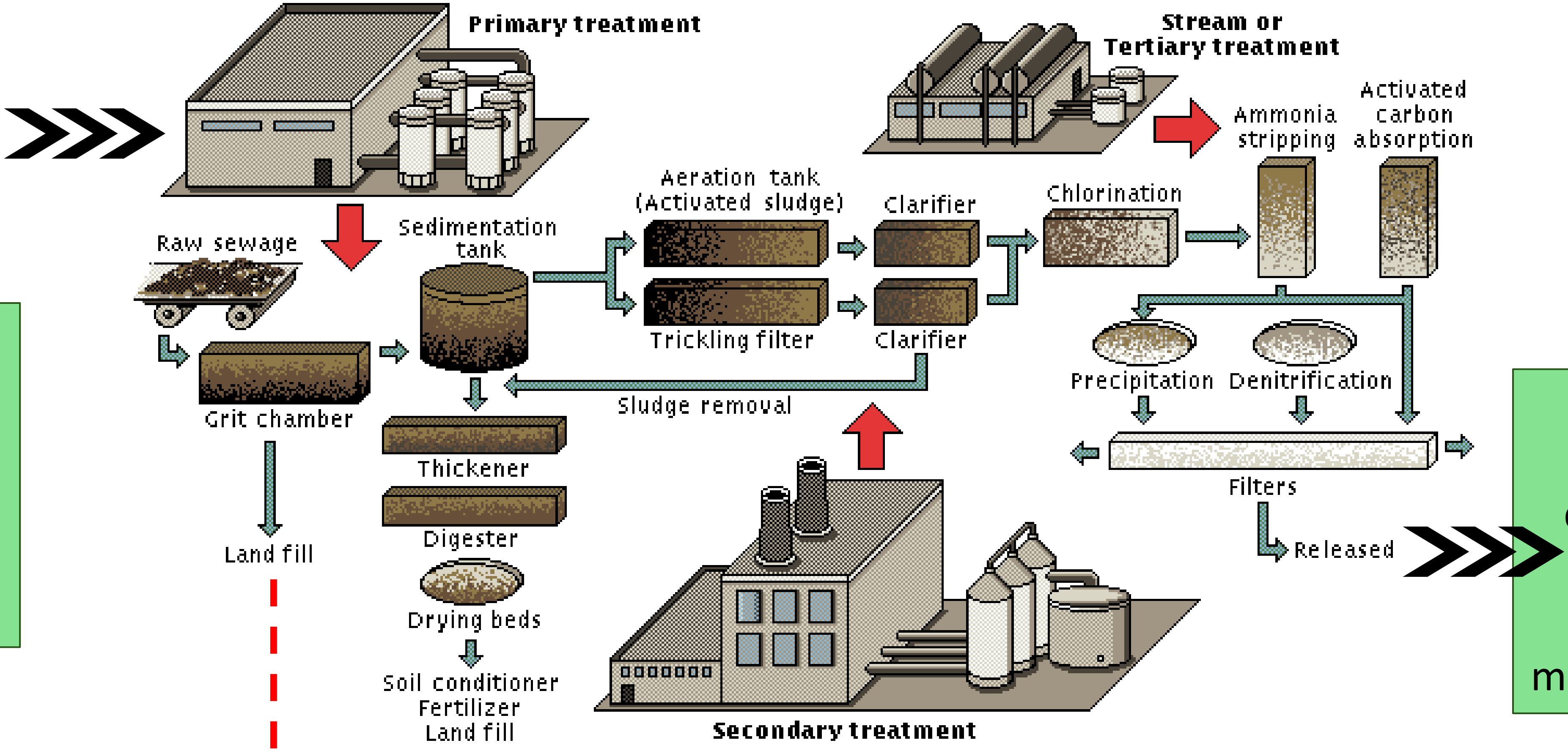
Technique	Advantages	Disadvantages
FTIR Spectroscopy	- High chemical specificity to identify polymer types. - Non-destructive. - Can analyse a wide range of sizes >10 μm (using ATR mode).	- Limited to relatively large particles (10–20 μm and above). - Less effective for opaque or thick samples. - Time-consuming for large sample sizes. - Sensitivity decreases for nanoscale plastics.
Raman Spectroscopy	- High spatial resolution (down to 1 μm). - Non-destructive. - Can detect pigments and additives. - Effective for dark and opaque samples. - Complementary to FTIR.	- Fluorescence interference from sample matrix. - More time-consuming than FTIR. - Sample preparation can be complex. - Limited performance in identifying highly degraded polymers.
Pyr-GCMS	- Can identify and quantify polymer composition. - Highly sensitive, even for nanoplastics. - Detects additives and degradation products. - Suitable for mixed and complex matrices.	- Destructive technique. - Time-intensive and requires expert interpretation. - Cannot provide particle size or shape. - Expensive equipment and maintenance.

Screening



Entry point of untreated wastewater (assess initial microplastic load).

1



2

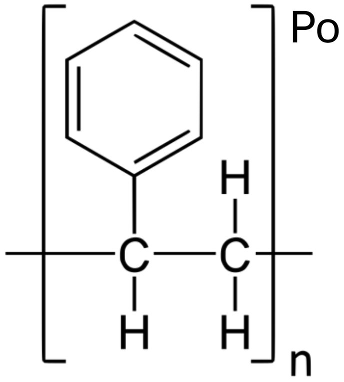
3

4

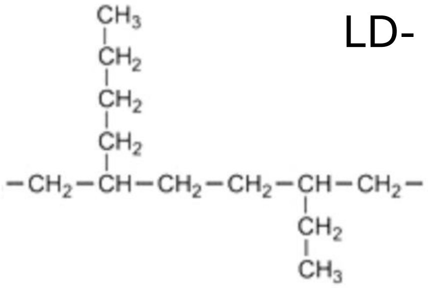
Treated effluent discharged (evaluate residual microplastics).

ANALYSIS OF COMMON POLYMERS.

- **Polyethylene (PE)**
- **Polypropylene (PP)**
- **Polystyrene (PS)**
- **Polyethylene Terephthalate (PET)**
- **Polyvinyl Chloride (PVC)**
- **Polyamide (PA, including Nylon)**
- **Acrylics (PMMA)**
- **Polytetrafluoroethylene (PTFE)**
- **Polyvinyl Alcohol (PVAL)**
- **Ethylene Vinyl Acetate (EVA)**
- **Polycarbonate (PC)**
- **Polysulfone (PSU)**
- **Polyether sulfone (PES)**
- **Phenolic Resins**
- **Acrylonitrile Butadiene Styrene (ABS)**
- **Alkyd Resins**
- **Styrene Butadiene Rubber (SBR)**
- **Cellulose Acetate**
- **Low-Density Polyethylene (LDPE)**
- **High-Density Polyethylene (HDPE)**
- **Thermoplastic Polyolefins (TPO)**
- **Epoxy Resins**

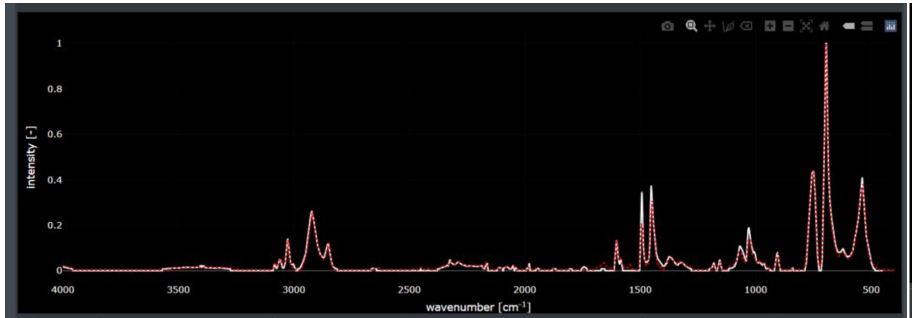


Polystyrene (PS)

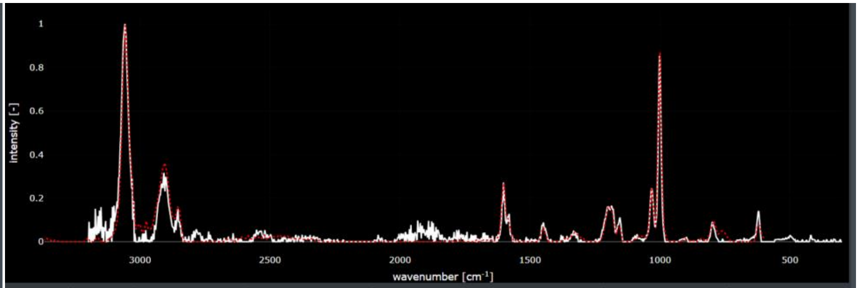


LD- PE

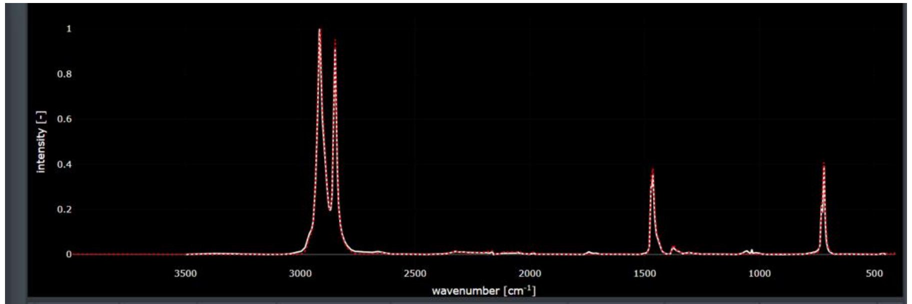
FTIR spectrum	Raman spectrum



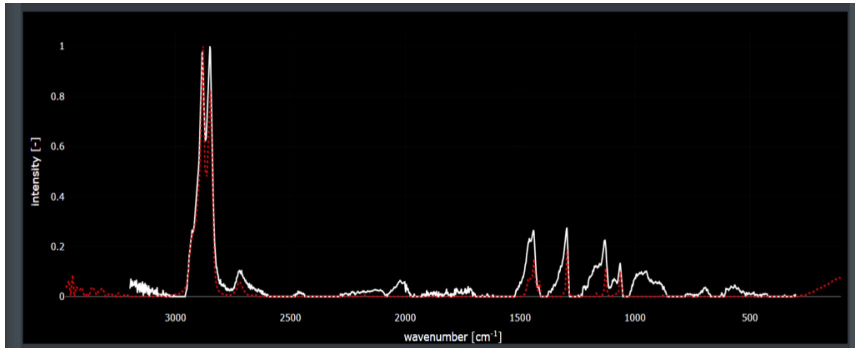
location	data_processing_proceedure	Plastic Pollution Category	plastic_or_not	material_class_specific	max_cor_class	max_cor_val
california	none	polystyrenes (polyphenylethylenes, -methylstyrene)	plastic	polystyrene	polystyrenes (polyphenylethylenes, -methylstyrene)	0.9897462345604796



ecifications	replicate	material_producer	Plastic Pollution Category	plastic_or_not	material_class_specific	max_cor_class	max_cor_val
ps	a	carat	polystyrenes (polyphenylethylenes, -methylstyrene)	plastic	polystyrene	polystyrenes (polyphenylethylenes, -methylstyrene)	0.9816895482467343



Material	librarytype	spectrumid	instrument_mode	other_info	material_color	material_form	material_producer	Pla Pol Cat
polyethylene low density	polymer, minerals, organic materials	312primpke	atr	https://doi.org/10.1007/s00216-018-1156-x	white, transparent	pellet	bayreuth university	pol (po



pe	Material	librarytype	spectrumid	Plastic Pollution Category	plastic_or_not	material_class_specific	max_cor_class	max_
	ldpe	polymers	nist_srm_pe1474b_1s_20ac_10x_25mw	polyolefins (polyalkenes)	plastic	poly(ethylene)	polyolefins (polyalkenes)	0.989973314

We calibrated 34 standards and aim to analyse them in the samples, with a particular focus on polymers most found in wastewater treatment plants, including those used as flocculants during the treatment process

University of South Wales
Prifysgol De Cymru

Summary

We used advanced analytical techniques such as FTIR, Raman spectroscopy, and pyrolysis-GC/MS proved essential in identifying and quantifying these microplastics.

The accumulation of microplastics in swage and sludge, when used as fertilizers, and their release into aquatic environments pose serious risks to ecosystems and human health.

It is critical for future research to focus on enhancing removal technologies, improving waste management practices, and exploring sustainable alternatives to reduce the entry of microplastics into wastewater systems.

These efforts are essential for mitigating the long-term impacts of microplastic pollution on the environment and public health.


THANK
YOU