SOILveR

Soil and land research funding platform for Europe

Project MISSOURI

Microplastics in soil and groundwater: sources, transfer, metrology and Impacts

Mid-report
January 2021

Acknowledgments

We thank the board members:
Hélène ROUSSEL and Frédérique CADIERE, ADEME (France)
Gita MAAS, Ministry of Infrastructure and Water Management (Netherlands)
Esther GOIDTS, SPW-Wallonie (Belgium)

as well as all contributors solicited for the survey.

How to refer to this report

Ineris, January 2021, MISSOURI: microplastics in soil and groundwater: sources, transfer, metrology and impacts, mid-report, 39 pages.
This report is available on https://www.soilver.eu/ and https://www.ineris.fr/

Project MISSOURI is supported by:

Project leader:
French National Institute for industrial environment and risks (Ineris, France)

Project co-partners:

- Vrije Universiteit (VU, Netherlands), department Environment & Health
- Institut scientifique de service public (ISSEP, Belgium)

Funded by:

SOILveR in brief

The SOILveR platform strongly believes in the need for integrated soil and land research and knowledge exchange in Europe. We acknowledge the added value of coordinating, co-funding and disseminating cross-border soil and land management research. SOILveR is a self-financed platform. The platform members have a common interest in sharing and implementing integrated multidisciplinary research. SOILveR builds on the experiences from other funding networks such as SNOWMAN and address knowledge needs identified by e.g. the Horizon 2020 project INSPIRATION and other initiatives as well as those proposed by the members of SOILveR.

1 Table des matières

ABSTRACT

The one-year European project MISSOURI focuses on microplastics (MP) in soil and groundwater and aims at conducting a state-of-the-art review along a “sources-transfer-exposure” continuum and at participating in a European-scale interlaboratory study (ILS) in order to provide recommendations on separation and analytical methods in an idea of harmonization.

This work aims at proposing a harmonized definition for microplastics, a set of laboratory methods for the separation and analysis of microplastics in soil and at identifying priorities for future projects. It also aims at giving first recommendations for decision-making and management of soil quality regarding the potential risks associated with microplastics in soil and groundwater.

This mid-report focuses on the work that has been achieved during the first 6 months of the project:
i/ soil spiking with MP microspheres in order to provide 5 soil samples for the ILS that started in October 2020, and
ii/ survey sent to stakeholders in December 2020 in order to collect their current difficulties and concerns encountered with MP management and treatment as well as their expectations on data collection and future studies.

Abbreviations and acronyms

ADEME	Agence de l'Environnement et de la Maîtrise de l'Énergie (France)
CV	Coefficient of variation
ILS	Interlaboratory study
Ineris	Institut national de l'environnement industriel et des risques (France)
ISSEP	Institut Scientifique de Service Public (Belgium)
MP	Microplastics
PMMA	poly methyl methacrylate
PE	polethylene
PS	polystyrene
pyr-GC-MS	Pyrolyse Gazeous chromatography- Mass spectrometer
VU	Vrije Universiteit of Amsterdam, department Environment & Health (The Netherlands)

1 Context and objectives of the project

The MISSOURI project is the acronym for **MIcroplasticS in Soil and grOUndwateR: sources, transfer, metrology and Impacts**, and focuses on these anthropogenic pollutants in terrestrial media.
Microplastics in marine and surface waters have been studied for many years whereas soil and groundwater are emerging environmental compartments for undergoing studies.
"Microplastic" is a catch-all phrase for plastic particles spanning six orders of magnitude in particle size (from 0.1 to 5000 µm) and a gigantic variety of chemical compositions: (co)polymers, chemical additives, residual monomers, fillers, catalysts, non-intentionally added substances (NIAS).

This project aims at conducting a state-of-the-art review and at organizing a European-scale interlaboratory study on the determination of microplastics in soil, in order to provide recommendations on separation and analytical methods. It will also provide guidelines for policy making and future projects.

This document is a mid-term progress report that details the tasks performed from the start of the project in July 2020 to January 2021.

2 Project description

2.1 Tasks and work packages

The following table and graphic representation (Figure 1) show the MISSOURI project's 4 work packages with internal links and status:

Work packages (WP)			
No. of WP	**Title**	**Lead organisation acronym**	**Status**
1	Project Management and Coordination	Ineris	All project duration
2	State-of-the-art review	Ineris	Start in July 2020 and in progress
3	Interlaboratory study: Microplastics in soil - Preparation and analyses of microplastics in soil	ISSEP & VU	Start in July 2020 and in progress
4	Dissemination and Exploitation	Ineris	In progress

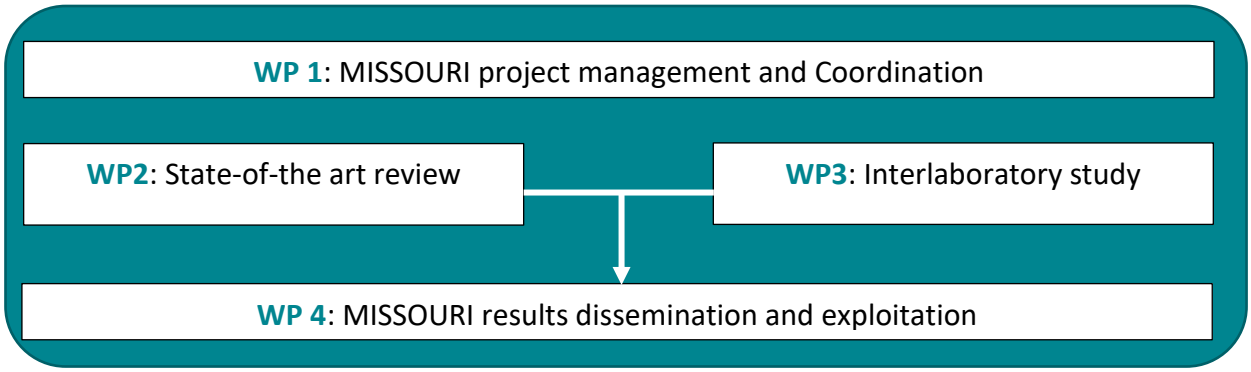


Figure 1 : Work packages in the MISSOURI project

2.2 Kick-off meeting and technical meetings

The kick-off meeting organized by SOILveR held on November, 17th 2020, after 2 postponements due to the Covid-19 context.

The first technical meeting organized by Ineris with the MISSOURI partners and funders held on December, 11st 2020.

2.3 Consortium agreement

As required, the consortium agreement was signed by the 3 partners and sent to the SOILveR secretariat in December 2020.

3 Methodology

The work that have been carried out during the first 6 months is hereafter related. Methodology is described below for each WP (WP2 to WP4).

3.1 WP2 – the state-of-the-art review

3.1.1 WP2 initial content

MISSOURI focuses on microplastics (MP) in soil and groundwater and aims at conducting a state-of-the-art review along a "sources-transfer-exposure" continuum as illustrated in the following figure:

It addresses the following topics for microplastics:

- emission sources specific to soil and groundwater;
- polymer types and their toxicity;
- existing methods for sampling and characterizing soil and groundwater;
- interactions of plastics with the terrestrial biota;
- main entry routes into environmental compartments (soil, groundwater and terrestrial food chain) and potential exposure pathways for humans;
- transport mechanisms and their scales;
- observed/expected impacts on terrestrial ecosystems and Human health.

The state-of-the-art review will provide a further understanding of microplastic sources, transfers and impacts in soil and groundwater.

3.1.2 WP2 changes

Compared to the initial proposal, changes occurred to take into account:

- As showed in Figure 2, between 2018 and 2019, there was a huge increase of published papers related to micro- and nanoplastics, thus, in agreement with the Board, the scope was narrowed to microplastics only : nanoplastics will not be tackled whereas the medium air will be considered as exposure pathway in the "Human health" topic and as a source of soil contamination to microplastics.

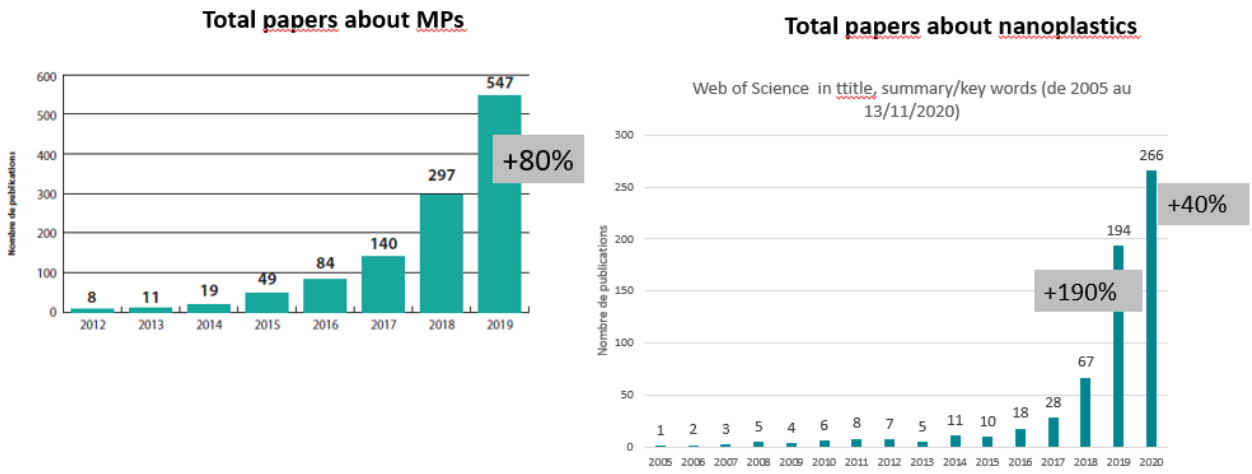
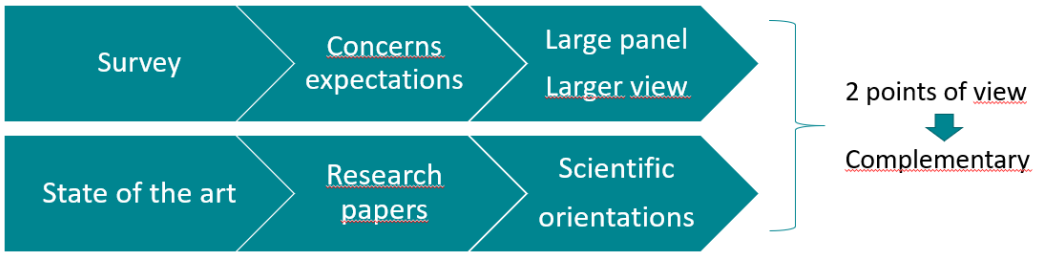


Figure 2: Temporal frequency of scientific publications

- Due to Covid-19 health context, the workshop initially scheduled at the beginning of the project was cancelled. This workshop was replaced by an on-line survey that involves experts, stakeholders and end-users in order to collect and discuss their expectations on data collection, current difficulties encountered with MP management and treatment, future studies.



3.1.3 Survey

Part of the MISSOURI project was to give its funders an overview of the general knowledge on microplastics and public expectations for future studies or legislations (Appendix 2).

To achieve this goal, a survey was designed and sent to an initial list of 257 European persons working on or interested in the subject of microplastics (Appendix 3) who consists of a compilation of the contacts given by Ineris, ISSeP, VU, Ademe, scientific articles and internet researches, both dealing with soil and water domains. It was amended with additional contacts when relevant ones where given by the persons we contacted.

The survey was designed to gather general knowledge and expectations as well as technical data (e.g. on laboratory experiments). It was designed from microplastics intrinsic properties to European legislation in order to cover the topic in its entirety:

- It first gives a synthetic explanation of the MISSOURI project, its goal and the goal of the survey. It then gathers data on the participant(s) for the project's information but also in order to determine how to conduct the survey as e.g. a laboratory person would not have the same interests or ways to answer the questions as would a policy maker;

- The survey then starts with general questions on the respondent's interest in microplastics' intrinsic properties (size, shape and definition of fractions). The last question on the definition of the fractions of plastics aims at answering one of the goals of the MISSOURI project (together with the results of the state of the art): *propose a harmonized definition of microplastics;*

- The following questions deal with the "source-pathway-receptor" continuum. They question the respondent's knowledge on the sources of microplastics and the compartments he thinks are impacted, studied and on which future researches should focus on. The respondent is then questioned on his knowledge on the impacts of microplastics on human health and the environment;

- Part of the survey focuses on analytical methods and is directed particularly towards laboratories even if the respondents were always asked if they would be able to answer this part. This parts also gathers the respondent's opinion on the ILS MISSOURI is participating in;

- The last part on policies and regulation also aims at answering one of the goals of the MISSOURI's project (together with the other tasks of the project): *identify priorities for future projects and give first recommendations for decision-making and management of soil quality.*

- The survey was then concluded by a general discussion.

It was written by Ineris, amended by ISSeP and VU then validated by Ademe before it was sent to the participants.

This list gathered different groups of people from which a selection would be invited to the workshop:

- **Associations (11)**: Non-governmental organizations and citizen associations;
- **Decision-makers (43)**: administrations, European regulators, governments, institutes for standardization, international governance, national institutions;
- **Laboratories (50)**: state-laboratories, private company-laboratories, analyses material provider, laboratories-performance control;
- **Professionals (62)**:
    - **Producers:** private company-bioplastic producer

- **Users:** private company-electricity provider, hospital, private company-plastic users, waste valorization company, waste recycling company, waste valorization company; waste treatment company,
- **Processors:** technical centres, technical center-plastic processes, water providing company,
- **Intermediate services:** competitiveness cluster, consulting company, online revue, professional association, private company-valorization, public collaborative platform,

- **Researchers (91)**: national institutes, network, research institutes, universities.

Survey conduction

The participants where solicitated to answer the survey a first time by e-mails in English and in French on December 8th 2020. Some organizations for which e-mail contacts were lacking were contacted through their internet websites: Rethink plastics, Color food, Plasticseurope and Léa Nature.

Only one contact in each organization was solicitated on December 8th. If Ineris did not receive an answer, the other contacts of that organization received an e-mail on December 21st.

On December 22nd, the persons contacted on December 8th who did not reply received a reminder.

When a person replied, a meeting was planned (between 1,5 hours to 3 hours).

The meeting started with an introduction of the MISSOURI project, then a presentation of the person conducting the meeting and of the person answering the survey. Each question was then read and answered, and discussions took place along the survey.

These meetings were also a chance to discuss possible opportunities for future collaborative work.

All participants were informed that they would be invited to the final workshop when the conclusions of the project would be presented. The outcomes regarding the respondents are presented in Section 4.

3.2 WP3 – European interlaboratory study

3.2.1 ILS presentation and organization

The consortium provided MP-spiked soil samples (see Appendix 4 for the description of spiked samples) to the **interlaboratory study (ILS) on the Analysis of Microplastics in Environmental Matrices** organized by WEPAL-QUASIMEME which covers proficiency tests related to the terrestrial environment (e.g. soil, plants, manure, compost and biomass), freshwater sediments, and the marine environment (e.g. sea water, marine sediment, biota and shellfish toxins (see Appendix 5 – round 2).

This study focuses on the characterization of microplastics in environmental matrices including soil but also tablets, sediment and fish. There is little to no standardization work taking place and existing methods have not been compared so far. The comparison of existing methods on

"reference soil samples" prepared on purpose for this project at a European scale is therefore an **innovation.**

The aim of this study is to respond to the need for quality controls at microplastics analysis laboratories. Contribution to the analysis and interpretation of results is carried out by the Vrije Universiteit of Amsterdam. The interlaboratory study is conducted in cooperation with WEPAL-QUASIMEME (https://www.wepal.nl/en/wepal.htm) to increase the number of participants. WEPAL-QUASISME is an organization that organizes for decades proficiency studies for many environmental pollutants and different matrices. The current ILS organized by WEPAL-QUASIMEME focused on sediment and fish, while the MISSOURI complements this study with soil samples. For specific environmental pollutants specific ILS rounds are organized, and often followed rounds are organized. For the MP analyses this is the second round. A round is an experimental methodology to determine reproducibility of a measurement method where tests are performed independently multiple times and the results are analyzed statistically to assess their variability. A round test provides a top-down evaluation of variability because it investigates the results directly, providing visibility of variation in results when the outcomes are produced by the different participants (Moylan et al., 2016)[1].

The ILS included five samples of soil from the MISSOURI project and other "environmental matrices" (sediments, aquatic organisms, etc.) that were prepared by WEPAL-QUASIMEME.

The participation of the consortium to the ILS will allow the identification of the most suitable analytical methods as well as provide preliminary guidance for the establishment of harmonized Standard Operational Procedures (SOP). It will also contribute to identify main knowledge gaps to be addressed in follow-up research either in the development of methods for microplastics sampling and characterization.

3.2.2 ILS calendar

QUASIMEME/NORMAN ILS (round 2):

September 2020:	Laboratories registration
October 2020:	Dispatch of test materials
January 2021:	Deadline for returning the results
April 2021:	Draft report
End of April 2021:	Final report
End of May 2021:	Second workshop and planning of round 3 that will be organized by WEPAL-QUASIMEME only, as this is outside the scope and timeline of the MISSOURI project.

[1] Moylan, S., Brown, C. U., & Slotwinski, J. (2016). Recommended Protocol for Round Robin Studies in Additive Manufacturing. *Journal of testing and evaluation*, *44*(2), 1009-1018. https://doi.org/10.1520/JTE20150317

3.2.3 Methodology for soil spiking

Soil selection

The soil spiking was realized by ISSEP during summer 2020. Two types of soils were individually mixed with white MP microspheres to provide 2 levels of difficulty for the MP separation (see Appendix 4 for more information on the composition).

- Soil A is a synthetic silica: technical Fontainebleau Sand provided by Filter Service. This sand contains no organic matter or other natural compounds that would adsorb MP on their surface. Grains size is below 350 µm, 3 times larger than microspheres. This material[2] will be used as a reference compared to soil B. The composition is described in Appendix 4.
- Soil B is a real sandy soil collected on an industrial Walloon site which is currently referenced at ISSEP (no further sampling was then organized for a sake of simplicity). The table in Appendix 4 gives its physical and chemical parameters including the organic matter level that is considered as medium to high. The sandy soil was sieved to deliver 2 fractions:
  - 250 µm in order to simplify the MP separation as soil grain size is closer to the MP size (sample more homogeneous);
  - 2 mm (fraction usually analyzed by laboratories for the analyses of chemical compounds) and should be more difficult to handle for the MP separation.

For this ILS, the presence of low concentration of PAH in soil B should not interfere with the MP separation since PAH can aggregate with MP.

Protocol of soil spiking

For the preparation of soil samples, the distributor Resch was used (photo on the left). First 200 g of soil were distributed in 10 small glass bottles. Then MP solely or mixes of MP were distributed in the same bottle. At the end, each bottle was mixed one hour in a flipper mixer (photo on the right). Regarding the 90 small bottle sampling stage, this protocol was made nine times to obtain 90 bottles by batches.

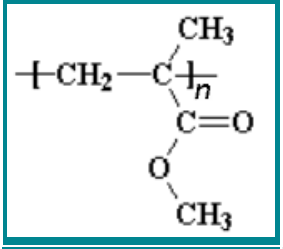
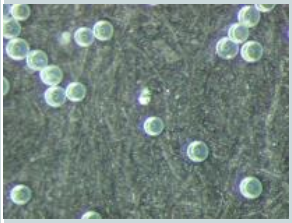
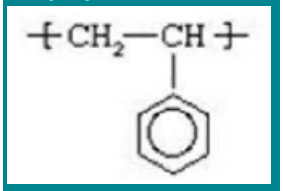
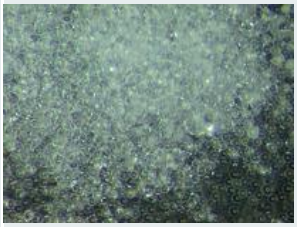
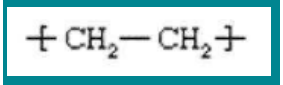
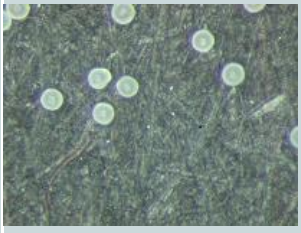
Distributor Resh

Flipper mixer

Microplastics microspheres

[2] Provision by **Cospheric Customer Service – Quotes (**quotes@cospheric.com; 805-687-3747)

The polymers were selected based on 1) the presence in the environment, 2) used in earlier interlab studies by WEPAL-QUASIMEME, and 3) the availability to buy a specific size class.
Microspheres provided by Cosphere (USA) are detailed in the following table as well as their formula, main uses, density, size used for the ILS. MP sizes are comprised between 85 and 106 µm which is the medium of MP size whose definition is comprised between 100 nm and 5 mm.

Microplastics	Type Resine code	Main uses	MP size Pictures (electronical microscopy- ISSEP)
PMMA – Polymethyl-methacrylate 	***Polysacrylics***  -others plastics	glasses (lenses), glazing, ruler, optical fiber, neon signs. Also called plexiglass	White microspheres 1.2g/cc . **90-106µm** 
Polystyrene PS 	***Polystyrenics*** 	CD cases, yogurt containers, cups, plates, cutlery, hinged takeout containers (clamshells), electronic housings, building isolation, medical products, packing, foamed coolers	White microspheres 1.07g/cc . **85-105µm** 
Microplastics	**Type Resine code**	**Main uses**	**MP size Pictures (electronical microscopy- ISSEP)**
Polyethylene PE 	***Polyolefines***  for HDPE  for LDPE	LDPE (low density) : bottles for shampoo, bags, films HDPE (high density) : rigid storage containers	White microspheres 0.96g/cc. **90-106µm** 

We failed to get PP (polypropylene) microspheres as this polymer is highly encountered in terrestrial media although several foreign providers were contacted (Cospherics LCC, Borealisgroup, Polysciences, Spherotech, Nexeo plastics). Not all polymers were available as microspheres forms with same size. This was the case for PP and PET.
The microspheres used for soil spiking were also those used for the other environmental samples in this ILS.

MP-spiked soil samples

For each batch (1 to 5), 90 bottles were prepared by ISSEP, that means that 450 bottles in total were sent in October 2020 to VU, collecting all QUASIMEME spiked samples before their dispatch to ILS voluntary laboratories. For the 1st batch, the goal was to get easy-to-measure sample. Differences between batch #1 and batch #3 are due to the small quantity of PS.

Batch number	ILS name	Matrix	Quantity of matrix in each bottle	MP microspheres	Quantity of MP in each bottle
1	QMP005SL	Sand (soil A)	20 g	PE	40 mg
2	QMP006SL	Sand (soil A)	20 g	PE	10 mg
				PMMA	15 mg
				PS	1.5 mg
3	QMP007SL	Real sandy soil 250 µm (soil B)	20 g	PE	25 mg
4	QMP008SL	Real sandy soil 250 µm (soil B)	20 g	PE	10 mg
				PMMA	15 mg
				PS	1.5 mg
5	QMP009SL	Real sandy soil (soil B) (25% 250 µm + 75% 2 mm)	20 g	PE	10 mg

3.3 WP4 – Dissemination and exploitation

Communication tasks related to the start of the MISSOURI project were conducted by Ineris, VU and ISSEP using social networks, web pages and national and international workshops as presented in section 4.

Firstly, a special webpage was elaborated in French and in English to inform about MISSOURI project and its coordination by Ineris.

Secondly, during September 2020 and February 2021, several workshops have been identified according as suitable for communicating about the MISSOURI purposes and outcomes not only at national level but also at European level.

Thirdly, for each main action of MISSOURI, the communication departments of ISSEP, VU and INERIS institutes were informed about either the process either the outcomes so that to communicate on social networks.

The planning of the final workshop and the way to organize it will be discussed after the Mid-Term report according to the main outcomes and the most important relay platforms in a context of COVID- form compliance.

The first six months were highly devoted to the soil spiking to respect the ILS calendar and to the survey in order to accommodate to the Covid context and the workshop cancellation.

4 Results

4.1 WP2 – the state-of-the-art review and the survey

4.1.1 The state-of-the art

A thorough collection of international reviews on microplastics in soil and groundwater was realized the 6th of November 2020 based on Web Of Science[3] collection of articles dated from 1956 to the beginning of November 2020. The following indices are represented in Web of Science:

- Science Citation Index Expanded (1956-current date)
- Social Sciences Citation Index (1990-current date)
- Arts & Humanities Citation Index (1990-current date)
- Conference Proceedings Citation Index- Science (2000-current date)
- Conference Proceedings Citation Index- Social Science & Humanities (2000-current date)
- Book Citation Index– Science (2005-current date)
- Book Citation Index– Social Sciences & Humanities (2005-current date)
- Emerging Sources Citation Index (2005-current date)
- Index Chemicus (1993-current date)

The following search equation was used :
“microplastic* OR "micro-plastic*" OR nanoplastic* OR "nano-plastic*"

With Indexes=SCI-EXPANDED, SSCI, A&HCI, CPCI-S, CPCI-SSH, BKCI-S, BKCI-SSH, ESCI, IC
Timespan=All years

The outcomes were 5758 found papers.

[3] https://apps.webofknowledge.com/WOS_GeneralSearch_input.do?product=WOS&search_mode=GeneralSearch&SID=E61cOlKcJjqXVYlePKw&preferencesSaved=

Further to the quantitative temporal analysis on publication numbers (cf. §3.1.1), the keywords associated to papers were analyzed with VosViewer® (van Eck & Waltman, 2010), a text-mining tool that optimizes the representation of co-occurrences between words so that to discriminate different topics. The outcomes are presented in Figure 3.

Figure 3

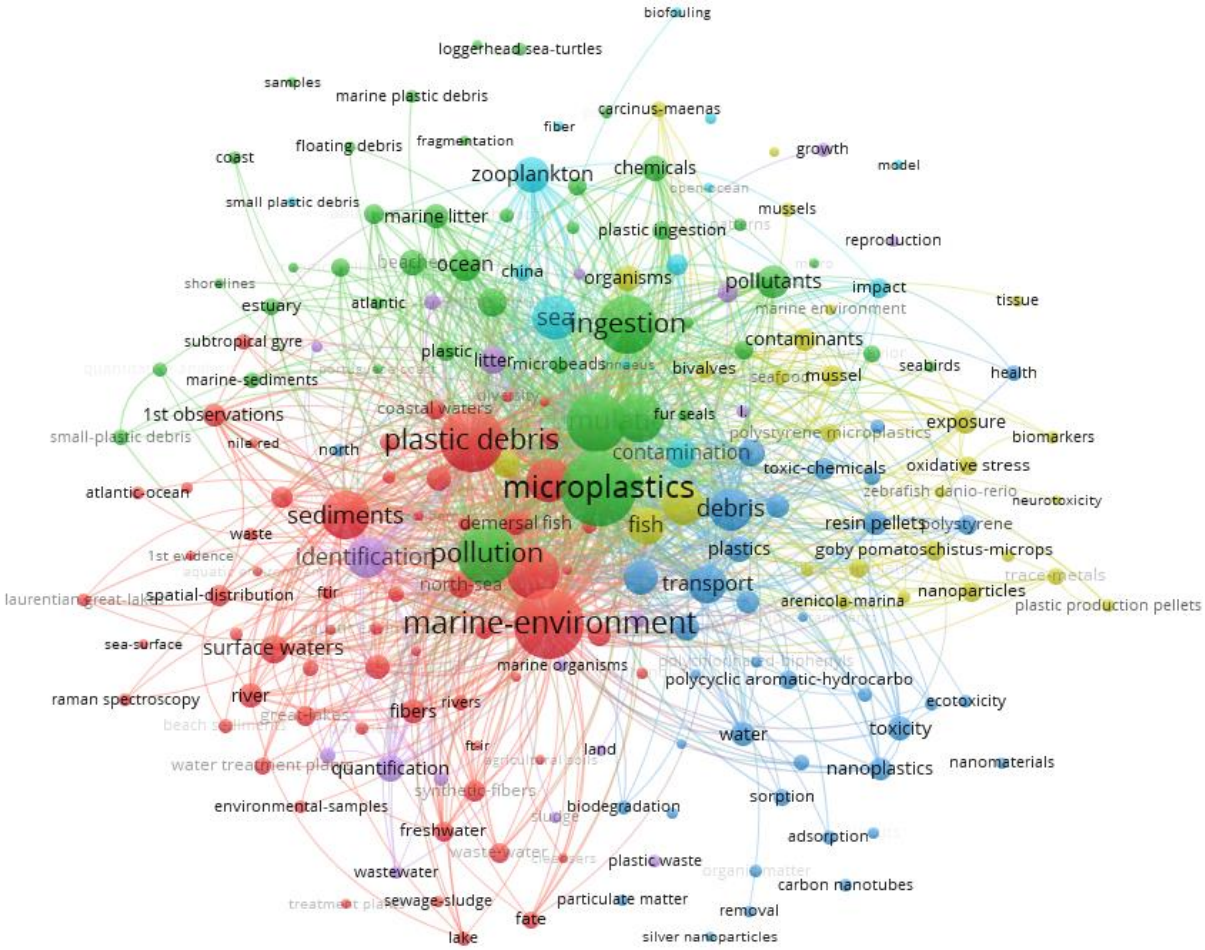


Figure 3: Links between abstract and title words

Main topics are dealing with marine environment, analytical methods for characterizing the microplastics and their additives, the ingestion by aquatic fauna and their transfer processes, the occurrence and spatial distribution in the aquatic environment, the different sources of pollutants and health and ecotoxicological models. As explained in §3.1.1, this analysis led to narrow the scope of MISSOURI on soil and groundwater exposures to microplastics, sources and transfers of pollution, impacts on ecosystems and humans. Regarding human health, the main exposure pathways through soil, water, biota and air media are included in the analysis.

An updated selection of papers was then made followed by a classification of the papers in terms of soil / groundwater exposure, microplastic fate, impacts of microplastics and chemical analysis domains. The state-of-the-art analysis will be made by using text mining (top-down approach) followed by focuses on specific topics discriminating each domain (bottom-up approach).

4.1.2 The survey

The survey was elaborated in fall 2020 and sent to a selection of 94 persons from the initial list based on their affiliation and expertise. After explaining the project in a few lines, it gathers a wide list of information through almost 30 questions as presented in section 3 (Appendix 2).

- **Respondent**: name, organization, knowledge and interest, published work etc.;
- **General expectations of MP's types/size**: which type to study in soil, which have the most impacts, which form to study and which definition of nano-, micro- and macroplastics;
- **Sources of MPs**: which sources are the most concerning and which should be studied;
- **Environmental compartments (media)**: which are the most impacted, which are most studied, which should be studied;
- **Impacts of MPs/risk assessments**: expectations for future researches for human health, and food chain, on terrestrial ecosystems etc.;
- **Analytical methods of MPs in soils**: what is the challenge when analyzing MPs in soils and other media, which methods of extraction, identification and quantification, benefits/drawbacks etc.;
- **Policies/regulation**: knowledge on European legislation, is there a road map in their country, what they expect from European and national policies and guidance.
- **Conclusion**: free discussion

To this date, 19 surveys were answered, most of them during a meeting with Ineris (one was answered by 2 organizations working together on a microplastics project) :

- 1 association
- 8 researchers
- 9 professionals
- 1 decision-maker
- 1 laboratory

LAST NAME	NAME	Contact	Organization	Country	Stakeholder
VONK	Sophie	sophie@plasticsoupfoundation.org	Plastic soup foundation	Netherlands	Association
DEPORTES	Isabelle	isabelle.deportes@ademe.fr	ADEME	France	Researcher

QUIK	Joris	joris.quik@rivm.nl	RIVM	Netherlands	Researcher
FLORIAT	Muriel	mfloriat@amorce.asso.fr	AMORCE	France	Professionals
SWEETLOVE	Cyril	csweetlove@rd.loreal.com	L'Oréal	France	Professionals
BROUSSARD	Orianne	orianne.broussard@citeo.com	CITEO	France	Professionals
KOOLS	Stefan	Stefan.Kools@kwrwater.nl	KWR Water	Netherlands	Researcher
TIREZ	Kristof	kristof.tirez@vito.be	VITO	Belgium	Researcher
RE	Viviana	viviana.re@unipi.it	University of Pisa	Italy	Researcher
MARCHAL	Romain	romain.marchal@spge.be	Société Publique de Gestion d'Eau (SPGE)	Belgium	Decision-maker
BRUZAUD	Stéphane	stephane.bruzaud@univ-ubs.fr	Université Bretagne Sud	France	Researcher
MORTAS	Nicolas	nicolas.mortas@organeo.com	ORGANEO	France	Professionals
THEVENIN	Nicolas	nicolas.thevenin@rittmo.com	Rittmo	France	Researcher
COLPAERT	Romain	romain.colpaert@gmail.com	UMR CNRS de Besançon	France	Researcher
COPIN	Dalyal	d.copin@irmatech.com	IRMA	France	Professionals
LEPROND	Hubert	hubert.leprond@edf.fr	EDF	France	Professionals
SAUR	Thibaut	thibaut.saur@suez.com	SUEZ	France	Professionals
TARCHALSKI	Christelle	christelle.tarchalsk@arteliagroup.com	Artelia	France	Professionals
DUFRESNE	Elsa	elsa.dufresne@ecogeos.fr	ECOGEOS	France	Professionals
REIBER	Jens	Jens.reiber@wessling.de	Wessling	Germany	Laboratory

Most of the respondents were researchers and professionals. Regarding the synthesis of the survey, the main representations will be taken into account by weighting the different views.

The results will be compiled in the final report and general trends will be drawn when possible.

4.2 WP3 – European interlaboratory study

4.2.1 MP-spiked soil samples

Soil samples underwent electronic microscopy shoots in December 2020 (Figure 4). Pictures of both soils before and after spiking are also included in Figure 4.

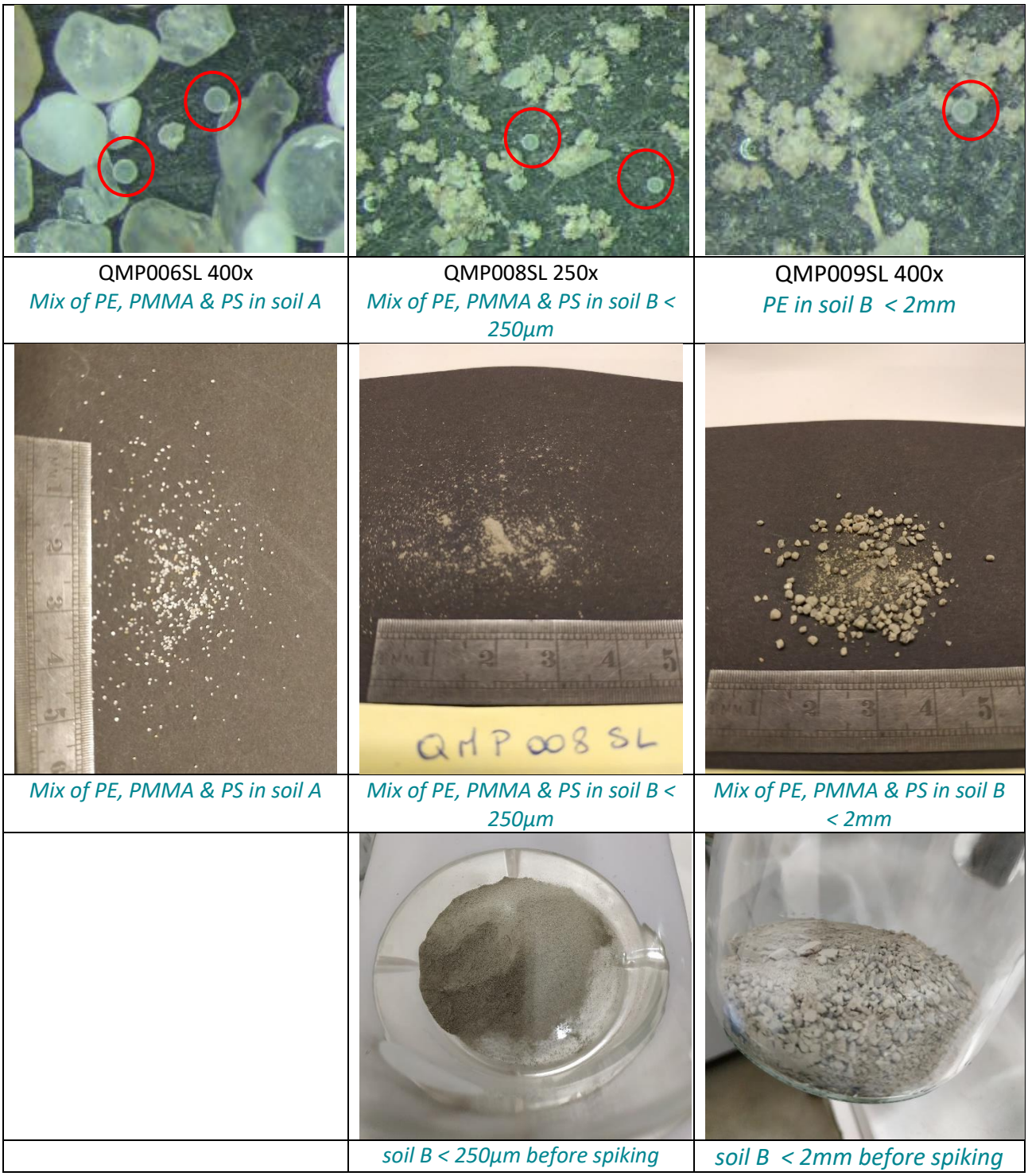
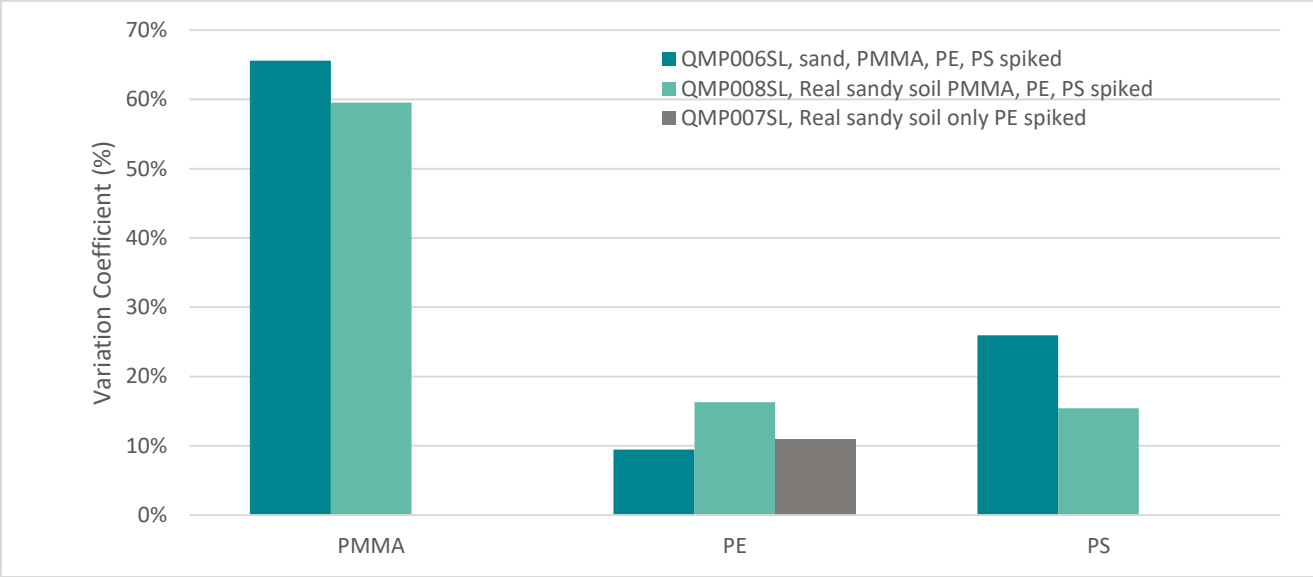


Figure 4 : Soil A and B before and after spiking with microplastics microspheres

4.2.2 First homogeneity test

To determine if the MP particles were homogeneously distributed in the soil spiked samples, eight samples from each matrix were analysed by pyrolysis GC-MS (pyr-GC-MS) after the MPs were extracted from the whole sample by Accelerated Solvent Extraction (ASE). This test was done by VU in December 2020.

The coefficient of variation (CV) of three samples are given in Figure 5. Firstly, the CVs for each MP vary within the same range between the sand (soil A) and the real sandy soil (soil B). Secondly, the homogeneity of the spiked PE and PS particles is satisfactory but seems high for the PMMA particles. It is currently unclear why the CV of PMMA is much higher than the PE and PS. One option could be that PMMA particles are partly bound to the glass wall of the bottles as PMMA particles could have an electric charge. A large part of the PMMA particles are bound to the glass wall of the sample bottles. This is an interesting result which will certainly be included in protocols and ILS studies. Despite the heterogeneity of the sample preparation methods (PMMA bound to glass wall), the samples were still sent to the ILS participants as we expected that the samples are homogeneous as they were spiked at the same moment as the PS and PE particles.



Sample QMP006SL: soil A sample spiked with PMMA, PE and PS. Sample QMP008SL: soil B spiked with PMMA, PE, and PS. Sample QMP007SL : soil B spiked with PE particles.

Figure 5 : Coefficient of variation (%) of the MP analysis in the spiked soil samples used for the interlaboratory study

Based on the current data, the spiking was successful and the soil samples are homogenous.

4.2.3 ILS voluntary laboratories

In total 58 laboratories participated in the interlaboratory study of which 33 are from Europe, 19 from America, 4 from Asia, and 2 from Oceania. For the 3 funders countries involved in the MISSOURI project (France, Belgium and the Netherlands), there are 11 laboratories. In total, 12 out of 27 European countries participated the ILS round 2 (all the countries will be reported in the final report).
The number of laboratories per country are given in Figure 6, which range from 1 to 5. No detailed information on the laboratories can be given since this information is confidential.

To this date, separation and analytical methods used by laboratories are not known. They will be completed when results will be sent to WEPAL-QUASIMEME through the electronical forms and that will be reported in the final document after further data processing.

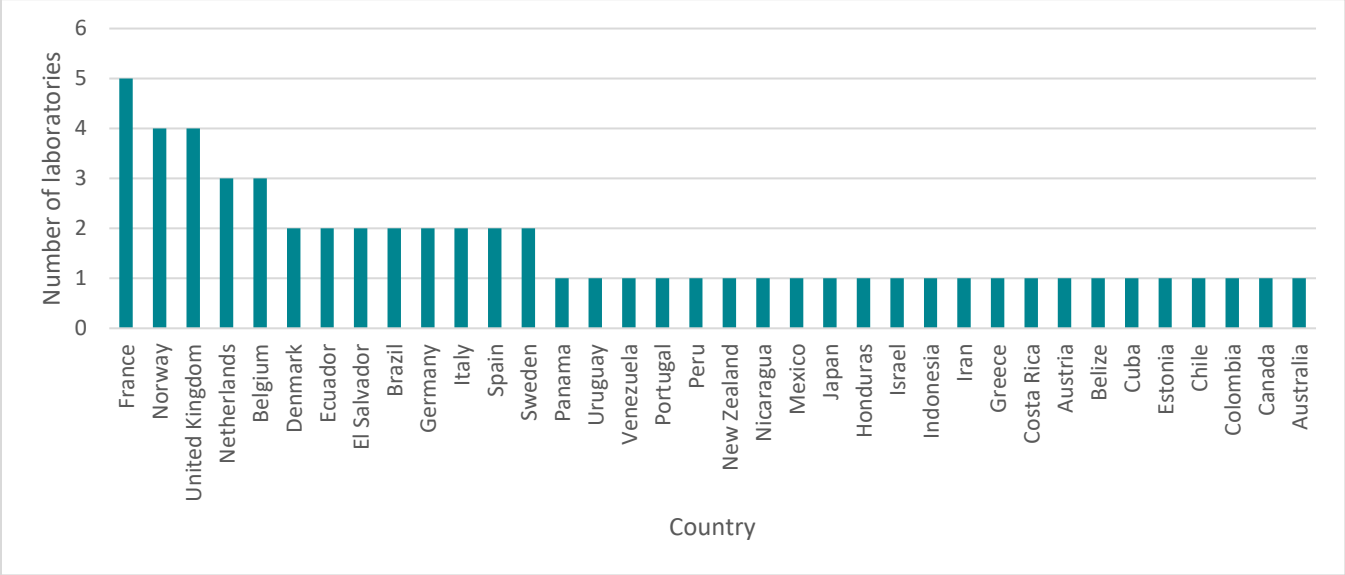


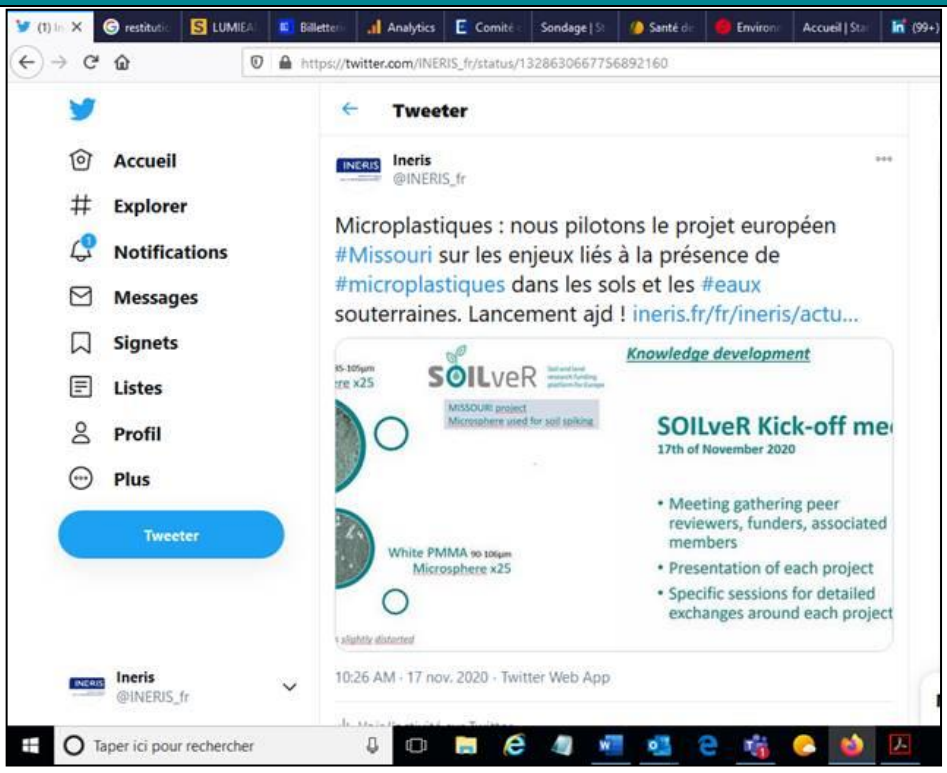
Figure 6 : Number of laboratories per country participating in the interlaboratory MP study (round 2)

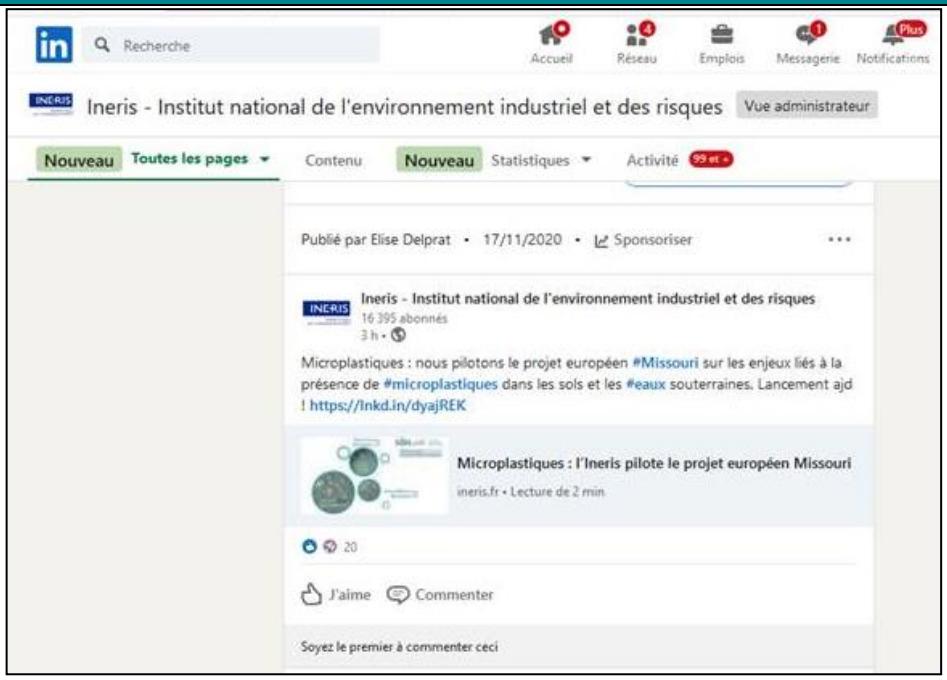
4.3 WP4 – Dissemination and exploitation

The following table shows i/ partners' webpages dedicated to the MISSOURI project (in addition to the SOILveR webpage), ii/ pushes done on social network to disseminate the start of the project:

November 2020 : SOILveR Kick-off meeting

- ***Tweeter***



	• ***LinkedIn*** 
Ineris wepages Nov 2020	https://www.ineris.fr/fr/ineris/actualites/microplastiques-ineris-pilote-projet-europeen-missouri https://www.ineris.fr/en/ineris/news/microplastics-ineris-leads-european-missouri-project
ISSEP webpages Dec 2020	https://www.issep.be/events/event/enjeux-lies-a-la-presence-de-microplastiques-dans-les-sols-et-les-eaux-souterraines/

Besides these actions, the MISSOURI project was also presented by Ineris to its Committee for Orientation of Research & Expertise (CORE[4]) on November 5th, 2020 as microplastics are identified as a global and transverse topic on which Ineris wants to mobilize its means and expertise. CORE brings society expectations and knowledge into the scientific challenges and issues faced by Ineris experts, in order to strengthen the Institute's strategy development.

[4] https://www.ineris.fr/en/ineris/building-dialogue-civil-society/building-dialogue-civil-society-our-approach

The project should be presented to:

- French Technical day dealing with polluted soil, initially forecasted in June 2021 and postponed in November 2021, organized by Ineris on behalf for the French Ministry of Environment (Paris, France). The initial meeting planned on November 2020 was postponed to the Covid-19 context;
- The 3rd international workshop on Emerging policy challenges on New SOil contaminants (ENSOr), online event, May 6th & 7th, 2021.

5 Deliverables

The following table shows all the deliverables of MISSOURI project:

Deliverables (D)			
No. of D	**Title**	**Work package No.**	**Date**
L1	Project mi-term report	WP1	January 2021
L2	Workshop summary *(cancelled and replaced by survey)*	WP4	/
L3	Pedagogic Brochure including further scientific and policy action recommendations	WP4	July 2021
L4	French Polluted sites management technical day (oral presentation)	WP4	November 2021
L5	Final report including the state-of-the-art review and the interlaboratory study (soil samples)	WP2 & 3	July 2021

6 Conclusion

The first six months were highly devoted to the soil spiking to respect the ILS calendar and to the survey in order to accommodate to the Covid context as the initial workshop was canceled.

The mid-report focused on work related to:
i/ the soil spiking with MP microspheres in order to provide 5 soil samples for the ILS that started in October 2020 (WP3), and
ii/ the survey sent to stakeholders in December 2020 in order to collect their current difficulties and concerns encountered with MP management and treatment as well as their expectations on data collection and future studies (WP2).

7 Index of figures

8 Appendices

Appendix 1 : Presentation Materials of the kick-off meeting

SOILveR Soil and land research funding platform for Europe

MISSOURI
MIcroplasticS in Soils and grOUndwateR: sources, transfer, metrology and Impacts

1

SOILveR Soil and land research funding platform for Europe

Consortium:

- Project leader:

INERIS maîtriser le risque pour un développement durable

French National Institute for industrial environment and risks (Ineris, France)

- Project co-partners :
  - Vrije Universiteit (VU, Netherlands), department Environment & Health
  - Institut scientifique de service public (ISSEP, Belgium)

Funded by:

ADEME Agence de l'Environnement et de la Maîtrise de l'Energie

Wallonie service public SPW

Ministry of Infrastructure and Water Management

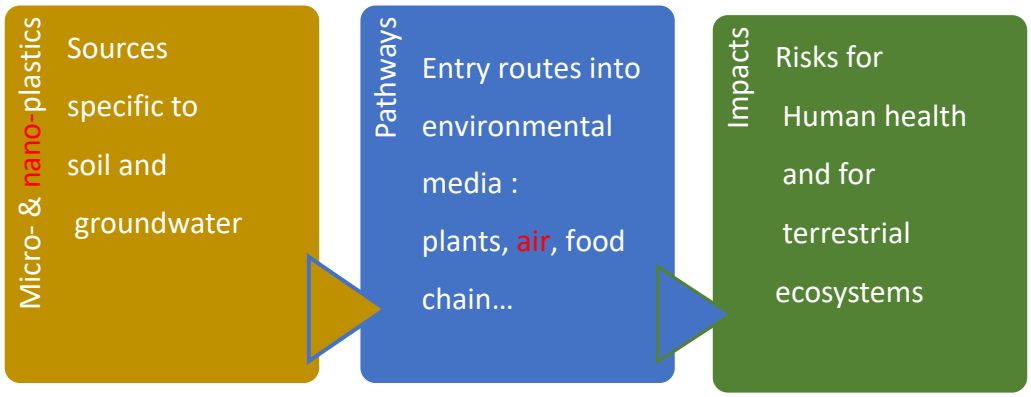
2

Aim/goals of the project

- MISSOURI answers to IRT14's topic: *Emerging contaminants in soil and groundwater*
- Goals:
  - Review laboratory methods for the separation and analysis of microplastics (MPs) in soils thanks to an ILS
  - Propose a definition for microplastics
  - Collect stakeholders/end-users' expectations for MPs' studies
  - Identify priorities for future projects for MPs
- 2 phases to respond to these goals:
  - **A state-of-the-art review**
    - Microplastics
    - Soil and groundwater
    - Types, emission sources, composition, transport mechanisms, foodchain, distribution, impacts on terrestrial ecosystems and human health
  - **A European-scale ILS** (*Interlaboratory study*)– separation and characterization methods – MPs in soils
    - Led by WEPAL-QUASIMEME
    - About 60 laboratories involved

3

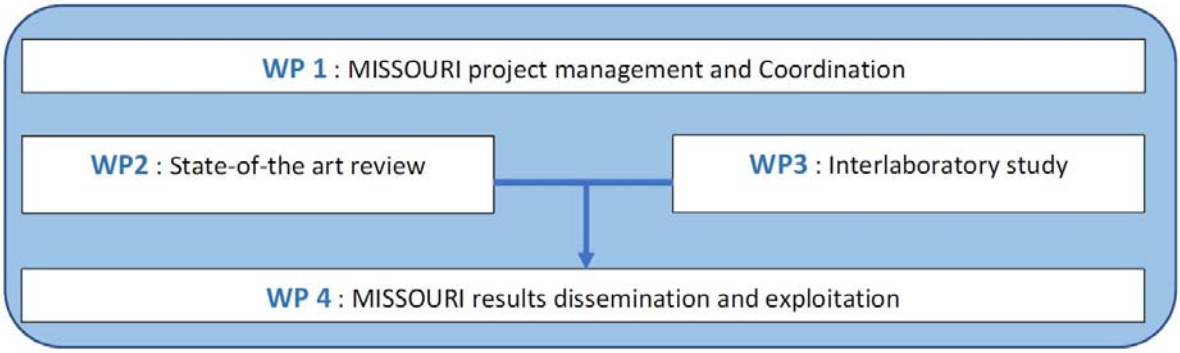
Aim/goals of the project



4

SOILveR Soil and land research funding platform for Europe

Overview of the project program (WP)



5

SOILveR Soil and land research funding platform for Europe

Challenges for the feasibility of the project

- Many recent studies, many types of MPs and many emission sources:
  - → need to narrow the scope to subjects/studies with enough reliable data
  - → need to identify issues with lack of data
- Overview of entire conceptual model: from sources, through exposure pathways to impacts on humans and ecosystems
- Participation to QUASIMEME/NORMAN Interlaboratory study (ILS) on the Analysis on Microplastics in Environmental Matrices
  - restricted to 2 types of soils
  - restricted to 3 types of plastics according to 2 modalities : i/PE; ii/mixture of PE, PMMA, PS
- Organisation of the workshop:
  - Workshop 1 (project's kick-off): replaced by survey → revision of its scope and goals
  - Workshop 2 (restitution): depends on survey and overall fate of MISSOURI

PE: polyethylene; PMMA: polyméthacrylate de méthyle; PS: polystyrene

6

Challenges for the feasibility of the project

Remaining questions:

- Sampling and characterization methods applicable to other types of soils?
- How do the forms of microplastics (bead, foam etc.) affect those methods?
- What is needed before conducting risk assessments for humans and ecosystems (parameters, other studies)?

7

Project results

- Mid-term report (before january 15th 2021)
- Pedagogic synthetic brochure on the state-of-the art review for a broad audience
- Oral presentations during next technical days in France
- Final report (spring 2021)
  - ILS's results (round 2 WEPAL-QUASIMEME): results interpretation in link with other media (sediments)– may 2021
  - Survey analyse: concerns and expectations from producers, end-users, researchers, environmental associations etc.
- Final workshop

8

Stakeholder / end-user involvement

- Survey:
  - Fall 2020
  - Submitted to a wide panel: laboratories, academics, national institutions, NGO, european regulators, private companies, water providing/treatment companies etc.
- French Polluted sites management technical day (june 2021)
- Internet, social networks
- Presentation and involvment to Ineris' CORE (French expertise and research's orientations commission), on November 5th, 2020

9

How will the results be put to use? Follow up of the project

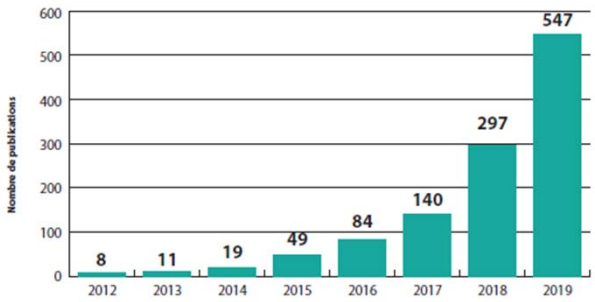
- All deliverables available on SoilveR and partners' website
- Response to Horizon 2021 calls on microplastics
- Results available to NORMAN network and WEPAL-QUASIMEME

10

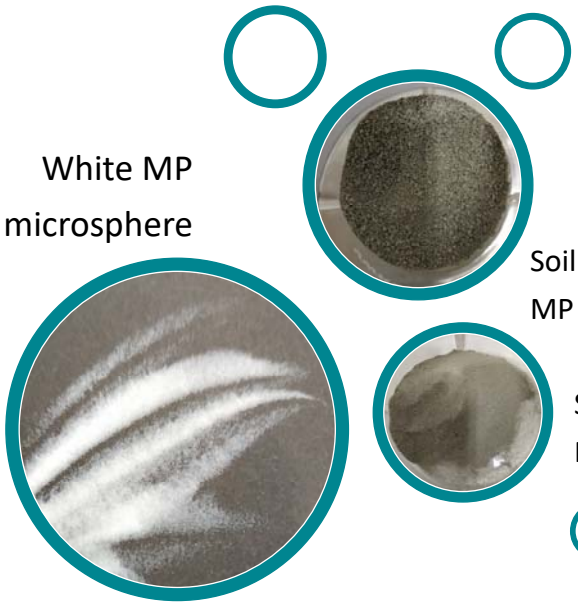
Overview of National or international projects in this research area

Title	Source
GRAINE : identifying needs to assess environmental impact of agricultural organic products spreading (occurrence and ecotoxicity of microplastics in soil, in compost and in digestate) and to understand the fate of biosourced plastics into the environment	ADEME (Ineris; on-going)
Microplastics in continental surface waters	AQUAREF (Ineris, Laboratoire national de métrologie et d'essai (LNE) and Ifremer)
MICROPLAST (presence and the ecological impacts of micro-plastics in river systems in Wallonia)	ISSEP
PLASTI-SOL (development of separation methods for micro-plastics in solid matrices)	
MICROPLASTSOIL (characterization methods adapted to agricultural soils fertilized with recycled organic sludge)	
PETHUNT (Novel method for identification and quantification of PET microfibres)	VU-EH
LIMNOPLAST (Microplastics in Europe's Freshwater Ecosystems: From Sources to Solutions)	
Method development for the identification of nanoplastics	Institute of Geography, University of Bern
Microplastics in roadside soils	
Synergies Between Microplastics and Pesticides in the Terrestrial Environment	Frontiers in Environmental Science
Fate of microplastics in groundwater: Assessing vulnerability of drinking water supplies and coastal environments	University of Edinburgh
Waste Plastics and Micro-plastics: Their Effects on the Terrestrial Environment	Future Directions International
Optimising the process for microplastic analysis	Institute of Environmental Science and Research (ESR), New Zealand

11



12



SOILveR Soil and land research funding platform for Europe

QUASIMEM/NORMAN ILS (round 2):
Sept 2020 : Laboratories registration
Sept 2020 : dispatch of test materials
Jan 2021: deadline for returning results
April 2021: draft report
End of April 2021: final report
End of May 2021: second workshop and planning round3

13

Appendix 2 : Survey

MISSOURI

(MIcroplasticS in Soil and grOUndwateR: sources, transfer, metrology and Impacts)

Survey

MISSOURI is a one-year European project funded by the SOILveR platform. Previously SNOWMAN, the SOILveR platform funds cross-border soil and land management research projects. MISSOURI responds to IRT14 call's topic: *Emerging contaminants' in soil and groundwater – ensuring long-term provision of drinking water as well as soil and freshwater ecosystem services*.

MISSOURI is a partnership between VU (Vrije Universiteit) in Amsterdam (the Netherlands), ISSeP (Belgium) and Ineris (France) and aims at answering research gaps in microplastics (MPs) characterization in terrestrial media. Its goal is to give a better understanding on the issue of MPs through a state-of-the-art review and an interlaboratory study of MPs in soils.

This survey is part of the project's launch as it aims at collecting concerns and expectations of a wide range of persons: from producers, users, researchers, to associations. Together with the results of the project, it will be used as a compass to propose future actions for decision-makers. Its results will be presented during a workshop closing the project, planned during the 2nd semester of 2021.

Respondent

- Date :
- Name :
- Organisation's name (private or governmental?) :
- Country :
- Title :
- Field of expertise
    - Scientist (public/private)
    - Laboratory (private/public)
    - Plastic producer
    - Civil society
    - Policymakers
    - Association
    - Other
- Describe briefly your knowledge of MPs and related issues

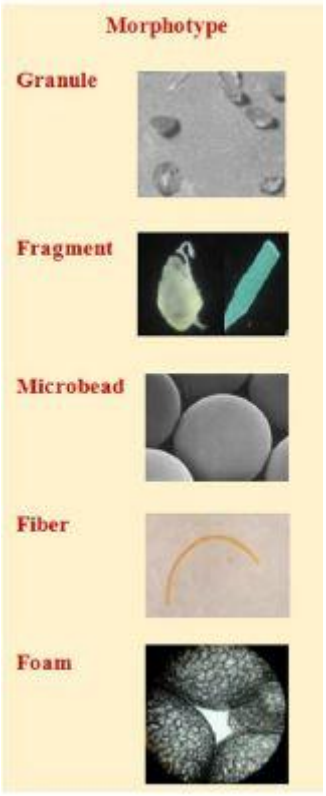
- Published work on MPs ?

- Will you be interested in attending the MISSOURI final workshop in 2021?

General expectations of MP's types/size

1. Which type(s) of plastic(s) would you study in priority in soils. Please explain:
    - PE (Polyethylene)
    - PP (Polypropylene)
    - PET (Polyester or Polyethylene terephthalate)
    - PS (Polystyrene)
    - PVC (Polyvinyl chloride)
    - PA (Nylon or polyamide)
    - Mixtures ?
    - Others (PMMA, ABS, PU, PLA[1] etc.) ?

2. Which types of plastics have the most impacts according to you?



3. Which form(s) of microplastics would be most relevant to study, according to you? Please explain:
    - Granule
    - Fragment
    - Microbead
    - Fibers
    - Foam
    - Others ?

4. Which fraction(s) of microplastic(s) would you be most interested in?

Do you agree with the following definitions? If not, please explain.

- Nanoplastics (< 100 nm)
- Microplastics ([5mm – 100 nm])
- Macroplastics (> 5mm)
    - Mesoplastics ([25 mm – 5 mm])

[1] PMMA: Polymethyl methacrylate; ABS: Acrylonitrile-butadiene-styrene; PLU: polyurethane; PLA: polylactic acid

Sources of MPs

5. According to you, which sources of emissions of MPs are the most concerning for terrestrial environments?

6. According to you, which ones should be studied in priority?
   Sources:
   - Plastic mulching
   - Land application of sewage sludge
   - Compost and fertilizers
   - Soil amendments
   - Wastewater irrigation
   - Washing machines (microfibers)
   - Vehicles transport (abrasion of tires)
   - Atmospheric deposition
   - Microplastics from macroplastics lanfilling
   - Cosmetics
   - Other

Environmental compartments (media)

7. Which compartments are, according to you, most impacted by MPs pollutions (rank)?
   - Surface waters
   - Oceans/seas (marine waters)
   - Groundwaters
   - Soils
   - Air
   - Plants
   - Terrestrial animals
   - Aquatic animals
   - Humans
8. Which compartments are, according to you, most studied (rank)?
   - Surface waters
   - Oceans/seas (marine waters)
   - Groundwaters
   - Soils
   - Air
   - Plants
   - Terrestrial animals
   - Aquatic animals
   - Humans

9. Which compartments should future research focus on, according to you?
    - Surface waters
    - Oceans/seas (marine waters)
    - Groundwaters
    - Soils
    - Air
    - Plants
    - Terrestrial animals
    - Aquatic animals
    - Humans

Impacts of MPs/risks assessments

10. What do you expect from future research on the impact of MPs on human health:
    - data on the concentrations of MPs in human biological matrices
    - data on the concentrations of MPs in food, inhaled air, drinking water, cosmetics etc.
    - types of MPs analyzed in the human body
    - types of MPs analyzed in human intakes
    - how MPs metabolize in the human body
    - how they impact human health / which effects they have on the human body
    - threshold values
    - Others

11. Do you expect studies on the human food chain (edible fruits and vegetables, animal feed, livestock etc.)?

12. What do you expect from future research on the impact of MPs on terrestrial ecosystems. Precise the considered ecosystem:
    - data on the concentrations of MPs in ecosystems/which ones?
    - types of MPs analyzed in ecosystems
    - how they impact those ecosystems/ which effects
    - threshold values
    - others

Analytical methods of MPs in soils

Questions directed particularly towards analytical laboratories

13. What is, according to you, the biggest challenge when analyzing MPs in soils?

14. What is, according to you, the biggest challenge when analyzing MPs in other media? Precise the medium (groundwater, surface water, sediments, marine water, air, sols, plants, biota, other)

15. If you practice MPs analyzes, which method do you use (non-exhaustive list)? Precise the medium(a)
    - Extraction

    Removal of the mineral fraction:
    - Manual extraction
      - visual sorting
      - microscope
    - Electrostatic extraction
    - Oil extraction
    - Density separation (using: NaCl, NaI, $Na_6[H_2W_{12}O_6]$, $ZnCl_2$, NaBr, others etc.)
    - Froth flotation
    - Magnetic extraction
    - Vertical density gradient separation
    - Other

    Removal of the organic fraction
    - Acid and alkaline digestion
    - Oxidisation with hydrogen peroxide
    - Enzymatic digestion
    - Identification and quantification
      - Visual identification
      - Chromatography
        - HT-GPC (High temperature gel-permeation chromatography)

        For identification of polyolefins in cosmetics
        - SEC (size-exclusion chromatography) and Pyr GC-MS (pyrolysis gas chromatography mass spectrometry)

        For identification and quantification of PS and PET in soil samples
        - TED GC-MS (thermal extraction desorption gas chromatography mass spectrometry)

- TGA (Thermogravimetric analysis)
- Vibrational spectroscopy
  - Raman of Fourier transform infrared (FTIR) spectroscopy

  Precise identification of polymer types, their abundance, shape and size
  - PFE (pressurized fluid extraction) coupled with ATR-FTIR
  - SWIR (short wave infrared spectroscopy)
- ^{1}H NMR (Proton nuclear magnetic resonance spectroscopy)
- In-situ identification
  - NIR (near infrared)
  - Hyperspectral imaging[2]

16. If you use a different method, can you describe it and its benefits/drawbacks?

17. Among the methods listed previously (or others not listed), do you have feedbacks on their effectiveness and benefits/drawbacks? Would you advise against one of those methods? Please explain

18. Did you face any failure with one or more of the methods you have tested?

19. What would you think of an ILS (interlaboratory study) of soils spiked with MPs in which laboratories are free to choose their analytical method?

[2] Shan, J.; Zhao, J.; Liu, L.; Zhang, Y.; Wang, X.; Wu, F. (2018). A Novel Way to Rapidly Monitor Microplastics in Soil by Hyperspectral Imaging

20. What kind of results would you expect from such an ILS?

21. Do you/does your organization plan(s) on purchasing an analytical item to separate/identify/count MPs? Are there any obstacles to that purchase?

Policies/regulation

22. What do you know about European legislation on MPs?
    - Are there European directives on MPs?
    - Do you know the regulatory institutions dealing with MPs issues?
    - Do you know if there has been a legislation on the reduction/ban of MPs? Which MPs?

23. Does a legislation and/or a roadmap on MPs exists in your country? If so, please provide references and explanations

24. What do you expect from European future policies on the intentional use of MPs? Clarification/incentive/Restrictions/ban?

25. What do you expect from European future policies on the unintentional release of MPs? Clarification/incentive/Restrictions/ban?

26. What do you expect from your country's future policies on MPs? Clarification/incentive Restrictions/ban?

27. Do you expect guidance on (precise if you expect national or European guidance): *characterization, limits and toxicology*:
    - Limit values of MPs in media. Which ones?
    - Toxicological values for health risk assessments
    - A ban of the MPs identified as most hazardous for human health? For ecosystems?
    - A compilation of MPs with characteristics (such as: sources, effects on human health and ecosystems, most impacted media etc.)
    - Others

28. Do you expect guidance on (precise if you expect national or European guidance): *solve the problem*:
    - How to mitigate MPs in everyday life
    - How to remove MPs from waste, effluents, products (WWTP sludge, waste storage facilities, wastewater etc.)
    - How to recycle MPs from waste products
    - others

Conclusion

Free discussion

Appendix 3 : list of persons contacted to answer to the survey

LAST NAME	NAME	Contact	Organization	Country
DEPORTES	Isabelle	isabelle.deportes@ademe.fr	ADEME	France
GUESNEY	Stéphane	s.guesney@adivalor.fr	ADIVALOR	France
CATTOIR	Sofie	s.cattoir@vmm.be	Agence de l'Environnement de Flandres	Belgium
CHARPENTIER	Ronan	ronan.charpentier@associationaglae.fr	AGLAE	France
DUTILLY	Gaëlle	gaelle.dutilly@association-aglae.fr	AGLAE	France
FLORIAT	Muriel	mfloriat@amorce.asso.fr	AMORCE	France
DUFLOS	Guillaume	Guillaume.DUFLOS@anses.fr	ANSES	France
MONLAU	Florian	florian.monlau@apesa.fr	APESA	France
TARCHALSKI	Christelle	christelle.tarchalsk@arteliagroup.com	Artelia	France
CHARBONNIER	Céleste	celeste.charbonnier@astee.org	ASTEE	France
JONES	Davey	d.jones@bangor.ac.uk	Bangor University	UK
CHESNEL	Ewen	ewen.chesnel@basf.com	BASF France	France
GIUDICELLI	François	francois.giudicelli@saria.fr	Bionerval-SARIA	France
NOUBETRA	Abdelkader	aboubetra@bipea.org	BIPEA	Europe
ZIEGLER	Eric	eziegler@bipea.org	BIPEA	Europe
GRILLON	Sandra	sgrillon@car-analyse.com	CARSO	France
GROULT	Roger	roger.groult@car-analyse.com	CARSO	France
GUY	Benoît	guyb@car-analyse.com	CARSO	France
LELAN	Maet	maet.lelan@bretagne.chambagri.fr	Chambre d'agriculture Bretagne (CRAB-SEHBS)	France
LIAPIS	Eleni	eliapis@chu-besancon.fr	CHU Besançon	France
BONNIER	Sophie	sophie.bonnier@citeo.com	CITEO	France
BROUSSARD	Orianne	orianne.broussard@citeo.com	CITEO	France
ORLIK	Rose	rorlik@clientearth.org	CLIENTEARTH	UK
RICHAUME	Agnès	agnes.richaume@univ-lyon1.fr	CNRS	France
ESTORGES	Marie		Color foods	France
MICHON	Cadice		Color foods	France
DOLE	Patrice	pdole@ctcpa.org	CTCPA	France
MARCO	Julie	julie.marco@danone.com	Danone	France
ACIKALIN	Véronique	veronique.acikalin@eaudeparis.fr	Eau de Paris	France
ESCUDEIRO	David	david.escudeiro@eaudeparis.fr	Eau de Paris	France
DUFRESNE	Elsa	elsa.dufresne@ecogeos.fr	ECOGEOS	France
MARCOUX	Marie-Amélie	marie.marcoux@ecogeos.fr	ECOGEOS	France
MATIAS-MENDES	Marta	marta.matias@ecogeos.fr	ECOGEOS	France
RATEAU	Fanny	fanny.rateau@ecostandard.org	ECOS	Belgium
VINCENT-SWEET	Pénélope	pvincentsweet@gmail.com	ECOS	Belgium
LEPROND	Hubert	hubert.leprond@edf.fr	EDF	France
RONGA	Sylvaine	sylvaine.ronga-pezeret@edf.fr	EDF	France
CHAUVIN	Delphine	delphine.chauvin@ehesp.fr	EHESP	France
LE BOT	Barbara	barbara.lebot@ehesp.fr	EHESP	France
MERCIER	Fabien	fabien.mercier@ehesp.fr	EHESP	France
LHERMITTE	Rozenn	projet3p@epab.fr	EPA Baie de Douarnenez	France
MARMO	Luca	Luca.Marmo@ec.europa.eu	European commission	Europe
-	-	fnccr@fnccr.asso.fr	FNCCR	France
MARSCHALEK	Regina	regina.marschalek@wien.gv.at	Government	Austria
NUUTINEN	Jari	ari.nuutinen@ymparisto.fi	Government	Finland
FORMANEK	Florian	florian.formanek@horiba.com	Horiba	Japan
COPIN	Dalyal	d.copin@irmatech.com	IRMA	France
COLPO	Pascal	Pascal.COLPO@ec.europa.eu	JRC Ispra	Europe
GILILAND	Douglas	Douglas.GILLILAND@ec.europa.eu	JRC Ispra	Europe
JONES	Arwyn	Arwyn.JONES@ec.europa.eu	JRC Ispra	Europe

LAST NAME	NAME	Contact	Organization	Country
WOJDA	Piotr	Piotr.WOJDA@ec.europa.eu,	JRC Ispra	Europe
KOOLS	Stefan	Stefan.Kools@kwrwater.nl	KWR Water	Netherlands
PROCHAZKA	Frédéric	info@lactips.com	LACTIPS	France
REUTENAUER	Philippe		Léa Nature	France
DRIS	Rachid	rachid.dris@u-pec.fr	LEESU	France
CHENEBLE	Jean-Charles		L'Oréal	France
FERMAS	Soraya		L'Oréal	France
SWEETLOVE	Cyril	csweetlove@rd.loreal.com	L'Oréal	France
MULLER	André	andre.muller@luxcontrol.com	Luxcontrol SA	Luxemburg
TOMASINI	Eric	eric.tomasini@luxcontrol.com	Luxcontrol SA	Luxemburg
SOULIAC	Laure	laure.souliac@developpementdurable.gouv.fr	Ministère de la transition écologique	France
BROU	Marjorie	marjorie.brou2@sante.gouv.fr	Ministère des solidarités et de la santé	France
BELARD	Laurent	l.belard@biopolynov.com	NATUREPLAST	France
QI	Yueling	Y.Qi@nioo.knaw.nl	Netherlands Institute of Ecology	Netherlands
-	-	-	NICOLE network	International
HURLEY	Rachel	rachel.hurley@niva.no	NIVA (Norwegian Institute of Water Research)	Norway
DE BOISSOUDY	Christophe	christophe.deboissoudy@novamont.com	NOVAMONT France	France
MORTAS	Nicolas	nicolas.mortas@organeo.com	ORGANEO	France
WEBER	Collin Joel	collin.weber@geo.uni-maburg.de	Philipps-University Marburg	Germany
Maria	WESTERBOS	maria@plasticsoupfoundation.org	Plastic soup foundation	Netherlands
VONK	Sophie	sophie@plasticsoupfoundation.org	Plastic soup foundation	Netherlands
-	-	www.plasticseurope.org	Plastics Europe	Europe
CASSART	Michel	Michel.CASSART@plasticseurope.org	Plastics Europe	Europe
Lindner	Sabine	Sabine.Lindner@Plasticseurope.de	Plastics Europe	Germany
GILBERT	Frédéric	frederic.gilbert@ofb.gouv.fr	PNR Landes de Gascogne	France
ROBB	Eilidh	-	Rethink Pastic	Europe
THEVENIN	Nicolas	nicolas.thevenin@rittmo.com	Rittmo	France
QUIK	Joris	joris.quik@rivm.nl	RIVM	Netherlands
CUENCA	Elodie	elodie.cuenca@danone.com	SAEME - SA eaux minérales Evian	France
LANCON	Maud	maud.lancon@danone.com	SAEME - SA eaux minérales Evian	France
CESAR	Guy	cesar.guy@neuf.fr	SERPBIO	France
BASTIN	Bénédicte	benedicte.bastin@spw.wallonie.be	Service publique de Wallonie (SPW-DGARNE)	Belgium
GENDEBIEN	Anne	anne.gendebien@spw.wallonie.be	Service publique de Wallonie (SPW-DGARNE)	Belgium
MENJOU	Laure	laure.menjou@siaap.fr	SIAAP	France
DIDY	Christian	christian.didy@spge.be	Société Publique de Gestion d'Eau (SPGE)	Belgium
MARCHAL	Romain	romain.marchal@spge.be	Société Publique de Gestion d'Eau (SPGE)	Belgium
ALBERTINI	Jéromine	jeromine.albertini@suez.com	SUEZ	France
DELAHAYE	Mathieu	mathieu.delahaye@suez.com	SUEZ	France
SAUR	Thibaut	thibaut.saur@suez.com	SUEZ	France
BRUNET	Hubert	hubert.brunet@sede.fr	SYPREA	France
COLPAERT	Romain	romain.colpaert@gmail.com	UMR CNRS de Besançon	France
COLPAERT	Romain	romain.colpaert@gmail.com	UMR CNRS de Besançon	France
BRUZAUD	Stéphane	stephane.bruzaud@univ-ubs.fr	Université Bretagne Sud	France

LAST NAME	NAME	Contact	Organization	Country
KEDZIERSKI	Mikaël	mikael.kedzierski@univ-ubs.fr	Université Bretagne Sud	France
GIMBERT	Frédéric	frederic.gimbert@univ-fcomte.fr	Université de Franche-Comté	France
BLOT	Denis	denis.blot@u-picardie.fr	Université de Picardie	France
PIETRAMELLARA	Giacomo	giacomo.pietramellara@unifi.it	University of Florence	Italy
RE	Viviana	viviana.re@unipi.it	University of Pisa	Italy
FILIPOVIC	Vilim	vfilipovic@agr.hr	University of Zagreb	Croatia
INGRAND	Valérie	valerie.ingrand@veolia.com	Véolia	France
LEROY	Gaëla	gaela.leroy@veolia.com	Véolia	France
PONT	Nicolas	nicolas.pont@veolia.com	Véolia	France
TIREZ	Kristof	kristof.tirez@vito.be	VITO	Belgium
VANERMEN	Guido	guido.vanermen@vito.be	VITO	Belgium
DONJON	Caroline	caroline.donjon@wessling.fr	Wessling	France
JEAMPIERRE	Frédéric	frederic.jeampierre@wessling.fr	Wessling	France
REIBER	Jens	jens.reiber@wessling.de	Wessling	France
DE FRANCE	Jennifer	defrancej@who.int	World Health Organisation	Switzerland

Appendix 4 : Description of spiked samples

Protocol for preparation of interlaboratory test - September 2020

ISSeP

1. Soil A: Synthetic silica from chemistry provider

Sand Fontainebleau, TECHNICAL

Formula: SiO_2
MW: 60.08 g/mol
Boiling Pt: 2230 °C (1013 hPa)
CAS Number: 7631-86-9
EINECS: 238-878-4

Soil A – MP 1: synthetic silica with Microspheres PE

Soil A – MP mix: synthetic silica with Mix Microspheres PE- PMMA-PS

2. Soil B: Real sandy soil available in laboratory ISSeP

Parameter	Unity	Sandy Real Soil – Soil B
pH	-	9,65
Dry matter	%	85,5
Clay	%	< 0,1
Organic matter	%	4,6
Metals/ metalloids		
Cr(VI)	mg/kg d.w.	0,35
As	mg/kg d.w.	11,7
Cd	mg/kg d.w.	0,61
Cr	mg/kg d.w.	174
Cu	mg/kg d.w.	34
Hg	mg/kg d.w.	0,21
Ni	mg/kg d.w.	45
Pb	mg/kg d.w.	73
Zn	mg/kg d.w.	591
Polycyclic Aromatic Hydrocarbons		

Naphtalene	mg/kg d.w.	0,11
Acenaphtylene	mg/kg d.w.	< 0,01
Acenaphtene	mg/kg d.w.	0,02
Fluorene	mg/kg d.w.	0,03
Phenanthrene	mg/kg d.w.	0,90
Anthracene	mg/kg d.w.	0,09
Fluoranthene	mg/kg d.w.	1,35
Pyrene	mg/kg d.w.	1,21
Benzoanthracene	mg/kg d.w.	0,78
Chrysene	mg/kg d.w.	1,10
Benzo(b)fluoranthene	mg/kg d.w.	1,09
Benzo(k)fluoranthene	mg/kg d.w.	0,42
Benzo(a)pyrene	mg/kg d.w.	0,46
Dibenzoanthracene	mg/kg d.w.	0,10
Benzo(g,h,i)perylene	mg/kg d.w.	0,57
Indeno(1,2,3-c,d)pyrene	mg/kg d.w.	0,40
Sum 16 PAH	*mg/kg d.w.*	*8,6*
BTEX		
benzene	mg/kg d.w.	< 0,075
toluene	mg/kg d.w.	< 0,25
ethylbenzene	mg/kg d.w.	< 0,15
mp-xylene	mg/kg d.w.	< 0,4
o-xylene	mg/kg d.w.	< 0,13

Soil B – MP 1: real sandy soil 250 µm with Microspheres PE

Soil B – MP mix: real sandy soil 250 µm with Mix Microspheres PE- PMMA-PS

Soil B – MP mix: real sandy soil (25% 250 µm + 75% 2 mm) with Mix Microspheres PE- PMMA-PS

Analytic results	Real sandy soil_EIL_2017-4.pdf
Particles size	Real sandy soil_particles size.pc

3. Microplastics

Microspheres ordered the 13/07/2020 from Cospheric.

PMMA Microspheres 1.2g/cc 90-106um - 10g	PMPMS-1.2 90-106um - 10g
Polystyrene Microspheres 1.07g/cc 85-105um - 500mg	PSMS-1.07 85-105um - 500mg
Clear Polyethylene Microspheres 0.96g/cc 90-106um - 10g	CPMS-0.96 90-106um - 10g

Microsphere of polypropylene aren't available in different providers:

- Cospherics LCC
- Borealisgroup
- Polysciences
- Spherotech
- Nexeo plastics
-

4. Protocol

For the preparation the distributor Resch is used:

- First, division matrix in 10 bottles.
- Second, division MP in same bottles.
- At the end, mix during one hour in flipper mixer.

Figure 1 : distributor Resch

5. Samples preparation

Batch number	Batch name	Matrix	Quantity of matrix in each bottle	MP	Quantity of MP in each bottle
1	QMP005SL	Sand	20 g	PE	40 mg
2	QMP006SL	Sand	20 g	PE PMMA PS	10 mg 15 mg 1.5 mg
3	QMP007SL	Real sandy soil 250 µm	20 g	PE	25 mg
4	QMP008SL	Real sandy soil 250 µm	20 g	PE PMMA PS	10 mg 15 mg 1.5 mg
5	QMP009SL	Real sandy soil (25% 250 µm + 75% 2 mm)	20 g	PE PMMA PS	10 mg 15 mg 1.5 mg

Appendix 5 : Interlaboratory study – flyer (round 2)

QUASIMEME/NORMAN Interlaboratory Study on the Analysis of Microplastics in Environmental Matrices

INVITATION TO

ROUND 2 - Development Exercise DE 17

open to all laboratories with an interest in microplastics analysis worldwide

Introduction

Microplastic' is a catch-all phrase for plastic particles spanning six orders of magnitude in particle size (0.1–5000 μm) and a gigantic variety of chemical compositions: (co)polymers, chemical additives, residual monomers, fillers, catalysts, non-intentionally added substances (NIAS) etc. The diversity of this analyte class has resulted in a range of different analytical methodologies being applied thus far. One of the challenges analytical scientists face with microplastics analysis is how to check and demonstrate analytical proficiency. The interlaboratory study (ILS) initiative for microplastics analysis described in this flyer has been designed to answer the need of laboratories working on analytical quality control of their microplastics analyses. Established in 2018, this initiative is dedicated to the development and collaborative improvement of microplastic analytical proficiencies, involving a large number of laboratories worldwide working towards common analytical goals. As a first step, a workshop on microplastics was organized in Amsterdam, the Netherlands, in November 2018. During this workshop it was generally agreed that an open ILS on microplastics was needed, preferably designed as a step-wise approach.

Step-wise ILS Study Design

The ILS consists of a minimum of three rounds, unless corrective actions or a repetition of one step is deemed necessary. Laboratories use their in-house methods, as currently no standard or harmonized methods exist. Because this ILS focuses on a new and difficult analysis, it is also called a 'Development Exercise' (DE). It is anticipated that after the entire study analytical methodologies will be harmonized and the microplastics could be included in the routine proficiency testing scheme of QUASIMEME (www.quasimeme.org). The ILS initiative is strengthened by feedback from the community of participants via workshops and bilateral communications, taking lessons learned from the opening round to future rounds.

- First Workshop (completed in 2018) Ca. 110 participants discussed analysis on microplastics in environmental matrices in Amsterdam, the Netherlands in November 2018.
- ILS Round 1 (completed in 2019) The first round focused on the identification of microplastics with pre-production pellets and identification and quantification of microplastics in tablets of eleven

different tests. The results and outcome of the first workshop and ILS round 1 have been described in a report, which has been sent to all participants of the first round.

- ILS Round 2 (current round) The second round will include besides tablets (as in first round) also the extraction of microplastics from more complex samples (e.g. sediments and/or fish).
- Second Workshop in 2020 (details will follow)
- ILS Round 3 (details will follow)

How to participate in the upcoming second round of ILS
All analytical methods are welcome. We encourage laboratories using Py-GC-MS to apply as well.
NB Laboratories who did not participate in the first round are welcome to join this round.

Participants should register on or before **1st September 2020**. To register, please return the completed 2020 Round 2 application form DE-17 Microplastics by email to quasimeme@wur.nl.
Suggestions with regard to the design of the study and the type of test materials are also welcome and may be added to your email. Upon receipt of your application form you will receive a confirmation of your participation and an invoice.

Participants in Round 2 may request a copy of the Round 1 report by emailing quasimeme@wur.nl.

Participation Fee The fee for participation in this study is 750 euro per round. If participants wish to register for rounds 2 and 3 together, a discount of 100 euro will be offered on the workshop registration fee, which will take place after round 2. Note that for each round, the test samples cannot be dispatched before receipt of participant fee.

Tentative 2020-2021 timeline

1 Spetember 2020	*Deadline registration*
6 October 2020	*Dispatch of test materials*
10 January 2021	*Deadline for returning results*
12 April 2021	*Draft Report sent to participants*
30 April 2021	*Final Report*
20-21 May 2021	*Second workshop and planning round 3*

ILS Initiators

This study is being coordinated by Dr. Heather Leslie, Dr. Louise van Mourik and Prof. Jacob de Boer of the Dept. of Environment and Health at the Vrije Universiteit (VU), Prof. Bert van Bavel of the Norwegian Research Institute for Water Research (NIVA) and Prof. Wim Cofino, Steven Crum and Esther van de Brug of WEPAL-QUASIMEME Laboratory Performance Studies (Quality Assurance of Information in Marine Environmental Monitoring in Europe). The ILS initiative is supported and promoted by the NORMAN network and the NORMAN working group on nano-and micro scale particulate contaminants. The four institutions have joined forces to set up a program to address the quality of microplastic analyses. QUASIMEME operates Proficiency Testing Studies for institutes making chemical measurements in the aquatic environment worldwide. As part of the improvement program, QUASIMEME co-operates with centers of excellence to provide workshops for discussion, and "hands on" experience to complement the development programs in Laboratory Performance Studies.

Questions or feedback? Please contact us by email at quasimeme@wur.nl

Website link for ILS: https://science.vu.nl/en/research/environment-and-health/projects/microplastics-ws-and-ils/index.aspx

Abstract

The one-year European project MISSOURI focuses on microplastics (MP) in soil and groundwater and aims at conducting a state-of-the-art review along a “sources-transfer-exposure” continuum and at participating in a European-scale interlaboratory study (ILS) in order to provide recommendations on separation and analytical methods in an idea of harmonization.

This work aims at proposing a harmonized definition for microplastics, a set of laboratory methods for the separation and analysis of microplastics in soil and at identifying priorities for future projects. It also aims at giving first recommendations for decision-making and management of soil quality regarding the potential risks associated with microplastics in soil and groundwater.

The mid-report focuses on work completed during the first 6 months of the project:
i/ soil spiking with MP microspheres in order to provide 5 soil samples for the ILS that started in October 2020, and
ii/ survey sent to stakeholders in December 2020 in order to collect their current difficulties and concerns encountered with MP management and treatment as well as their expectations on data collection and future studies.