A REVIEW ON ANALYTICAL TECHNIQUES FOR **DETECTION OF MICROPLASTICS IN SOIL AND** WATER

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Abstract- Microplastics (MPs) are synthetic solid particle or polymeric matrix, of either primary or secondary manufacturing origin, that is insoluble in water, with regular or irregular form and a size ranging from 1 µm to 5 mm. MPs has gained worldwide attention as an acquiring environmental concern. Some fundamental topics are reviewed, such as the occurrence, sources, and potential risk of MPs in soil and water. This review provides an extensive summary of analytical methods used for detection of MPs in various soil sample and water sample. MPs are detected by using Spectral analysis, Thermal analysis, Microscopic analysis, Chromatographic methods and hyphenated techniques. These techniques are used for quantitative identification of the various types of polymers in MPs.

Key Words- Microplastics, Soil pollution, Water pollution, Human risk, Analytical Methods.

I. INTRODUCTION

Introduction to Microplastics (MPs)

MPs are defined as "Any synthetic solid particle or polymeric matrix, of either primary or secondary manufacturing origin, that is insoluble in water, with regular or irregular form and a size ranging from 1 µm to 5 mm."[1]

Plastics are widely employed in a variety of contexts, including transportation, construction, manufacturing, healthcare, aerospace, packaging, and agriculture, due to their benefits in being inexpensive, versatile, durable, and strong. [2]

Table 1 Types and application of commonly used MPs [2]		
TYPES	APPLICATIONS	
PE	Packaging bags; plastic film; agricultural mulch; toys;	
	milk bottles; microbeads	
PP	Microwave containers; medicine bottles; dishware;	
	microbeads	
PS	Plastic-foam dinnerware; meat trays; building insulation;	
	plates	
DVC	Water pipes, wallboard, artificial leather, tablecloth,	
PVC	raincoat	
PET	Bottles for water, soft drinks, juices	
PAs	Wear-resistant parts, transmission structure parts, chemical	
	machinery parts.	
POM-POLYOXYMETHYLENE Machine parts, auto parts, gears		

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According to their sizes, plastic fragments in the environment may be divided into four size categories: megaplastics (>50 cm), macroplastics (5–50 cm), mesoplastics (0.5–5 cm), and MPs (<0.5 cm).

MPs with irregular shapes including nurdles, fragments microbeads, fibers, films or foams have been found in the environment. In addition, MPs can be further divided into nanoplastics (<0.3 mm), small MPs (0.3-1 mm) and large MPs (1-5 mm) on the basis of the properties of decomposition products.[3]

Sources of MPs [4]

Primary Sources of MPs:- These are MPs made from microbeads used in personal care goods like body washes, toothpaste, and industrial and medical products.

- Plastic Pellets (Raw Materials)
- \triangleright Personal Care Products
- ⊳ Paint
- AAA Washing Wastewater
- Sewage Treatment Plants
- Plastic Running Tracks in Schools
- Artificial Turf

- Rubber Road in Cities
- Vehicle Tire Wear

Secondary Sources of MPs:- These are minute plastic particles and fibres produced when synthetic clothing is washed.

- Municipal Debris- plastic bags, plastic bottles, disposable plastic tableware, plastic packaging,
- Fishing Wastes
- ➢ Farming Film

MPs in Environment[4]

- 1. Air
- 2. Water
- Oceans
- Ice cores
- Freshwater
- 3. Soil
- 4. Human body [5]

Sources(Distribution) and Transport of MPs



Figure 1 Sources, pathways and biophysiochemical impacts of MPs in the environment [6]

Sources (Distribution) and Transport of MPs in Soil

MPs are deposited in soil by water and wind from a variety of sources, including sewage sludge, plastic mulch, street runoff, trash, landfill leachate, and air dust. Dust, biosolids from wastewater treatment, and plastic mulch used for weed control are the three main sources of MPs in agricultural soils.[7]

When sowing crops, plastic mulch is frequently used to inhibit weed development, preserve moisture levels, and keep the soil at the appropriate temperatures. As a result, MPs build up in the soil. Mulch film, which is made of PVC and PE, has become a widely employed technology in the world's agricultural output as a result of its outstanding economic advantages, including increased harvest, higher fruit quality, and enhanced water consumption efficiencies. [8][9] It has been determined that irrigation and urea fertilisers coated with MPs are the principal sources of MPs in wet agricultural land-use systems (such as paddy rice fields). Particularly in places with limited access to water, the use of fertilisers and irrigation encourages crop growth and yield. For agricultural planting, humic acid polymer-coated fertiliser can promote crop growth. The two primary sources of MPs in the soil are thought to be plastic mulch and sewage sludge in rain-fed farmland systems (such as a sweet potato farm. MPs can easily penetrate the soil through illegal dumping and littering. Plastic waste is typically dispersed beside roadways, in the soil, or at illegal dump sites. Many researchers who conduct fieldwork discover plastic bottles and bags used for insecticides and fertilisers scattered across the field. Other significant sources of MPs in the soil include incorrect municipal waste disposal and tyre abrasive wear.[9] MPs downward migration is supported by the fact that they are found in the deep soil. Because soil has permeable nature, leaching can transport MPs in the µm range along with soil pores. External factors, like biological disruption and agricultural practices, cause larger MPs to migrate into the soil. The bioturbation of plant roots in the soil, which includes root movement, root expansion, root water extraction, and other processes, might also have an impact on the movement of MPs. The vertical and horizontal movement of MPs in the soil may be facilitated by soil abiotic factors. MPs are transferred and dispersed by earthworms and certain collembola species, either through adhesion or excretion. Additionally, the dry climate's effect on soil cracking allows MPs to penetrate deeper soil.[8]

Sources (Distribution) and Transport of MPs in Water

MPs can enter in freshwater bodies by variety of sources:

i.passage through WWTPs, either from MPs use in personal care products or release of fibres from textiles during the washing of clothes, to surface waters.

ii.application of biosolids from WWTPs to agricultural lands.

iii.storm water overflow events.

iv.incidental release (e. g., during tyre wear).

v.release from industrial products or processes.

vi.atmospheric deposition of fibres.[7][10][11]

After rain, MPs are typically seen at their highest concentration. [3] The bottles and caps of some bottled waters are made of plastic, which themselves may be a source of MPs in drinking-water.[12]

Plastic materials won't stay still after they've been released into the environment. They will move among environmental divisions (e.g., from land to freshwater). In the freshwater environment, MPs are either released or generated. Similar fundamental mechanisms, such as advection, dispersion, aggregation, sedimentation, deterioration or disintegration, resuspension, and burial, regulate the transport of MPs in water.[7]

Effects of MPs

1. Effects of MPs on Soil

MPs type	Hypothesized in soil structure	Potential effect and concern for crop plants	Examples of MPs
Fiber	Alterations in soil structure	Not known clearly	PE, PP, PS
Film	Higher evaporation of water from soil	Lower water availability for crops leading to slow growth	PP, PE, LDPE
Bead/microbead	Soil texture variations	Uptake by roots, less effects	PP, PVC, PE, PS
Fragment	Changes in properties of soil	Unclear effects	PP, PS, LDPE

When MPs particles enter soil ecosystems, they are quickly incorporated into the soil matrix by soil ploughing, bio-disturbance, or wet-dry cycles. This can cause changes in the physical characteristics of the soil, including its structure, ability to hold water, and bulk density. In a study using PES microfibre, it was discovered that 0.4% may greatly increase the loamy soil's ability to retain water and its rate of water evaporation, as well as noticeably lower the bulk density and water-stable soil aggregates. Additionally, the presence of MPs may compromise the structural integrity of the soil, leading to desiccation cracking on the soil's surface and major alterations in soil moisture and nutrient transport MPs have an impact on the chemical as well as the physical characteristics of soil. High concentration of PP MP (>28%) considerably enhanced the amount of dissolved organic carbon, nitrogen, and phosphorus in soil and encouraged the release of nutrients. The quantities of organic matter, N-OH and accessible P and K in the soil were considerably reduced by the plastic mulch residues. which has a negative impact on soil productivity. The pH level of the soil is also impacted by the different types of MPs. For instance, polylactic acid MPs may significantly raise soil pH whereas PE MPs can significantly lower soil pH. Because of their enormous surface areas and hydrophobicity, MPs are viewed as a poisonous vector. In comparison to soil, PE film residue included substantially greater concentration of pesticides.

MPs in the soil can indirectly impact plant seed germination and seedling growth by reducing the biomass and activity of the soil's microbiological and functional diversity as well as by affecting the nutrient cycle. There haven't been many research done on the possible impact of MPs pollution on plant development. For instance, the incorporation of PE and biodegradable plastics (1% w/w) significantly reduced the grain yield of wheat (Triticum aeasitvum), which was most likely caused by the changes in soil properties (such as changes in nutrient content, water holding capacity, bulk density, and soil structure) induced by the incorporation of plastics.

Because many soil microbes are tiny and have a limited lifespan, it is still difficult to understand how MPs and bacteria interact in the soil ecosystem. The exact process through which MPs affect microorganisms in soil is yet unknown. Based on changes in soil enzyme activity, which is favourably connected with soil microorganism activity, the current research mostly evaluated the possible impact of MPs on soil microorganisms. To some degree, different types, sizes, and shapes of MPs and their exposure levels are responsible for soil microbial activity.[3]

Due to the contamination of the soil environment by MPs, the soil properties, protist communities, plants, and even agricultural ecosystems are affected. According to reports, MPs pollution might have an impact on soil fertility, bulk density, water holding capacity, and stable water aggregates, with PES fibres showing the most obvious impact. Protists were likely to consume μ m-sized or even smaller plastic particles, according to a study on soil protists. Soil protists like earthworms may transmit MPs between soil organisms. [11]

It's essential to assess how MPs affect the soil environment in order to comprehend their toxicological properties. According to, soil is said to be contaminated if it contains even a trace amount (0.1% by weight). Large concentrations of these residues will clog and plug soil pores, reducing the soil's capacity to absorb water over time. This will interfere with the cycle of nutrients in the soil, change the microbial population, and eventually hinder crop development. [14]

2. Effects of MPs on Water

The mobility and breakdown of plastics in the aquatic environment will result in a combination of parent materials, fragmented particles of various sizes, and other non-polymer degradation products.

MPs environmental consequences primarily include the chemical effects of adsorbed pollutants, the danger of plastic additives, and possible threats to creatures and people. One of the greatest issues concerning MPs is the possible damage to aquatic species. MPs can move across tissues and remain inside corals and bivalves after being consumed. Additionally, MPs have been shown to cause hepatotoxicity, neurotoxicity, and inflammation. There have also been reports of immunotoxicity, changes in olfactory-mediated behaviours, infertility of metabolism, blockage of the digestive tract, and other undesirable effects.

Plastic additives represent a serious harm to the biota even at very low concentrations and have the ability to discharge noxious chemicals into the environment. Additionally, it is hypothesised that MPs with a large specific surface area facilitate the leaching of chemicals. As a result, aquatic creatures exposed to MPs may have endocrine disruption and possibly experience changes in their locomotion, growth, and reproduction. [3]

MPs were also found in algae, fish, shrimp, and other freshwater creatures, which might lead to harmful consequences. MPs have the potential to harm animal liver and gastrointestinal systems as well as prevent phytoplankton from photosynthesis. A recent study discovered that MPs might encourage Microcystis aeruginosa production. This type of algae may lead to cyanobacterial blooms in water, which are harmful to both human health and the ecology of the water.[11]

3. Effects of MPs on Human Health

In general, MPs are thought to be inert materials that are easily absorbed by people or other living things, have a low chemical reactivity, and are tiny in size. Recently, they have even been discovered in human faeces. According to research, MPs s can enter animal cells through macrophages or vascular endothelial cells. Humans may consume MPs directly from soil or groundwater by drinking the water or by eating plants or animals that have ingested the MPs and passed them on to us through the food chain. Additionally, MPs have the potential to infect the skin or be swallowed through human skin. MPs include a range of additives that, once ingested by the body, may result in undesirable side effects or serious health issues. The presence of MPs in the human body may pose a number of health problems. MPs may have harmful effects, lead to oxidative stress via oxidative stress via free radical generation, immunological responses, alteration of gene expression, genotoxicity, endocrine disruption, neurotoxicity, reproductive abnormalities, transgenerational effects, and behavioural abnormalities, damage human tissue, and result in chronic inflammation. [9][15]

Currently, it is difficult to determine whether extensive regulatory actions are necessary to protect public health and wellbeing globally in harmony with food safety and ecosystem integrity. This is due to the lack of information regarding the potential human health risks associated with MPs exposure. International scientific organisations have emphasised the need for information on how NMPs affect human health in order to offer the essential evidence foundation for successful policymaking. In response to increased public concern about this problem, as well as requests from national health authorities, numerous organisations (including the World Health Organization (WHO), the European Food Safety Authority (EFSA), and Science Advice for Policy by European Academies (SAPEA)) have conducted their first expert reviews of the overall state-of-the-art knowledge and risk categorization in connection to single exposure sources (i.e., drinking water, sea food). [15]

One of the major MPs entry points into the human system is represented by the ingestion of contaminated food [16]. Humans could also assume an estimated intake of 80 g per day of MPs via plants (fruits and vegetable) that accumulate MPs through uptake from polluted soil.[17] The presence of MPs in marine species for human consumption (fish, bivalves and crustaceans) is now well-known. Therefore, following exposure via diet, uptake in humans is plausible, as evidenced by the capacity for synthetic particles smaller than 150 μ m to cross the gastrointestinal epithelium in mammalian bodies, which causes systemic exposure. However, scientists speculate that only 0.3% of these particles are expected to be absorbed, while a lower fraction (0.1%) that contains particles that are bigger than 10 μ m should be capable of reaching both organs and cellular membranes and passing through the blood–brain barrier and placenta. Another MPs entry point to the human body is the aerial one through inhalation. The low deterioration of microfibers has been found in patients suffering from pulmonary cancer as a confirmation of the bio-persistence of these synthetic particles. By attaching to MPs surfaces in order to be protected from UV radiations, microorganisms could reach the lung and become another threat of infections to human health. The last exposure pathway of MPs to the human body could be skin contact through water while washing or while using scrubs and cosmetics that contain MPs. [8]

Sampling of MPs

1. Sample Collection in Soil

Soil samples are so easily affected by human interaction that it is even more difficult to obtain representative samples. It is recommended to use composite sampling, that is, samples from multiple discrete sites of the same sampling area are combined and homogenized into a single sample. Small sampling units $(1 \text{ m} \times 1 \text{ m}, 15 \text{ cm} \times 15 \text{ cm} \text{ and } 20 \text{ cm} \times 20 \text{ cm})$ are the most commonly used techniques. When the depth of pollution is to be understood, stratified samplings should be conducted. The sampling tool of soil are stainless steel corer, shovels, metal grabs or box corers. Soil samples are usually stored in bags made of non-plastic, such as aluminium foil bags or glass bottles. The soil samples were stored at 4°C and naturally air-dried before analysis.[2][6]

Sampling approaches (e.g., judgmental, random, systematic, transect, unaligned grid and stratified sampling) may be considered based on different research objectives on MPs in soils. Sampling points include single and composite samples. The typical sampling tools for MP collection in soils are shovel/scoop/spade and core samples for surface and deep layers, respectively. For the soil environment, a bulk sample of the preliminary first few centimeters may be sufficient whereas core sampling is required for deep layer of the soil given the vertical transport of MPs, agricultural tilling, and topsoil erosion by wind and water. the sampling depth for MPs in soils is ranged from 2 to 40 cm, most of which are in the focus of top 10 cm. a top layer soil sampling ranged from 0 to 20 cm for MP analysis. The reference units for soils and groundwater are particles/items/numbers per kg as dry weight (n/kg) and particles/items/numbers per liter (n/L), respectively. [10]

2. Sample Collection in Water

Table 3 Methods of sample collection in water [17]			
ТҮРЕ	ADVANTAGES	DISADVANTAGES	
Neuston and Manta nets	Easy to use; Sample large volumes of water; Largely used (good to compare between locations); Produces large numbers of MPs for further testing.	Expensive equipment; Requires boat; Time- consuming; Potential contamination by vessel and tow ropes; Lower limit of detection is 333 µm.	
Plankton net	Easy to use; Lowest limit of detection 100 μ m; Quick to use; Samples medium volumes of water.	Expensive equipment; Requires boat; Static sampling requires water flow; May become clogged or break; Sampling of lower volumes of water than Manta trawl	
Sieving	Does not require specialized equipment nor boat; Easy to collect samples	Laborious and time consuming; Samples medium volumes; Manual transfer of water with buckets	
Pumps	Samples large volumes of water; Effortless; Allows choice of mesh size.	Requires equipment; Requires energy to work; Potential contamination by the apparatus; May be difficult to carry between sampling locations	
Filtration or Sieving ex situ	Easy to collect samples; Known volume of water; Allows choice of mesh size.	Sampling of low volumes; Transportation of water samples to the lab; Potential contamination by the apparatus; Time consuming depending on mesh size. Variation with sampled area and depth.	

Sample Preparation

The general sample preparation steps for MPs in soils are dying, grinding, and sieving. Air-drying (25–35 °C) or machine-drying (35–60 °C) is typically used to remove moisture content from soil samples before grinding. However, grinding with mortars or wood hammers may damage samples, leading to further fragmentation of MPs. Thus, it is used occasionally to prevent big aggregates. As a sample preparation method instead of separation, sieving is often used for sample size reduction and sortation (usually < 5 mm), and even distribution. Compared to the traditional soil sieving method (mesh size < 2 mm or even < 1 mm), the recommended mesh size is 5 mm (particles > 5 mm are discarded) for MP-containing soil samples. The range of sieving mesh size is from 1 to 5 mm in previous studies. Additionally, metal materials are recommended for sample sieving to prevent MP contamination. Coning and quartering or multipoint method is also recommended for sub-sampling. For MPs in water samples including groundwater samples, sieving is also frequently used. [6]

Sample Preparation Method [18]

- Physical method a.
- \triangleright Sieving
- ⊳ Filtration
- Visual sorting
- Density separation
- b. Chemical method
- AAAA Digestion of sample
- Acid digestion
- Alkali digestion
- Digestion by other chemical
- c. Biological / enzymatic digestion
- d. Self - contamination

PROCEDURE	METHOD	ADVANTAGES	DISADVANTAGES
Separation g	Density separation	Pre-concentration of plastic; simplify detection of plastic in further analysis	Might be hampered by adherent soil components; insufficient for removal of organic matter
	Oil separation	Use lipophilicity of most plastics to separate MPs	Varied recovery rates
	Vertical density gradient separation	Use liquid containing colloidal magnetic particles to separate MPs	Little is known for MP separation; expensive
	Electrostatic separation	Suitable for water, sediments and sand; more gentle method than acid treatments, no degradation of plastic	Time consuming; not suitable for moist sample; suitability for soil MPs separation needs to be tested
Digestion	Acid/base digestion	High removal of organic matter in sediments and biological samples	Digestion of plastic itself; time consuming; inaccurate
	H2O2 oxidation/ Fenton's reagent	Used in soil science for removal of organic matter; suitable for water, soil and	Digestion of plastic; temperature control; aging may affect results

Table 4 Different extraction methods [6]

		sediment samples; better than acid/base digestion	
	Enzymatic digestion	Suitable for water samples and marine organisms; more gentle method than acid treatments; no degradation of plastic	Suitability for soil organic matter removal needs to be tested; expensive
	Manual extraction	The simplest way; can combine other technologies such as hot needles	Time consuming; inaccurate
Other extraction methods	Pressurized fluid extraction	low price; high efficiency; extract the size of MPs $< 30 \ \mu m$	Not allow larger plastic fragments extraction (>30610 μ m); may change the particle morphologies
	Magnetic extraction	Use particle hydrophobicity to extract MPs	Nonspecific binding; need post extraction; interfere of iron containing particles
	Froth flotation	Use particle hydrophobicity to extract MPs	Nonspecific binding
	Liquid extraction	Fast; large sample amount; molar mass of polymer contents	Limit to some types of polymers; may damage sample

Analytical Techniques for Identification and Chemical Composition Detection of MPs



Figure 2 Different analytical techniques for detection of MPs[3][19]

METHODS	ANALYZABLE PARTICLE SIZE
ATR-FTIR	>500 µm
μ-FTIR	20-500 μm
FPA-based µ-FTIR	5–10 µm
NIR	>1 mm
1H NMR	Not affected by particle size
Hyperspectral Imaging Technique (HIT)	Not affected by particle size
Raman spectroscopy	>1 µm
Pyr-GC/MS	Not affected by particle size (>500 µm)
TG-MS	Not affected by particle size
TED-GC-MS	Not affected by particle size
Optical microscope/ SEM	>100 µm
AFM	50 to 100 nm

Table 5 Different analytical methods with their analyzable particle size[3][8][10][18-23]

METHODS	ADVANTAGES	LIMITATIONS
ATR-FTIR	Easy simple preparation; low cost	Unable to identify small samples; unsuitable for convex particles or severely aged or contaminated samples
μ-FTIR	Suitable for small samples	Sample preparation is complex; unable to analyze PAs; high cost
FPA-based µ-FTIR	Suitable for small samples; no need for visual sorting	Time consuming; high cost
NIR	It penetrates deeper in the plastic, no pre- treatment is required,	Cannot identify shape and chemical characteristics; low detection accuracies; poor detection limit
H NMR	Cost efficient; fast	require a complete organic digestion of the sample
Hyperspectral Imaging Technique (HIT)	Accurate; non-destructive; provide spatial distribution, size and specific spectrum for samples	Limit to sample size and shallow soil sample
Raman spectroscopy	better spatial resolution than FTIR. Suitable for very small samples; lower water interference	Sensitive to fluorescence interference; laser- induced degradation; high cost
LC-MS/LC-UV/HPLC	Good detection limit; no need for density separation; mass input of samples; fast; automatic	Suitability for soil and water sample needs to be tested
Pyr-GC-MS/ TED-GC-MS/ TG-MS	Organic plastic additive can be analyzed. comprehensive MPs analysis for chemical structure and component separation	Destructive, time consuming, no information on sample size, shape or color, prone to contamination. limit to sample amount per run.
Optical microscope/ SEM	Offer surface topography, crystalline structure, chemical composition; can couple with EDS for elemental analysis	Time consuming; expensive; coating of sample; conduct only in vacuum atmosphere
AFM	Three-dimension images; conduct an ambient or fluid environment; no need for coating samples	Suitability for soil and water sample needs to be tested
TOF-SIMS	Non-destructive	Only provide size and polymer composition of MPs

II. LITERATURE REVIEW

Detection of MPs in Soil

1. Wander L. and Lommel L. (2022) has developed Near-infrared (NIR) spectroscopy for mass quantification of MPs in soil sample. They used flotation of MPs in a NaCl solution, accelerated by centrifugation, which allowed to remove up to 99% of the matrix at recovery rates of 83%-104% of the stainless-steel filters used to collect the extracted particles. Partial least squares regression models were used to identify and quantify the extracted MPs in the mass range of 1–10 mg. The simple and fast extraction procedure was systematically optimized to meet the requirements for the quantification of MPs from common PE, PP, and PS-based packaging materials with a particle size <1 mm found in soil with high natural organic matter content (>10% determined by loss on ignition). High recovery rates above 80% were obtained for all three polymer types and polymer particle sizes [23]

2. **Kononov A. and Hishida M. (2022)** has extracted MPs from agricultural soil by canola oil and **density separation process** using sodium chloride (NaCl). They have quantified low density PE, PP and PVC. 1) soil and MP mixtures were prepared, 2) 5 g L -1 NaCl and canola oil were added and shaken thoroughly, 3) the oil phase containing MPs were separated after sedimentation, 4) the extracted MPs were rinsed with 99.5% ethanol, and 5) the organic adherents to the extracted MPs were digested with hydrogen peroxide. After drying and incineration, the substantial recovery rates were calculated. In the spiking and recovery test for MP particles (<1 mm) from 5 agricultural soils, the recoveries of LDPE, PP, PVC were 95.2-98.3 %, 95.2- 98.7% and 76.0%-80.5% respectively, higher than those obtained by the density separation using saturated NaCl solution. [24]

3. **Müller A. and Goedecke C. (2020)** has performed **liquid chromatography with UV detection** (LC-UV) for the estimation of PET in **agricultural soil and sewage waste water treatment plants.** They present a simple, specific tool for the analysis of PET MPs particles based on alkaline extraction of PET from the environmental matrix and subsequent determination of the monomers, terephthalic acid, using liquid chromatography with UV detection (LC-UV). The applicability of the method is shown for different types of PET in several soil-related, terrestrial environmental samples, e.g., soil, sediment, compost, fermentation residues, but also sewage sludge, suspended particles from urban water management systems, and indoor dust. Recoveries for model samples are between 94.5 and 107.1%. Limit of determination and limit of quantification are absolute masses of 0.031 and 0.121 mg PET, respectively. In order to verify the measured mass contents of the environmental samples, a method comparison with thermal extraction-desorption-gas chromatography–mass spectrometry (TED-GC/MS) was conducted. Both methods deliver similar results and corroborated each other. PET mass contents in environmental samples range from values below LOQ in agriculture soil up to 57,000 mg kg–1 in dust samples. [25]

4. **Scopetani C. and Chelazzi D. (2020)** has performed **oil-based extraction** technique. They have extracted MPs in soil sample includes PE, PS, PVC, PC, PET and polyurethane and analyzed using **FTIR**. The obtained results are promising, and the polymer density had only a small role in the recovery rate: low, medium and high density polymers reached a mean recovery rate of 90% $\pm 2\%$, 97% $\pm 5\%$ and 95% $\pm 4\%$, respectively.[26]

5. **Dierkes G. and Lauschke T. (2019)** has performed **pressurized liquid extraction (PLE) and pyrolysis GC-MS**. The automated extraction includes a pre-extraction step via methanol followed by a subsequent PLE using tetrahydrofuran. For the most frequently used synthetic polymers PE, PP, and PS, limits of quantification were achieved down to 0.007 mg/g. Recoveries above 80% were attained for solid matrices such as soil and sediments. The developed method was applied for MP quantification in roadside soil and potting soil sample and sewage sludges. In all these matrices, PE and PP were detected with concentrations ranging from 0.03 to 3.3 mg/g. In sewage sludge samples, all three polymers were present with concentration levels ranging between 0.08 ± 0.02 mg/g (PP) and 3.3 ± 0.3 mg/g (PE). The analyzed potting soil contained PE in similar concentration as in sewage sludge, but only traces of PP were detected and PS was not detected at all. [27]

6. Yu J. and Wang P. (2019) has performed TGA coupled to FTIR to identify and quantify certain MPs in soil sample. Samples were pyrolyzed in TGA and the pyrolysis gases were analyzed by FTIR. The result of field sample experiments showed that TGA-FTIR can be used to identify and quantify PVC and PS in soil. Recovery rates for PS and PVC were obtained between 97%–101.5% and 98.0%-108.0% respectively.[28]

7. **Zhang S. and Yang X.** have performed **floatation method** to extract the light density MPs from agricultural soil. They have identified MPs using a heating method. ((3–5 s at 130 °C). The number and size of particles were determined under **microscope**. The floatation and heating can be used to extract, distinguish and quantify light density PE MPs in soils. with recovery rates of approximately 90%. After being exposed to heat, the MPs in the soil samples melted and were transformed into circular transparent particles while other impurities, such as organic matter and silicates were not changed by the heat. Regression analysis of MPs weight and particle volume (a calculation based on image J software analysis) after heating showed the best fit (y = 1.14x + 0.46, R2 = 99%, p < 0.001). Recovery rates based on the empirical model method were > 80%. They prove that their method of repetitive floatation and heating can be used to extract, distinguish and quantify light density PE MPs in soils. MPs mass can be evaluated using the empirical model. [29]

8. **David J. and Steinmetz Z**. has performed **thermogravimetry- mass spectroscopy** to estimate MPs in soil sample. They have estimated PET. For this, soil sample containing 1.61 ± 0.15 wt% organic matter were spiked with 0.23-4.59 wt% PET bottle recyclate MPs. DL-cysteine was used as internal standard (IS).sample matrix were pyrolyzed with a 5 K/min ramp(40-100°C) while recording sample mass loss and MS signal intensity of typical PET pyrolysis products. They found MS signal intensities linearly responding to MPs concentrations. The most promising results were obtained with the IS-corrected PET pyrolysis product vinylbenzene/Benzoic acid (m/z=105, R2 = 0.987). Limit of detection and quantification were 0.07 and 1.72 wt% PET. Respectively. Their result suggest that TGA-MS can be easy and viable complement to existing methods such as pyrolysis or thermogravimetry-thermal desorption assays followed by gas chromatography-mass spectroscopy detection or to spectral microscopy techniques.[30]

9. Watteau F. and Marie-France Dignac (2018) has performed Transmission Electronic Microscopy (TEM-EDX) and Pyrolysis coupled to gas chromatography and mass spectroscopy (Py/GC/MS) in agricultural Soil Amended with Municipal Solid Waste Composts. They detected titanium and barium in the soil, as these are constituents of the plastic and are added during polymer production. Their results provide evidence that plastics and MPs were present in the soil that was amended for 10 years with compost, but not in the control soil. MPs were mostly observed as individualized particles, present in the coarsest fractions as well as some of the fine soil fractions, but they were little associated with the soil matrix.[31]

10. **Fuller S. and Gautam A. (2016)** has performed **Pressurized Fluid Extraction (PFE)** for quantification of MPs in municipal waste samples and soil sample. They analyzed the MPs using **FTIR Spectrophotometer**. The method was initially developed by recovering 101% to 111% of spiked plastics on glass beads and was then applied to a composted municipal waste sample with spike recoveries ranging from 85% to 94%. The results from municipal waste samples and soil samples collected from

an industrial area demonstrated that the method is a promising alternative for determining the concentration and identity of MPs in environmental samples.[32]

11. Dümichen E. and Anne-Kathrin Barthel (2015) have performed thermal desorption gas chromatography mass spectrometry (TDS-GC-MS) for estimation of MPs in soil sample. This was achieved by thermal extraction in thermogravimetric analysis (TGA), connected to a solid-phase adsorber. These adsorbers were subsequently analysed by thermal desorption gas chromatography mass spectrometry (TDS-GC-MS). the average of the determined concentration of PE in soil (4.9 ± 0.8 wt%) using the three decomposition products is in good accordance with the real PE concentration in the sample (5.0 wt%). This is not surprising because the calibration curves for PE in soil achieved an R2 factor of around 0.99 and the relative error of the method is known from former investigations to be ~10%.[33]

Detection of MPs in Water

1. **Gomiero A. and Plamas L. (2021)** has performed **pyrolysis gas chromatography-mass spectrometry** sample's analysis (pyr-GCMS). They have estimated MPs in drinking water. Samples has been taken at different stages of the drinking water supply chain. The total amount of polymers per sites ranged from 6.1 to 93.1 μ g/m3. They have detected the higher levels in the raw water, but significant reduction rates ranging from 43% to 100% depending on the polymer type were scored after the water treatment processes. PP, PAs, and PES were the most frequently detected polymer types.[34]

2. Hong Y. and Oh J. (2021) has performed micro-Fourier transform infrared spectroscopy (μ -FTIR), Raman spectroscopy, or pyrolysis–gas chromatography/mass spectrometry (Py-GC/MS) for estimation of MPs in sewage treatment plant. have proposed an innovative method which quantifies total organic carbon (TOC) of plastic particles to estimate the mass of MPs in sewage. Then, the method was applied to evaluate the fate of MPs in sewage flowing into and out of a sewage treatment plant (STP). In the proposed method, sewage samples were collected and filtered using a sampling module equipped with stainless steel filters to harvest particles between 45 μ m and 500 μ m. Then, the retentates of the filter were digested by Fenton's reagent to remove organic matters other than plastic particles before TOC determination. The method detection limit of the proposed method was 0.003 mg (0.15 μ g L-1 for a 20 L sample), and the recovery efficiencies estimated with six different types of plastic particles were ranged from 76% to 98%. Using the proposed method, the performance of a STP in Seoul in excluding MPs from sewage was evaluated; more than 99% of MPs could be removed.[35]

3. **Funck M. and Yildirim A**. (2020) has performed **Pyrolysis-Gas chromatography–Mass Spectroscopy (Py-GC–MS)** for estimation of MPs in waste water treatment plants. They developed steel based cascadic MPs filtration uses steel basket filters with mesh sizes of 100mm, 50 mm and 10mm and a mean recovery of 86 % without cross contamination. The presented platinum filament based Py-GC–MS method requires little sample preparation and quantification limits for PS and PE were 0.0 3µg and 1µg absolute, respectively. The relative standard deviation of the analytical method is 11 %. The combined method allows representative sampling and analysis of MP from water bodies and waste water treatment plants within 48 h.[36]

4. Becker R. and Altmann K. has performed pyrolysis gas chromatography – mass spectrometry (Py-GC-MS), thermal extraction desorption followed by gas chromatography coupled to mass spectrometry (TED-GC-MS), thermogravimetry-infrared spectroscopy (TGA-FTIR) and thermogravimetry coupled to mass spectrometry (TGA-MS). They identified MPs in sedimented freshwater suspended organic matter fortified with particles of PE, PP, PS, PET was employed in an interlaboratory comparison of thermos-analytical methods for MPs identification and quantification. It was seen that the participants per forming Py-GC-MS, TED-GC-MS, and TGA-FTIR were able to correctly identify all polymers and to report reasonable quantification results in the investigated concentration range, PE: 20.0 µg/mg, PP: 5.70 µg/mg; PS: 2.20 µg/mg, PET: 18.0 µg/mg.[37]

5. Wolf S. and Kerpen J. (2019) has performed Raman micro spectroscopy for estimation of MPs in waste water treatment plants. They collected samples using a stainless-steel centrifugal pump and filtered through a 10 mm stainless steel cartridge filter. MPs particles (MPPs) and MPs fibers (MPFs) were recovered by chemical and physical sample purification. Inorganic materials were subsequently removed by density separation in ZnCl2 (r ¹/₄ 1.9 g/cm3) using a centrifuge. Special centrifuge tubes were developed for this purpose. Sample analysis was performed on a Si filter by Raman micro-spectroscopy. MPPs detected were in the 30 mm < dp < 100 mm size range. The MPFs ranged between 100 mm and 1000 mm in length.[38]

6. **Majewsky M. and Bitter H. (2016)** has performed **TGA-DSC** to estimate MPs in wastewater samples. They identified PE and PP in waste water samples. calibration curves were run for PE and PP for qualitative measurements. 240 and 1540 mg/m3 of solid material (12μ m to 1mm) was extracted from two wastewater effluent samples of a municipal WWTP of which 34% (81 mg/m3) and 17% (257 mg/m3) could be assigned to PE, while PP was not detected in any of the samples. The presented application of TGA-DSC provides a complementary or alternative method to FT-IR analyses for the determination of PE and PP in environmental samples.[39]

III. SUMMARY AND CONCLUSION

From the various literature review, we concluded that different analytical methods were used such as; TDS-GC-MS, NIR, TG-MS, TEM-EDX, PY/GC/MS, FTIR, MICRO-FTIR, LC-UV, TGA-FTIR, PLE-GC-MS, TGA-DSC, TGA-MS, TED-GC-MS, can be used for analysis of MPs from different soil and water samples. From results and literature reviews we can conclude that above methods are efficient for estimation of MPs in soil and water. In microscopic analysis, only the size of the MPs N 20 µm could be clearly detected based on the resolution of the microscope and the camera combination. This method can't be used to distinguish the chemical components of MPs, which can be detected using the method of thermal analysis, IR or Raman micro-spectroscopy. A comprehensive particle size distribution cannot be obtained with the Py-GC–MS, only spectroscopic methods like RAMAN and FT-IR are suitable for this propose. FTIR is inexpensive and can be applied to isolate various polymers from solid samples. FTIR is used for the identification and down to 5 µm with a microscope-FTIR instrument. µ-FTIR or GC/MS and TGA-MS uses only a small amount of digestion reagent and requires less time. TGA-DSC is user-friendly and cost-effective. In NIR method, there is no

need for cleaning or other breaks between samples. The maintenance and operating costs of a compact NIR spectrometer are extremely low compared to instruments required for thermos-analytical analysis The devices are very simple and does not require expert knowledge. PLE-GS-MS method is not able to determine the number and the size of the particles. TGA-FTIR is cost-effective method. The total mass of PVC, PS and PA can be measured directly by TGA-FTIR. But the method is destructive and can't get information about the size, shape and items of MPs. So, it can be used as a complementary or alternative measurement to micro-FTIR imaging techniques. PE, PP, and PET are not as distinguishable and cannot be reliably determined by this method. LC-UV is easy to automatize, a measurement took only 30 min and evaluation of the data did not need any high user knowledge.

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