

MICROPLASTIC TESTING AT EUROFINS

Thursday November 28rd, 2024 15h10 – 15h40 (CET)

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Agenda



Eurofins Company Worldwide

Introduction

- Plastics & Microplastics
- Environmental pollution

Worldwide Eurofins Testing Sites

- Eurofins Environment Testing Norway: Joakim Skovly
- Eurofins Analytical Services Hungary Kft Hungary: Gabor Bórdos
- Eurofins SF Analytical (Food Testing lab) USA: David Riggs & Eurofins Eaton Analytical (Environment Testing lab) – USA: John Cosgrove
- Eurofins Environment Testing Australia: Bob Symons
- Eurofins BLC Leather Technology Centre UK: Peter Hughes & Eurofins Textile Testing – Spain: Enrique Rivas

Conclusions



EUROFINS, A WORLDWIDE PARTNER



Service offering of over 200,000 analytical methods in the following areas:

- + Environment Testing
- + Food & Feed Testing
- + BioPharma Services
- + Clinical Diagnostics
- + Agroscience Services
- + Genomics & Forensic Services
- + Consumer Product Testing
- + And more...



EUROFINS ENVIRONMENT TESTING



Contaminated Sites and Waste Water Testing



Water & Hygiene Testing



Air Testing



Waste, Compost, Energy & Biofuel Testing



Built Environment Testing



Special Analytics





Introduction

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Microplastics - Extensive press coverage



Awareness, policies and legislation

Environmental groups are pressuring governments and companies in order to **regulate the use, production** and **recycling of microplastics** with the goal to **reduce environment contamination** and **maintain healthy ecosystems**.

Studies also from Eurofins are launched all around the world in order to **monitor plastics** and **microplastics in the environment**.



Plastics & Microplastics



Plastics are mostly synthetic materials used in a wide variety of products coming from very different types of industries.

Plastics can be formed into:

- polyesters for use in fabrics and textiles,
- polyvinylidene chloride for food packaging,
- polycarbonates for eyeglasses and compact discs,
- Etc.





Plastics & Microplastics: Definition

9

2 Groups of Microplastics

Primary microplastics

Directly used in products *(example: microbeads in cosmetics)*

Secondary microplastics

Derived from plastic shedding (example: car tires)



Environmental pollution

Microplastics have been found everywhere in the environment:

- In water (sea, oceans, rivers, lakes, etc.)
- In cultivated soil
- In the air (as dust)

As a result, microplastics have been found in the food chain:

- Drinking and bottled water
- Marine life: shellfish, fishes, etc.
- Sea salt
- In larger animals which are contaminated by insects, which are contaminated from their own environment







Worldwide Eurofins Testing Sites

fit en naférra



Presentation of Eurofins' offer

Eurofins laboratories offering microplastic testing:

- Eurofins Environment Testing Norway
- Eurofins Analytical Services Hungary Kft Hungary
- Eurofins SF Analytical USA
 Eurofins Eaton Analytical USA
- Eurofins BLC Leather Technology Centre UK
 Eurofins Textile Testing Spain
- Eurofins Environment Testing Australia



Microplastics @ Eurofins



Sampling



100 or 300 µm mesh plastic spare parts not precise volume measurement larger organic materials caught



Customer Case Study





Technical aspect, Matrice(s) & Sampling method

Continuous sampling (trap for suspended matter)



Punctual sampling (fractioned filtration)







Eurofins Analytical Services Hungary

Instrumentation





MP workflow – Analysis



Sample preparation

- Density separation
- Alkaline and/or enzymatic digestion if needed
- Oxidation
- Filtration

Analysis

FTIR microspectroscopy (ca. 20 polymers)

- Particle numbers
- Down to 5 µm
- Polymer types
- Particle sizes
- Shapes (fragment, fibre)

Pyrolysis GC-MS (12 polymers+rubber)

- Total polymer mass
- Down to 1 µg/sample
- Polymer types



Eurofins Environment Testing Norway

Technical aspect, Matrice(s) & Sampling method

Pyr-GC-MS calibrated for 10 polymers and 3 rubber materials

Special made filtration setup in polymerdust-free lab facility.







Normativs



Test report

10 plastics PE, PP, PS, ABS, PVC, PET, PC, PMMA, PA6, PA66 and

3 elastomers BR, NR, SBR

are possible to quantify with PYR-GC-MS

ANALYTICAL REPORT

This analytical report replaces previous version of this report. Please destroy

report. AR-23-MX-014665XX

Sample code: 441-2023-0621-135		Sampled on:			
Description: Surface water		Sampled by:			
1	Client Sample: Test sample		Analysis date:		
A	Inalysis		Result	Unit	LOQ:
•	Microplastics 27	µm-1000µm			
•	Polyethylene		25.0	µg/l	0.2
	Polypropylene		61.0	µg/I	0.4
÷	Polystyrene		12.0	µg/l	0.1
	Acrylonitrile butad	lene styrene (ABS)	<0.2	μg/i	0.2
1	Polymethyl metac	rylate (PMMA)	~0.2	µg/l	0.2
•	Polycarbonate (PO	C)	<1.0	µg/i	1
1	Polyvinyl chloride (PVC)		<3.0	µg/I	3
÷ - 1	Polyethylene terep	phtalate (PET)	0.5	µg/I	0.2
	Polyamide 6 (PA6)	<0.1	μg/i	0.1
	Polyamide-6,6 (PA 66)		<1.0	µg/l	4
٩.	Sum of quantified polymers		98.5	µg/l	
	Rubberparticles I	in waste water(>27µm)			
1	Polybutadiene		<1.0	µg/I	1
1	Polylsoprene		13.6	µg/l	1
	Styrene-butadiene	rubber (SBR)	21.2	µg/I	0.2
	Evaluation of rubb	er content	Contains rubber		
			components		
0	Sum rubber comp	onents	34.8	hðy	
•	Filtered/analyzed	volume			
•	Volume		1000	mi	

Detriff	Mineralwasser still 1,51 Glas Mehrweg
Einsender	Herr Alexander Borek
Auftraggeber	Frau Heike Drinds
Eingangsdatum	17.01 2020
Probennummer Kunde	SON2019_001VP001
Beginn Analyse	17.01.2020
Ende Analyse	17.01.2020
Prüfergebnisse	

SFMP1 Methode	Mikroplastik >20 µm in souberem Wass Hausmethode, LA-IR-005, Spektropho	ker kometrie (FT-IR)
Proben	menge filbriert	1,00
Polyami	id 6 (Nylon 6, PA8) 20 - 100 µm	nicht nachweisbar
Polyam	id 6 (Nylon 6, PA6) >100 - 1000 µm	nicht nachweisbar
Polyami	id 6 (Nylon 6, PA6) >1000 - 5000 µm	nicht nachweisbar
Polycar	bonat (PC) 29 - 100 µm	nicht nachweisbar
Polycel	tionat (PC) >100 - 1000 µm	nicht nachweisbar
Polycar	bonat (PC) >1000 - 5000 µm	richt nachweisbar
Polyeth	ylen (PE) 20 - 100 µm	nicht nachweisbar
Polyety	ylen (PE) >100 - 1000 µm	nicht nachweisbar
Polyette	ylene (PE) >1000 - 5000 µm	nicht nachweisbar
Polyeth	ylecterephthalat (PET) 20 - 100 µm	2
Polyette	ylentarepinthalat (PET) >100 - 1000 µm	nicht nachweisbar
Polyeth	ylenlerephthalat (PET) >1000 - 5000 µm	nicht nachweisbar



Methodic aspects Pyr-GC-MS

Polymer	Marker compound	Marker ion (m/z)
PE	1,20-Heneicosadiene	82
PP	2,4-Dimethyl-1-heptene	126
PS	2,4,6-Triphenyl-1-hexene	91
ABS	2-Phenethyl-4-phenylpent-4-enenitrile	170
SBR	4-Phenylcyclohexene	104
PMMA	Methyl methacrylate	100
PC	Bisphenol A	213
PVC	Napthalene	128
PET	Benzoic acid	122
N6	Caprolactam	113
N66	Cyclopentanone	84
NR	Dipentene	93
BR	4-vinylcyclohexene	79



Important note: 4-vinylcyclohexene (m/z 79) is not specific for only BR, but receives also contribution for 4vinylcyclohexene from butadienemonomers in ABS and SBR



Eurofins Environment Testing Norway

Examples Pyr-GC-MS





Combination analytical methods





Commercial aspect

- Pyr-GC-MS is on the market since November 2018.
- At present time there is no accredited services available.
- Laboratories offers quantitative results as mass concentration. This to be able to meet the future demand for regulatory limits. Ability of the Pyr-GC-MS technic.
- The FTIR is by date the most popular and used method to determine presence of MP in scientific publications. This is a powerful qualitative and informative method.
- By combining the two methods this would put Eurofins in a commercial front position.



Future developments

- 1. Developing method for dirty waste water (as municipal waste water) PYR-GC-MS
- 2. Validation a new method for qualitative / quantitative determination of rubber in presence of high PP-Contents
- 3. Developing combination method FTIR / PYR-GC-MS on same filter and preparation
- 4. Establish method for determine MP in sewage sludge and sediments
- 5. Developing a transportable method for sampling MP in different environments



Eurofins Eaton Analytical and SF Analytical USA

Technical aspect: Matrice(s) & Sampling method

Matrices:

- Fresh water,
- Marine water,
- Drinking water,
- Bottled water,
- Greywater,
- Solid/liquid foods.

Sampling Methods:

- Surface waters manta net or grab sample,
- Drinking water grab sample,
- Bottled water water as bottled.







Technical aspect: Analytical method

Dye Imaging Method (SUNY) - State University of New York at Fredonia

- Relatively "clean" (drinking) water.
- Bottled water/drinking water (size < 1000 μ m).
- Nile Red dyeing, filtration, microscopic investigation.

NOAA Method (National Oceanic and Atmospheric Administration)

- Relatively "dirty" (surface) water.
- Environmental water (300 μm < Size < 5 mm).
- Chemical treatment, mass determination,
- microscopic investigation.

Hybrid of Dye and NOAA methods for foods.









Eurofins Eaton Analytical and SF Analytical USA

Technical aspect: Analytical method

- Particle enumeration and morphology shapes found:
 - fibers, fragments, pellets, foam.
- Plastic identification by FTIR



- Determine source of contamination
- Improve bottling or packaging processes





Eurofins Environment Testing Australia

- Melbourne rated "world's most liveable city" for seventh year running.
- Eurofins \$21m "state-of-the-art" laboratory completed in December 2018.
- Emerging Contaminants including Microplastics (MPs) currently being developed.
- Partnering with Agilent Technologies Australia for the development of LDIR (Laser Direct Infrared) instrument for determination of MPs





Agilent Technologies



Technical aspect: Matrice(s) & Sampling method



Analysis Methods

- Compound or stereo microscopy
- Fluorescence microscopy
- Scanning electron microscopy (SEM)
- Py-GC-MS/TGA-GC-MS
- Single point Raman IR (µRaman)
- FTIR
- ATR-FTIR
- LDIR



Eurofins Environment Testing Australia

Typical Workflow





Eurofins Environment Testing Australia

Typical Workflow







Typical Workflow





Laser Direct Infrared (LDIR)



- The Agilent 8700 LDIR reduces analysis time to only 2-60 minutes.
- LDIR particle sizes goes down to the diffraction limit, typically IR light in this region has a spatial resolution between 5-10 µm.
- The visible high magnification camera spatial resolution goes down to 1 µm.
- Intuitive visualization Clarity software also facilitates complex data interrogation and reporting, in a user-friendly way.



Eurofins Environment Testing Australia

Quality Assurance/Quality Control (QAQC)

- Laboratory design includes positive pressure and air-lock.
- Pass through locker.
- Laminar flow cabinet.
- Cotton laboratory coats.
- Nitrile gloves.
- Procedural blanks.
- Laboratory Control Samples (Sand spiked with MPs).







Eurofins Environment Testing Australia

Research Aspect

- Prof. Frederic Leusch -Toxicology Research Group at the Australian Rivers Institute, Griffith University.
- "Unwrapping our plastic present: Fate, toxicity and removal of microplastics".
- To determine the occurrence of MPs and NPs in biosolids from Australian WWTPs.





Queensland, Australia

I IrbanUtilities



Sydney

WAT &R









Eurofins BLC Leather Technology Centre UK & Eurofins Textile Testing Spain



Microplastic Testing

Overview of Eurofins | BLC microplastic testing capabilities



urofins BLC Leather echnology Centre UK

Target materials

Target Market

Garments and textile materials that have the potential to shed microplastic / microfibres during washing.

Material tested

- Textiles,
- Synthetic materials, PU,
- Leather,
- Washing cloths and sponges .





Process Overview

- Sample material is isolated then the washing process is simulated through mechanical action in distilled water with relevant controls.
- Sample material is removed from the wash solution, solution is passed through a 5 micron filter (or larger).
- Filter is weighed to determine mass of shed material per kilo of sample.
- Shed material is viewed using electron microscopy and visually analysed by technicians.





Report

Output

- Report detailing findings with commentary,
- Microscopy images.

Size of Microplastics

Filtration down to 5 micron. Following research, we have concluded that below this level it would be hard to determine microfibres shed from the sample material from background microfibre contamination.







Costs

No standard currently exists for the release of microplastics.

Therefore we recommend that benchmarking products against competitor products or other products in your range is the most valuable approach to understanding microplastic release.

- Initial setup fee for one sample against a control: unique high costs (includes report and consultancy to support understanding of results).
- Each additional sample: costs are less to factor 10 (includes report and consultancy).



urofins BLC Leather echnology Centre UK

Further Information

https://www.blcleathertech.com/what-we-do/testing/microplastics-testing-consulting Contact us at info@leathersustainability.com .





Eurofins Textile Testing Spain

Eurofins Textile & Footwear Testing Alicante



Microplastic Testing in Textiles



Eurofins Textile & Footwear Testing Alicante

No standard currently exists to quantify the amount of **microplastics/microfibers** release from fabrics.

There is some labs and garment companies with a washing machine and filtration methods. This method have very low repeatability and reproducibility. We can perform it, but not recommended.

Therefore we develop a new test methodology with a high repeatability and reproducibility. This test is based in other environmental test to quantify particles in air pollution.



Workflow



Eurofins Textile Testing Spain

Output

Report/study detailing findings with commentary.

 mg/m^2

mg/kg

Microscopy images

Filtration down to 5 micron. Following research, we have concluded that below this level it would be hard to determine microfibres shed from the sample material from background microfibre contamination.

The study included up to 5 washings.



Eurofins Textile Testing Spain

Eurofins Textile & Footwear Testing Alicante



Testing blocs

Washing simulator





Conclusions

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Possible Microplastic testing at Eurofins

	METHOD	INFORMATION OBTAINED	PROVIDED BY EUROFINS
	Light Microscopy		0
	Fluorescence Microscopy	Number eine Membelenu Coler	5
Microscopy	Scanning Electron Microscopy	- Number, size, morphology, Color	2
	Atomic Force Microscopy	-	
	Raman Spectroscopy	Type in the size range 1 - 20 um	S
Spectroscopy	Fourier Transformation Infrared Spectroscopy (FTIR)	Type in the size range 25 - 5000 um	0
	Laser Direct Infrared Imaging (LDIR) with Attenuated Total Reflectance (ATR)	Type in the size range 20 - 5000 um	0
	Pyroysis (PYR) GC-MS	Tuno ao Concentration	0
i nermai Analysis	Thermal Desorption(TD) GS-MS	Type as concentration	S



Contacts overview

Norway (Bergen), Hungary (Budapest) Water testing

Contact NO: bergen@etn.eurofins.com Joakim Skovly, BU manager www.eurofins.no/microplastic Contact HU: kornyezet@laboratorium.hu Gábor Bórdos

UK & Spain Textile shedding analysis

Contact UK: Peter Hughes <u>peter@blcleathertech.com</u> info@blcleathertech.com +44 (0)1604 679 999 **Contact Spain:** Enrique Rivas textile_spain@eurofins.com atencioncliente@iproma.com

USA (New Berlin, WI & Monrovia, CA) Water testing

Contacts: Eurofins SF Analytical (Food Testing lab) Sales & Technical: David Riggs, BS, PE & Amber Skaretka, MS Eurofins Eaton Analytical (Env. Testing lab) Env.Marketing@ET.EurofinsUS.com

Australia (Dandenong South) Water testing

Contacts: Dr. Bob Symons: <u>BobSymons@eurofins.com</u> Dr. Edward Nagul: <u>EdwardNagul@eurofins.com</u> <u>EnviroSales@eurofins.com</u>

Any Questions



