Net4mPlastic project

Activity 3.1

D 3.1.1 b

State of art on

Sampling/treatment methodology

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CONTRIBUTING PARTNERS	UNITS

INDEX

Herein there is an extract from the literature to build a shared protocol for the collection and treatment of samples of microplastic (mP).

mP definitions

There are several definitions for mP.

For Arthur et al., (2009) mP are all plastic particles <5 mm in diameter. Hartmann et al., (2019) gives a more detailed size description, reported in the following table.

Name	**Size**
Macroplastics	> 1 cm
Mesoplastics	1 to < 10 mm
Microplastics	1 to < 1000 μm
nanoplastics	1 to <1000 nm

Frias & Nash, (2019) propose a definition for mP:

“Microplastics are any synthetic solid particle or polymeric matrix, with regular or irregular shape and with size ranging from 1 μm to 5 mm, of either primary or secondary manufacturing origin, which are insoluble in water”.

What is consider “plastic” is defined in the Figure 1 by Hartmann et al., (2019).

Criterion	Recommendation	Examples
I: Chemical composition		
Ia: Polymers	All synthetic polymers:	
✓ Include	▪ Thermoplastics	All commodity plastics
	▪ Thermosets	Polyurethanes, melamine
	▪ Elastomers	Synthetic rubber
	▪ Inorganic/hybrid	Silicone
✓ Include	Heavily modified natural polymers (semi-synthetic)	Vulcanized natural rubber, regenerated cellulose
× Exclude	Slightly modified natural polymers	Dyed natural fibers
Ib: Additives		
✓ Include	All polymers included in Ia disregarding their additive content	Plasticized PVC with >50 % additives
Ic: Copolymers		
✓ Include	All copolymers	ABS, EVA, SBR
Id: Composites		
✓ Include	All composites containing synthetic polymer as essential ingredient	Reinforced polyester and epoxy
✓ Include	All surface coatings containing polymers as essential ingredient	Paints containing polyester, PUR, alkyd, acrylic, epoxy resin
✓ Include	Tire wear (and road) particles	-
? Open question	Is it necessary to define a minimum polymer content?	
II: Solid state		
✓ Include	All polymers with a T_m or T_g >20 °C	See examples in Ia
× Exclude	Polymer gels	PVA, PEG
? Open question	Should wax-like polymers (T_g <20 °C) be included?	
III: Solubility	All polymers with a solubility <1 mg L^{-1} at 20 °C	See examples in Ia
✓ Include		
IV: Size	▪ Nanoplastics: 1 to <1000 nm ▪ Microplastics: 1 to <1000 µm ▪ Mesoplastics: 1 to <10 mm ▪ Macroplastics: 1 cm and larger The largest dimension of the object determines the category. Comprehensive reporting of multiple dimensions is preferred (e.g., for fibers).	
V: Shape and structure	Spheres: Every surface point has the same distance from the center Spheroid: Imperfect but approximate sphere Cylindrical pellet: Rod-shaped, cylindrical object Fragment: Particle with irregular shape Film: Planar, considerably smaller in one than in the other dimensions Fiber: Significantly longer in one than wide in two dimensions Additional information on the structure (e.g., porosity) can be included.	
VI: Color	Not crucial but useful in some biological contexts. Use a standardized color palette.	
VII: Origin (optional)	Primary: Intentionally produced in a certain size Secondary: Formed by fragmentation in the environment or during use Origin should only be used if the primary origin can be established.	

Figure 1: Overview for definition and classification of plastic debris (https://doi.org/10.1021/acs.est.8b05297)

1 Sampling

It is possible to summarize the methods in 4 items:

1. Identify location of sample collection (geographic)
2. Identify if water or sediment (beach or seabed)
3. Samples treatment, depending on the first 2 points
4. Samples analysis

1.1 Identify location

From the various studies (Vianello et al., 2013,Barrows et al., 2018, Luo et al., 2019, Ding et al., 2019, Yan et al., 2019, Alam et al., 2019, Unice et al., 2019, Barletta et al., 2019, Kataoka et al., 2019), it comes evident the plastics transportation from river to sea.
Focusing on the Adriatic sea, the paper of Atwood et al., (2019) reports the situation of coastal accumulation of microplastic particles (1–5 mm) emitted by the Po River over 1.5 years, beside models based both on hydrodynamic and remote sensing (satellite platforms). The worst situation was find in Caleri beach, as reported in the table2 of Atwood et al., (2019).

Table 2: (Atwood et al., 2019) Sediment microplastic overview for all 9 beaches sampled, listed north to south. Percent contribution from each plastic type identified is listed: PE polyethylene, PP polypropylene, PS polystyrene (1 also includes ABS acrylonitrile butadiene styrene and SAN styrene acrylonitrile), PA polyamide, EV accounts for EVOH ethylene vinyl alcohol and EVA ethylene vinyl acetate, PEST polyester, PET polyethylene terephthalate, PVC polyvinyl chloride, PUR polyurethane, PVAL polyvinyl alcohol, SBR styrene butadiene rubber, C/U accounts for either composite particles or unknown plastic types. Total microplastic particles found as well as particles/DW kg is indicated for each beach sampled.

Beach	% contribution												Tot. part.	Part./DW kg
	PE	PP	PS[1]	PA	EV	PEST	PET	PVC	PUR	PVAL	SBR	C/U		
Caleri	45.0	8.6	28.0	< 1	18.0	< 1	< 1	< 1	< 1	0	0	< 1	3080	78.8
Levante	62.2	14.6	16.4	< 1	5.7	< 1	< 1	< 1	< 1	< 1	< 1	< 1	2032	59.4
Boccasette	42.9	13.2	42.9	0	< 1	0	0	0	0	0	0	< 1	182	3.9
Pila North 1	27.0	14.8	54.8	0	< 1	0	< 1	0	0	0	0	< 1	115	2.2
Pila North 2	60.2	9.7	20.4	1.9	0	< 1	4.9	0	< 1	0	0	< 1	103	3.6
Pila South	45.7	18.9	34.1	0	1.4	0	0	0	0	0	0	0	440	8.4
Allagamento	10.0	5.0	85.0	0	0	0	0	0	0	0	0	0	20	0.5
Barricata	19.2	13.8	66.3	< 1	< 1	0	0	0	0	0	0	0	652	14.3
Goro	52.0	19.0	27.8	0	< 1	0	0	0	0	0	0	< 1	248	5.2

The model show how the wind direction can influence the mP dispersion in the sea (Figure 2 Atwood et al., 2019).
So, it is important to define, beside the location, the season and the weather of sampling time too.

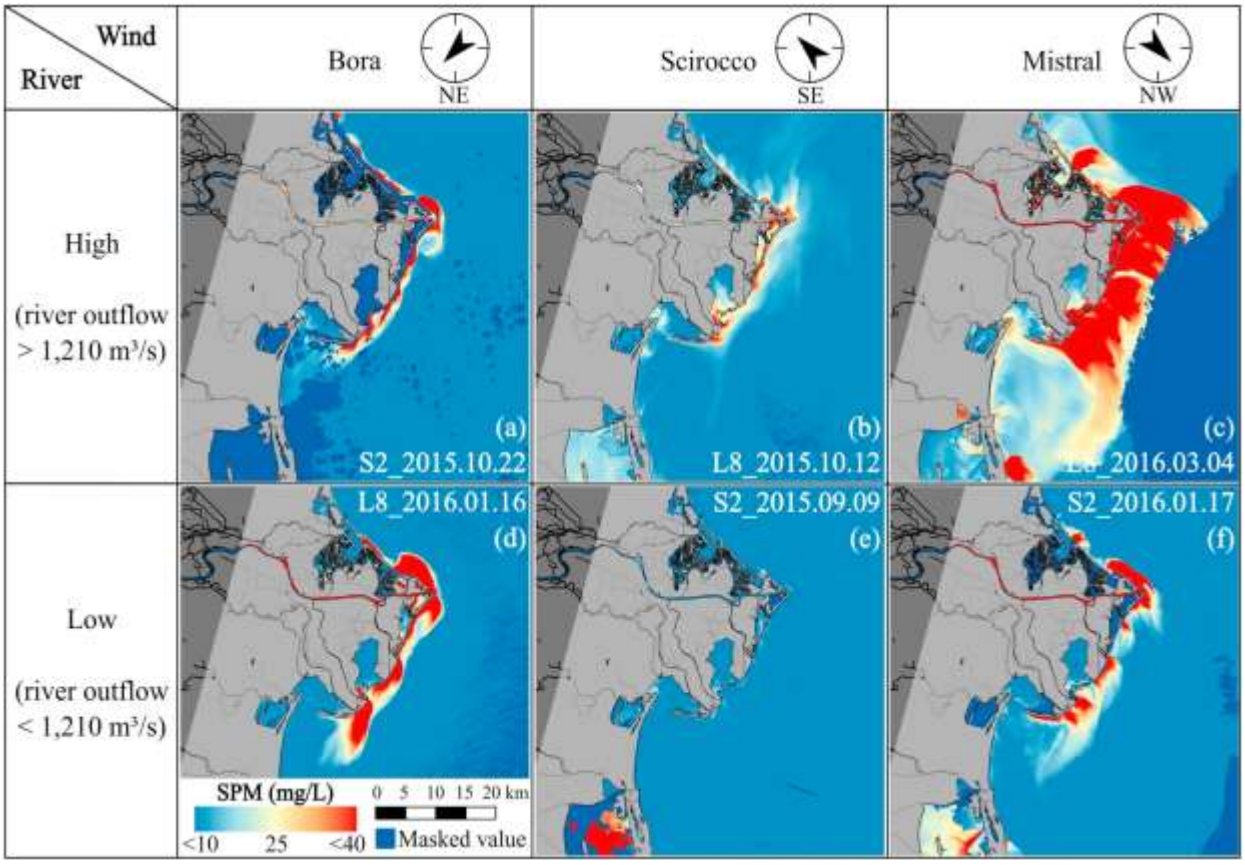

Fig. 2: Combined effect of different wind regimes (Bora, Scirocco or Mistral) with differing river discharge conditions on river plume transportation along the Western Adriatic. River discharge is termed "high" (panels a, b, c) or "low" (panels d, e, f) depending on daily discharge relative to the median (1210m3/s) over the entire simulation period. Wind events were classified based on wind direction (indicated by wind compass in each column, pointing in the direction that wind is blowing) and strength (winds in excess of 5 m/s). Suspended Particulate Matter (SPM) values, ranging from low in blue to high in red, depict river plume shape. Masked pixels are depicted in dark blue, land in light gray (outside of area of interest in dark gray). (Atwood et al., 2019)

1.2 Identify if water or sediment

From Literature (Wang & Wang, 2018) a generalized classification in sampling strategy can be reduce to 3 types:

Selective – in beach; plastics are large enough for identification with the naked eye and thus can be extracted directly from environment;

Bulk – in sediments or in water; collect the entire sample without reducing its volume during the sampling process; only a relatively small amount of a sample can be collected, which may negatively affect the representativeness of the sample (Hidalgo-Ruz et al., 2012).

Volume-reduced – most popular for water samples; refers to reducing the entire volume of a bulk sample by fast filtration during sampling and preserving only a small fraction of the sample for subsequent analysis.
The different equipment can be summarized in the table 3 (Wang & Wang, 2018, Silva et al., 2018, Prata et al., 2019).

Table 3: Equipment usable for different type of site

site	type	equipment
water	surface	Trawl with a rectangular opening and a net connected with a collecting bag/neuston net/plankton net / catamaran
		Bottles / pumps
	mid level	Bongo nets / pumps
sediments	bottom	Box corer
	surface	Iron spoons / non-plastic sampling spades
	seabed	Core or bottom trawl

The net mesh sizes vary widely, ranging from 53 μm to 3 mm, thus influencing the volume and nature of the microplastics obtained from samples (Silva et al., 2018). Manta nets allow the sampling of large volumes of water and are widely used allowing some standardization of methods.
Plankton nets also have smaller mesh sizes (~100 μm), allowing sampling under a minute and recovering concentrations 30 times higher than manta nets (Dris et al., 2015).

1.3 Samples treatment

Following the NOAA recommendation (Masura et al., 2015) different schemes are useful depending on sample type.

mP in Water samples
Samples collected by a surface net:

- Filtration of solids in a 0.335 mm surface sampling net (e.g. a manta net for surface water tows) through 5.6-mm and/or 0.3-mm sieves to isolate the solid material of the appropriate size
- Drying the solid → determination of the solid mass in the sample
- Chemical treatment for eliminate the organic matter without alter the plastic debris
- Physical separation by density (flotation) – density separator
- Drying the separate fractions → weight for mP concentration determination
- Sample analysis for mP chemical type determination

About the chemical and physical separation, there are several method used, as in Figure 3 (Renner et al., 2018).

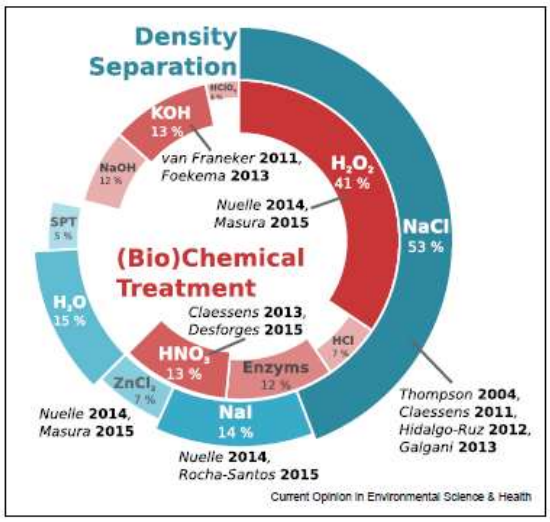

Fig. 3: Overview of the most common density separation (outer ring) and (bio)chemical treatment (inner ring) protocols including their reference authors. The % values are related to 67, or 53 articles, respectively, in which density separation or a (bio)chemical treatment are described. (Renner et al., 2018)

Hydrogen peroxide treatment (WPO) is the recommendable approach to purify microplastics (Masura et al., 2015, Renner et al., 2018), although for Hurley et al., (2018) the optimum protocol for efficient organic matter removal is the Fenton's reagent (a solution of hydrogen peroxide (H2O2) with ferrous iron (typically iron(II) sulfate, FeSO4) as a catalyst).

In Figure 4 the flow diagram of the procedure for mP in water sampling and treating is reported. For detail on the single operation unit see Masura et al., (2015).

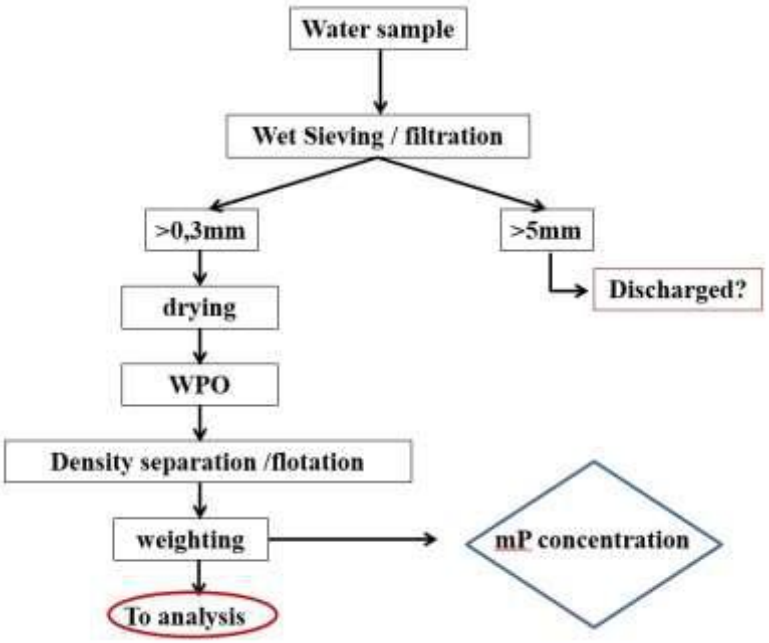

Fig. 4: Flow diagram of water sample treatment procedure.

mP in Beach samples

Samples collected by shovel or spade.

- Sieving dry beach samples to 5 mm to remove large macroscopic debris

The procedure is similar to that for water samples, so similar is the flow diagram of Figure 3.

mP in Bed samples

Samples collected by corer or grab sampler (e.g. Ponar sampler).

- Drying the sediment sample
- Disaggregation of the dried sediments
- Sieving using stacked 5-mm and 0.3-mm sieves
- It follows the operation units of water samples (chemical/physical purification) as in Figure 3.

Coppock et al., (2017) present a portable equipment to separate microplastics from sediments of differing types, using the principle of density floatation (Figure 5 Coppock et al., 2017).

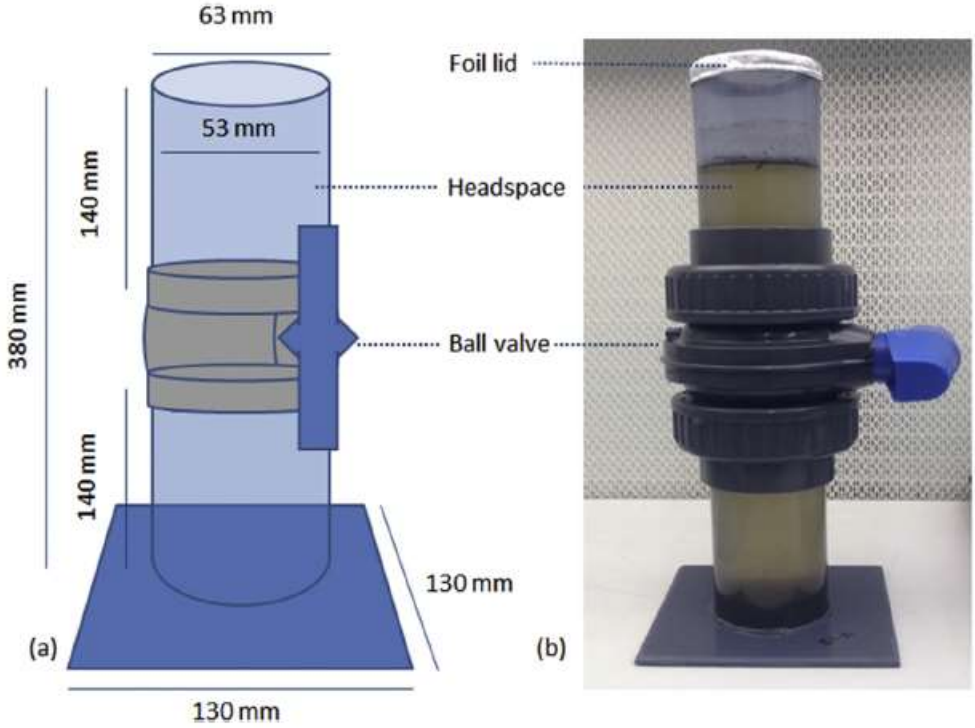

Fig. 5: Schematic (a) and photograph (b) of Sediment-Microplastic Isolation (SMI) unit. Photograph depicts SMI unit within contamination control cabinet with ball valve in closed position, denser sediment settled at the bottom of ZnCl2 solution (1.5 g cm3) and less dense particles floating on top. (Coppock et al., 2017)

Generalizing the process from sampling to analysis can be described as in Figure 6 (He et al., 2018).

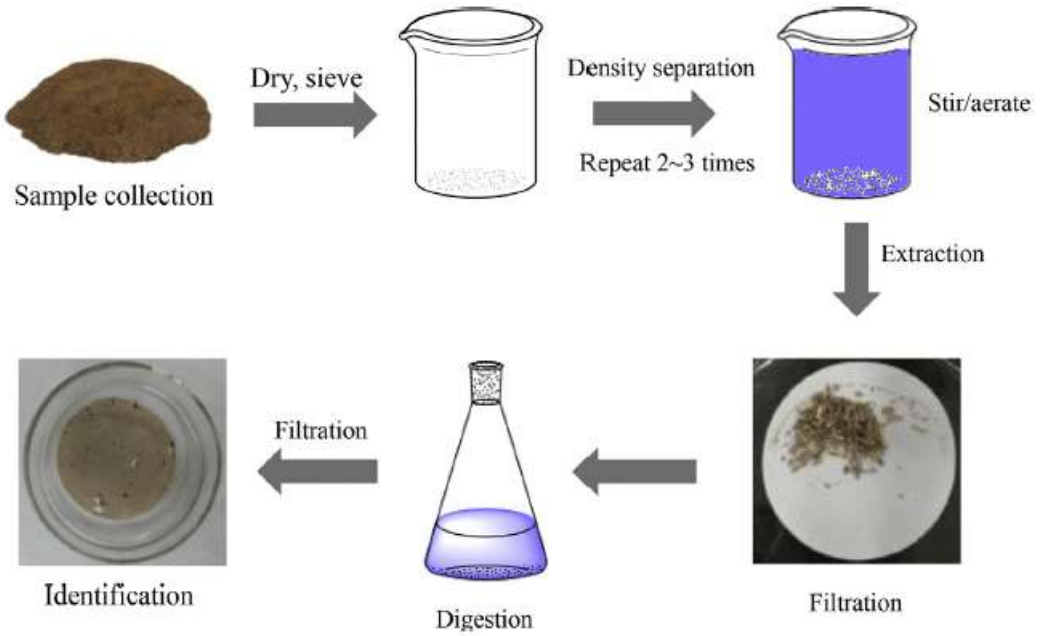

Fig. 6: Schematic diagram in analytical procedures for microplastics in soil samples (He et al., 2018)

Regarding the floatation unit, it must be considered that the specific densities for most plastics range from 0.8 to 1.70 g cm-3, while average densities for sand or other deposits are typically 2.65 g cm-3 (Wang & Wang, 2018).

Several salts can be used for preparing solutions with the desired density, the most cited are NaCl, NaI, $ZnCl_2$, $Li_2O_{13}W_4$ (lithium metatungstate) (among many: Wang & Wang, 2018, Prata et al., 2019, Coppock et al., 2017, Li et al., 2018). NaCl saturate solution (d= 1.202 g cm-3) is inexpensive and appropriate for low-density plastics (PE, PP, PS), not for other types (e.g. PET or PVC) (Wang & Wang, 2018).

It is possible to prepare different floating solutions, as in table 4 , (Li et al., 2018) with NaI or with $ZnCl_2$, being the latter the less expensive (Coppock et al., 2017, Li et al., 2018):

Table 4: Formula of density gradient solutions (20mL) in each density from0.8 to 1.8 g/cm3, using the bases of ethanol (0.8 g/cm3), ultrapure water (1.0 g/cm3), NaI or ZnCl2 (1.8 g/cm3). '–', none. (Li et al., 2018)

Volume (mL/mL)	Density gradient solutions (g/cm^3)										
	0.8	0.9	1.0	1.1	1.2	1.3	1.4	1.5	1.6	1.7	1.8
Ethanol/water	20/0	8/12	0/20	–	–	–		–	–	–	
NaI/water	–	–	–	3/17	5/15	7/13	9/11	11/9	15/5	17/3	20/0
$ZnCl_2$/water	–	–	–	2/18	5/15	7/13	10/10	12/8	15/5	18/2	20/0

1.4 Samples analysis

The outputs of the mP analysis cover different aspects and can be summarize as:

- Quantification of mP, given by mass/area or by mass/volume, referring to sea area or sample volume (the quantification by number is not satisfying, although several studies report it)
- Qualification
    - by dimensions
    - by shape
    - by colour
    - by plastic type
    - by origin, primary or secondary

- ***by dimensions /shape / colour***

Length measurements were assisted by optical microscopy; each particle was in length categories, for example: <100 µm, 100–500 µm, 500–1000 µm, and >1000 µm (1000–5000 µm) (Jiang, 2018).

Polymer size, shape, density, colour and chemical composition are properties that can influence sorption dynamics (Rodrigues et al., 2019, Rocha-Santos & Duarte, 2015, Fisner et al., 2017, de Sá et al., 2018).

Related to shape, microplastics can assume spheres, fibers, films, fragments, pellets and irregular forms, and have densities that range from 0.016 to 2.2 g/cm^3 (de Sá et al., 2018).
The most commonly reported types of microplastics recorded in the literature worldwide are pellets, fragments and fibres, with films, ropes, filaments, sponges, foams, rubber and microbeads in decreasing order. However, it should be noted that different countries use different terminology to classify the same object or plastic type (Frias & Nash, 2019).

Aspects such as colour are not considered to be crucial to defining microplastics, because colour differentiation is subjective, and it cannot contribute to the visual identification of microplastics by itself (Lusher et al., 2017). However, recording microplastic colour is considered important, for studies concerning aquatic organisms, as some species are thought to potentially ingest microplastics based on a colour preference behaviour (Frias & Nash, 2019, Wright et al., 2013).

- ***by plastic type***

The most common approach for the detection of microplastics consists in the visual identification of apparent/possible plastic particles followed by confirmation through chemical composition analyses, usually combining optical and spectroscopic techniques (Hanvey et al., 2017).
The different techniques more cited are:
Optical microscopy, SEM (EDS), FTIR and µFTIR, Raman and µRaman, gas chromatography coupled with mass spectrometry (GC-MS) with pyrolysis (PY-GC-MS) or thermal extraction (TED-GC-MS), Thermal analysis.

According to Schwaferts et al (2019) the right technique depends on the fragments dimensions, as in Figure 7.

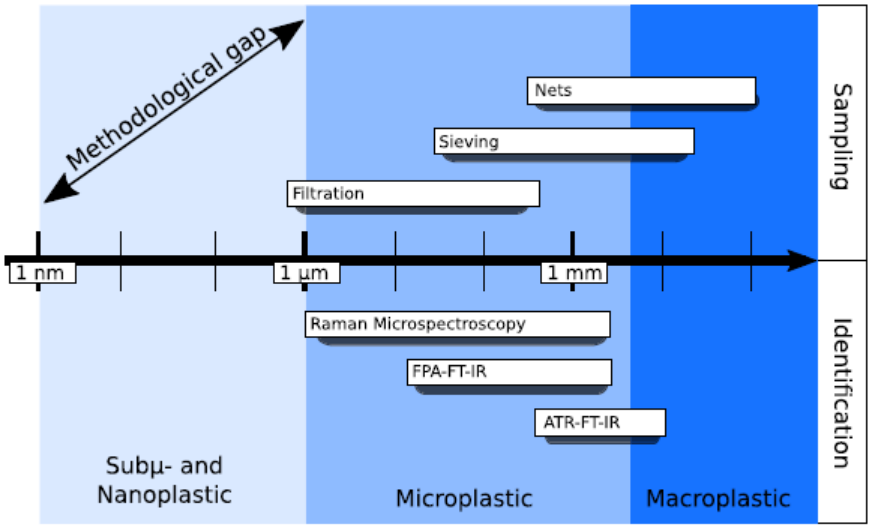

Fig. 7: The analysis of MP is established for particles down to 1 µm. Below, there is a methodological gap (Schwaferts et al., 2019)

Corradini et al., (2019) propose the use a portable spectroradiometer for soil sample analysis avoiding the extraction steps (Figure 8); with this apparatus it is possible to identify and quantify LDPE, PET and PVC using a vis-NIR analysis technique.

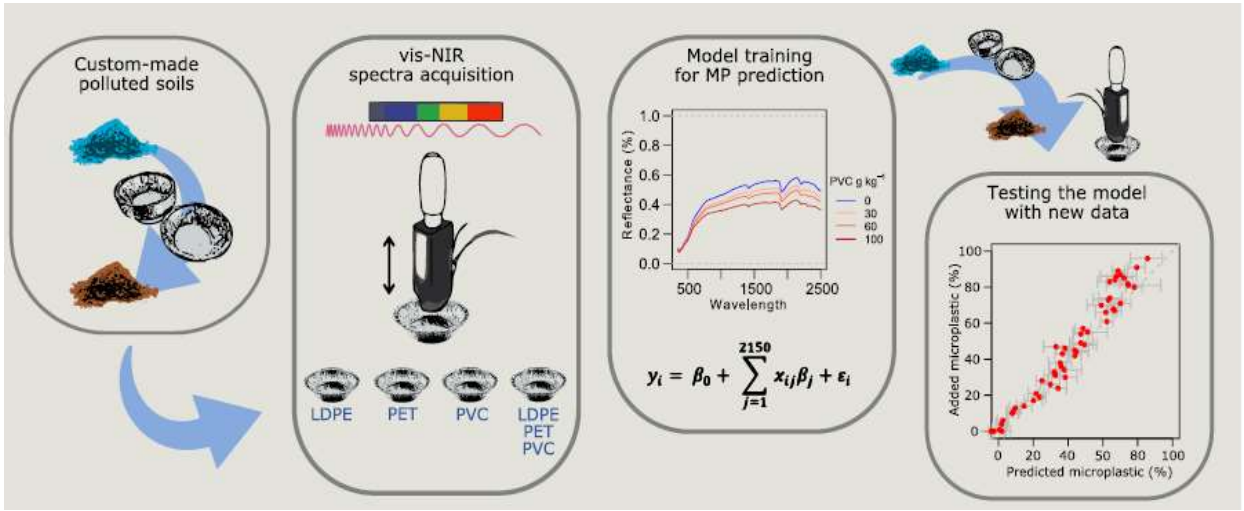

Fig. 8: Schematic representation of the vis-NIR spectroscopy method for mP quantify and qualify in soils (Corradini et al., 2019)

Harrison et al., (2012) investigated the applicability of micro-FT-IR and reflectance micro-FT-IR spectroscopy for the determination of microplastics in marine sediments, and proved the ability of such techniques for molecular mapping analysis to detect microplastics in sediments successfully, based on their spectral characteristics, without the need for visual inspection of fragments for characterization (Jiang, 2018).

In Figure 9 is reported a general scheme that group sampling approaches, including analytical techniques already used and established for other analytes (Oliveira & Almeida, 2019).

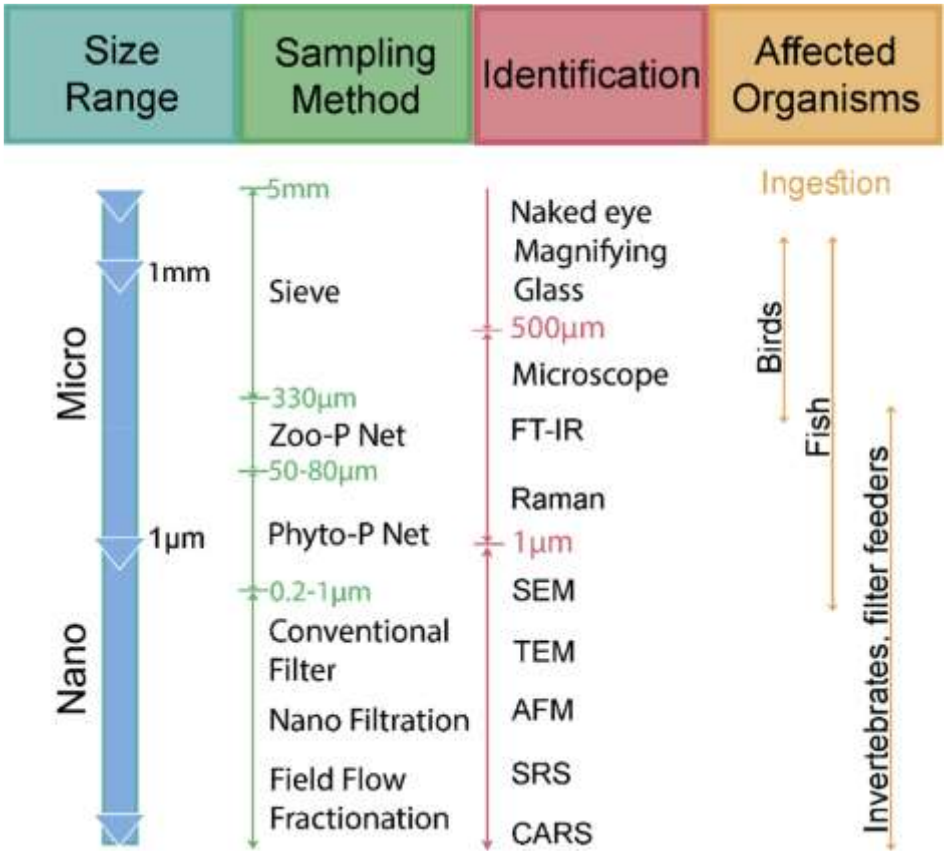

Fig. 9: Diagram representing methodologies to sample and identify micro(nanoplastics) and examples of affected organisms. AFM - Atomic force microscopy; Cars - Coherent anti-Stokes Raman scattering; FT-IR - Fourier-transform infrared spectroscopy; Phyto-Pe Phytoplankton; SEM - Scanning electron microscopy; SRS - Stimulated Raman scattering microscopy; TEM - Transmission electron microscopy; Zoo-P e Zooplankton. (Oliveira & Almeida, 2019)

Regarding the Thermal analysis, only few papers have been found that used it for recognition of mP. Those who used the DSC, was only as confirmation (through the melting T) of the FTIR analysis (Kühn et al., 2018, Tunçer et al., 2018), or for crystallinity determination for plastic degradation studies (Arhant et al., 2019). Only one paper was found (David et al., 2019) based on TGA. In this case, a model is proposed for qualification and quantification of microplastics in soils without sample pre-separations or pre-treatments. The soil universal model method (SUMM) proposed in this work is based on the determination of the difference between the modelling relationships between TML/LTML and measured values. (TML = thermal mass losses; LTML = larger thermal mass loss).

Finally, a detailed protocol, used for the collection of mP on sea surface during the project "DeFishGear" (project, str/00010, IPA-Adriatic, Cross Border Cooperation 2007–2013), is reported (Kovač Viršek et al., 2016).

2 “DeFishGear” protocol

2.1 Sampling of microplastics on the sea surface

1. Deploy the manta net from the side of the vessel using a spinnaker boom or »A-frame« using lines and karabiners.
2. Deploy the manta net out of the wake zone (approx. 3 - 4 m distance from the boat) in order to prevent collecting water affected by turbulence inside the wake zone.
3. Write down the initial GPS coordinates and initial time in the data sheet.
4. Start to move in one straight direction with a speed of approx. 2 - 3 knots for 30 min and begin the time measurement.
5. After 30 min stop the boat and write down final GPS coordinates, the length of the route (the most correct way is to calculate the length from the GPS coordinates) and the average boat speed into the data sheet provided and lift the manta net out of the water.
6. Rinse the manta net thoroughly from the outside of the net with seawater using a submersible pump or water from the boat water reservoir.
   Rinse in the direction from the manta mouth to the cod end in order to concentrate all particles adhered to the net into the cod end.
   Note: Never rinse the sample through the opening of the net in order to prevent contamination.
7. Safely remove the cod end and sieve the sample in the cod end through a 300 µm mesh size sieve or less.
8. Rinse the cod end thoroughly from the outside and pour the rest of the sample through the sieve. Repeat this step until there are no longer any particles inside the cod end.
9. Concentrate all material on the sieve in one part of the sieve.
10. With the use of a funnel, rinse the sieve into a glass jar or plastic bottle using 70 % ethanol.
11. Close the bottle, wipe it with paper towels and label the lid and outside of the jar with the sample name and date with waterproof marker (you should also put a second label written with a pencil on velum paper in a jar to avoid the possible loss of the sample name due to the erased label on the jar). Transfer labeled plastic bottle into the cool box.
    Note to general sampling conditions: The wind speed should not be more than 2 Beaufort, since the waves are too high and the net is not stable on the sea surface. It is important to maintain a steady linear course at a constant speed during the trawls. Half of the manta net opening should be submersed during sampling. Duration of sampling should be 30 min (in cases where there is a large amount of natural material, e.g. plankton bloom, the duration of sampling can be shorter). Avoid the use of plastic tools and containers. Avoid synthetic clothing (e.g. fleece), ropes and contact of manta net with vessel to prevent contamination of the sample. Be very careful not to damage the manta net or the boat hull while deploying and capturing the net.

2.2 Separation of microplastics from the sea surface samples

1. If the sample does not contain any items larger than 25 mm and appears to be clean, continue directly with step 3.
2. Pour sample through the sieve (≤300 µm mesh size) and remove all natural or artificial litter objects of a size >5 mm (macro and meso litter) from the sample, using visual identification and tweezers. Be careful to rinse each removed object carefully with distilled water in order to remove any microplastic litter adhered to it. Store all natural and artificial litter objects in separate containers. Dry all natural and artificial litter objects in a desiccator (or in the open air, but in a closed dish) and weigh them. Identify all litter objects >25 mm (macro litter) according to the Master List of Categories of Litter Items.
3. After removing all larger objects, concentrate all remaining pieces in one part of the sieve using squirt bottles or tap water. Pour the sample into a glass container using a minimum amount of 70 % ethanol with the help of a funnel.

   Note: In this step the use of 70 % ethanol is crucial to preserve the sample. Also in the step of visual inspection of the sample, ethanol helps to discolor the organisms and colorful plastics therefore become easier to find.
4. Take a small amount of the sample (subsample) and pour it into a glass Petri dish. Analyze the sample with the use of a stereomicroscope (20 - 80x zoom) and search for microplastic particles.
5. Each microplastic particle should be categorized into one of the categories listed in Table 1 and put into a Petri dish or other glass vials, marked with a category name. The Petri dish needs to be closed at all times.

   Note: When separating microplastics from your sample be conservative and select more rather than less particles for the analysis. The real chemical structure of particles will still be determined later on. Be sure to analyze larger objects from all sides as microplastics may be stuck and therefore hidden under larger items. It may also be helpful to move already analyzed objects to one side of the Petri dish.
6. Put the Petri dish under the microscope with measuring equipment (ocular ruler calibrated by the micrometer slide or image analysis software) and measure the size of each particle (measure the longest diagonal), except filaments, and note its color. Each subsample should be reviewed by another person. Be careful to rinse the glass container containing the sample so that all particles adhering to the glass walls are washed into the Petri dish.
7. Weigh the microplastic particles of each category separately by the use of analytical scale. Microplastic particles need to be dried prior to weighing. The closed Petri dish can be put in a desiccator or the samples can be left to dry in a closed dish till particles became dry (the weight of closed petri dish with particles is constant).
8. Identify micro litter. When analyzing a sample in search of microplastics, please consider that some particles will be easily visible (color, shape, size) while others may be trickier to find. For example, no cell structure, uneven, sharp, crooked edges, uniform thickness, distinctive colors (blue, green, yellow, etc.).

2.3 Chemical identification of microplastics

1. ATR-FTIR spectroscopy
    1. Prior to the analysis clean the detection system with alcohol and a lint free cloth.
    2. Record a background spectrum. Place the sample on the sample holder and collect the spectra. Identify the obtained ATR- FTIR spectra using an automated comparison of the obtained spectrum with spectra in a database.

2. Micro ATR-FTIR spectroscopy
    1. Prior to the analysis clean the detection system with alcohol and a lint free cloth.
    2. Place the sample on a glass filter. Note: Other filters can be used but their polymer nature can interfere with the characterization.
    3. Place the filter with the sample on the automatic scanning table and use the joystick to locate the sample.
    4. Record an optical image and mark an area (e.g. 20 by 20 µm) where the sample will be characterized.
    5. Record a background spectrum.
    6. Place the sample on the sample holder and collect the spectra at the predefined location.
    7. Identify the obtained micro ATR-FTIR spectra using an automated comparison of the obtained spectrum with spectra in a database.

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