Source, Characterization and Analysis Methodology of Microplastics in River Sediment: A Literature Review

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PROJECT REPORT APPROVAL

It is hereby certified that the work presented in this thesis was carried out by the following final year students of session 2020-2021 under the direct supervision of Dr. Md. Rezaul Karim, Professor, and Head of Department of Civil and Environmental Engineering (CEE), Islamic University of Technology, Gazipur, Dhaka.

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DECLARATION OF CANDIDATE

It is hereby declared that this entitled "**Source, Characterization and Analysis Methodology of Microplastics in River Sediment: A Literature Review**" thesis/project report or any part of it has not been submitted elsewhere for the award of any Degree or Diploma (except for publication).

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DEDICATION

We dedicate our thesis work to Mr. Jamilur Reza Choudhury (1942-2020), a legendary civil engineer who was awarded Ekushey Padak in the category of science and technology in 2017 and inducted as a National Professor in 2018 by the Government of Bangladesh.

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"In the name of Allah, Most Gracious, Most Merciful"

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ABSTRACT

Microplastic pollution has gained global attention as an emerging environmental issue considering ambiguity, chemical inertness, and adverse impact. This study illustrates significant sources of microplastics and their pathways to the river sediment. Degradation and biofouling- two concrete terms related to the fate of microplastics in the aquatic environment are briefly explained in this study. Moreover, this study summarizes the outcomes and methodologies of some peer-reviewed articles on microplastics in river sediment. The result indicates great geographical variation in the concentration of microplastics around the globe. High concentration of microplastics was recorded in Wen-Rui Tang river, China, with an average abundance of 32947±15342 items/kg, whereas in the Qin river, the concentration of microplastics ranges from 0 to 97 items/kg only. Fibers, transparent and small-sized microplastics are predominant in shape, color, and size, respectively, in most studies. In terms of chemical composition, Polyethylene (PE) and/or Polypropylene (PP) are the dominant types of microplastics. However, a wide variation in technical approaches for microplastic analysis is observed in various studies. This study includes comparative discussion on different sampling methods and tools, benefits and drawbacks of various chemicals used for digestion and density separation, as well as ease and difficulties of different spectroscopy. Last but not least, some recommendations are proposed in the conclusion of this study for the advancement of research on microplastics in the future.

CHAPTER ONE: INTRODUCTION

1.1 Background

Plastics can be defined as polymer-based materials manufactured from by-products of fossil fuels and usually processed with a variety of chemical additives (Fan et al., 2019; Gong & Xie, 2020). Due to light-weight, low cost, durability, high persistency, and sound insulation property, the application of plastics has increased exponentially in the last few decades (Gong & Xie, 2020; Tang et al., 2020). More than 8 billion tons of plastics have been produced since their invention, and approximately 55% of them were fabricated in the last two decennaries(Khalid et al., 2021). From 2010 to 2018, in just eight years, global production of plastics has been estimated to rise by 80 million tons (A. Lusher et al., 2017; D. Zhang et al., 2020). Even in 2020, the production of single use plastic bags alone was around 0.84 million tons (Foschi & Bonoli, 2019). Plastics with a size range between 100 nm to 5mm refer to microplastics, which can be both directly manufactured at this size range or deteriorated from larger plastics by environmental processes (Stock et al., 2019).

Microplastics are omnipresent throughout the world and reported to be detected even at sea around Antarctica, where population density is almost zero (Barnes et al., 2010). Microplastics cause more danger to the environment than larger plastics(L. Zhang et al., 2020), and it is estimated that almost 10% of the total plastic litters in the aquatic environment eventually converts into microplastics by various external forces such as UV radiation, heat, water, biota, etc. (Wu et al., 2020).

From tiny-sized resin pellets to extensive packaging material, no matter the size, any product made of plastic have the potential to contribute to the occurrence of microplastics in river sediment. However, the significant sources of microplastics include personal care product(Sun et al., 2020), pellet (Yurtsever, n.d.), blasting agent (Duis & Coors, 2016), fabric

(Dalla Fontana et al., 2020), packaging material (Foschi & Bonoli, 2019), vehicle tire (Chen et al., 2020), fishing gear (Xue et al., n.d.), etc. The distribution of microplastics around the globe is not uniform and mainly depends on the nature of the aquatic system, climate, as well as characteristics and sources of microplastics (Redondo-Hasselerharm et al., 2018). Several researchers had investigated the condition of microplastic pollution in river sediment at various locations (Constant et al., 2020; Eo, 2019; Jiang et al., 2019; Nel et al., 2018; Peng et al., 2018). Nevertheless, the results of these studies are difficult to compare as they have performed microplastic analysis using different methodologies. Moreover, a wide range of variation is observed in the criteria of microplastics classification depending upon researchers. For example,(Wu et al., 2020) classified microplastics into four shapes such as fiber, foam, film, and fragment but (Liu et al., 2021) separated lines and fibers into two different categories.

1.2 Significance of the study

Microplastics can be easily uptaken by aquatic creatures and conveyed to the food web due to their weeny size (Carbery et al., 2018). Till 2017 the evidence of microplastics ingestion by aquatic biota was found in more than 690 different species (Provencher et al., 2017). For instance, In Norway, microplastics were reported in 83% of the total lobster samples(Setälä et al., 2014). Ingested microplastics can accumulate in the digestive tracts and cause sever torment in the tissues and organs of aquatic animals (W. Wang et al., 2019). The extent of the negative impact can be so immense that it can alter natural growth, body weight, jumping height, swimming velocity, pulse rate, and even mortality rate depending on the nature of exposed organisms and characteristics of microplastics(Khalid et al., 2021; W. Wang et al., 2019). Due to small size, large specific surface area, hydrophobic property, and high adsorption ability, microplastics provide a good surface for the assemblage of heavy metals,

pathogens, persistent organic pollutants, and other toxic chemicals (Gong & Xie, 2020). The combination of microplastics and these pollutants poses more risk and toxicological effect on the aquatic environment than microplastics alone (W. Huang et al., 2021).

Microplastic pollution is considered one of the most significant environmental issues due to its pervasiveness and massive adverse impacts. However, it is believed that 80% of the total microplastics in the marine environment have been moved from the land through river networks (Horton et al., 2017), relatively less attention has been given to investigate the state of microplastic pollution in rivers than the ocean (Adomat & Grischek, 2020; Simon-Sánchez et al., 2019; Yang et al., 2021). From benthic sediment to the shoreline, microplastics reside everywhere in a river system (Li et al., 2018), and their vertical distribution largely depends on the flow velocity of the river, density, and shape of the particles (Waldschläger et al., 2020). Usually, light particles with a density lower than water float in the water column unless biofouling occurs and increases its density (Andrady, 2011; Coyle et al., 2020). Almost 50% of the microplastics in the aquatic environment pose higher density than water and subsequently settle down in the sediment (Ballent et al., 2013). Assessment of microplastic pollution in river sediment is essential for its role as a sink (Matsuguma et al., 2017). In contrast, accumulation of microplastics in river sediment can alter some physical properties of the sediment, such as bulk density, water-holding capacity(Adomat & Grischek, 2020), and affect the functioning of benthic organisms (Bour et al., 2018). Moreover, microplastics from sediment can return back to water column due to de-fouling, erosion and high flow velocity (Adomat & Grischek, 2020; Andrady, 2011; Waldschläger et al., 2020) .

1.3 Objectives of the study

The followings are the key aim of this research project:

- To sort out the potential sources and transmission pathway of microplastics in the river sediment.
- To analyze the current condition and driving factors of microplastic pollution, as well as characteristics of microplastic in the river sediment.
- To provide a comparative discussion between different methodologies used for the analysis of microplastics in the river sediment.

1.4 Literature review strategy

In December 2020, “microplastics” in combination with “river” and “sediment” were used as keywords to find out articles on microplastics in river sediment from ScienceDirect (https://www.sciencedirect.com/) database. Articles assessing surface water and sediment of other water bodies such as ocean, lake, reservoir were excluded. Publications on both riverbed sediment and shoreline sediment were included. A total of 27 studies published since 2017 were selected. Besides, some relevant topic-wise searches such as biofouling, photodegradation, biodegradation, FTIR, Raman, etc. was also performed on google scholar (https://scholar.google.com/).

CHAPTER TWO: SOURCES AND PATHWAY OF MPS IN RIVER SEDIMENT

Identification of the sources of microplastics is important to perceive the pathway and impact of microplastics and to evolve the mitigation strategies (Briain, n.d.; Tang et al., 2020)). To develop an understanding of the sources of microplastics, we need to differentiate between primary and secondary microplastics (Waldschläger et al., 2020). Primary microplastics are designed and produced at the size range of microplastics, (<5 mm) which can be used as the raw material of personal care products, cosmetics, and other industrial products (Gong & Xie, 2020; Simon-Sánchez et al., 2019; Yang et al., 2021). On the other hand, secondary microplastics are the result of physical, chemical, and biological degradation and fragmentation of larger plastics when exposed to the environment(Fred-Ahmadu et al., 2020; Waldschläger et al., 2020)). Cracks, ditches, and fouling are good indicators of this deterioration process (Yang et al., 2021).

2.1 Sources of primary microplastics

Microplastics (microbeads, sodium tetraborate decahydrate, etc.) with a diameter of less than 5mm are used as polishing agent in personal care products such as cosmetics, hand sanitizer, facewash to remove dead cells from the surface of the skin (Duis & Coors, 2016). An investigation on peeling, toothpaste, body wash, and scrub found that the amount of polyolefin microplastics used in consumer products ranges from 0.45 %(w/w) to 7.48 % (w/w) (Hintersteiner et al., n.d.).Another study found that approximately 94500 breads could be exerted from each facial cleansing product(Ngo et al., 2019). Moreover, on average, 2450 particles/g were detected in facewash, and in Slovenia, this count reaches the maximum

(3.11×106 particles/g)(Sun et al., 2020). Comparatively, fewer microplastics (2.15 particles/g) were found in body wash (Sun et al., 2020).

Resin pellets used in the production of plastic and other industrial activity is one of the significant sources of primary microplastics (Duis & Coors, 2016; Yang et al., 2021; Yurtsever, n.d.). Though numerous studies were conducted on the occurrence of plastic production pellets in the beach samples (Acosta-Coley & Olivero-Verbel, 2015; Antunes et al., 2013; Turner & Holmes, 2011), investigation in river sediment is still in headway. In Wen-Rui Tang River, pellets were 12.8% of the total microplastics (Z. Wang et al., 2018).

Glitters which can be defined as tiny, smooth, and beautifying material made of biaxially-oriented polyethylene terephthalate (BoPET) also comprise the source of microplastics (Yurtsever, n.d., 2019). An investigation performed on the wastewater treatment plants in Norway found that glitters contribute 1.7% (in weight) of total microplastics detected in the sample (A. L. Lusher et al., 2017)

Microplastics such as acrylic, polyester (PES) used as blasting agents to remove paint or other contaminants from the metal surface, roughen any surface or clean mechanical engines are another possible source of primary microplastics (Duis & Coors, 2016; Waldschläger et al., 2020).

2.2 Sources of secondary microplastics

Due to high removal efficiency in the sewage treatment plant and proper caution during handling, usually less primary microplastic is identified in the river sediment (Duis & Coors, 2016; Gong & Xie, 2020). So, secondary microplastics are the main contributor of microplastics in the river (Yang et al., 2021).

Around 60% of the total manufactured fibers in the world are synthetic fibers such as polyester, acrylic, cotton, nylon (Dalla Fontana et al., 2020). These synthetic fibers can be detached during the laundering process of fabric and disposed into the environment as secondary microplastics (Waldschläger et al., 2020). In Ciwalengke River, around 93% of the detected microplastics in the sediment were fiber, and the result from Raman spectra analysis indicates that these microfibers were produced from shredded fabrics(Alam et al., 2019). Depending on clothing and washing process, one to ten hundred microplastics was extracted from washing effluent in the laboratory with a filter of 5mm width and 4.7mm dia (Falco, 2017). Furthermore, it is estimated that approximately 6000000 microplastics per 5 kg wash load can be released in the effluent of a textile industry (Yang et al., 2021). Even in the domestic wash, the amount of released fiber can be around 700000 per 6 kg wash(Napper & ThoMPSon, 2016). Another study investigated that the rate of microplastics release of finished clothing lies between 175 to 560 microfibers per gram (30000-465000 microfibers per m^2) fabric (Belzagui et al., 2019). The detachment rate of microfibers is comparatively higher in woven polyester, and this rate can be decreased by more than 35% by using softener instead of regular detergent during the washing process(Falco, 2017).

Plastic is a cheap, light-weight material which gives good protection against moisture (Andrady, 2011). Due to these properties, plastic is widely used as packaging material for food, dish, cutlery, and other products (Foschi & Bonoli, 2019). The global production of plastic packages is 75-80 million tons each year(Andrady, 2011). Therefore, in Europe and China, packaging industries are considered as the most substantial source of plastic pollution(Tang et al., 2020). Most of these packages are disposable one-time use products, discarded into the environment, and end up as secondary microplastics. Moreover, there is evidence of generating microplastics during the scissoring or tearing of these packages (Nir, n.d.).

Plastics such as low-density polyethylene (LDPE) are commonly used in the production of rope, floating drilling rig, and other fishing gears used in aquaculture (Tang et al., 2020). Due to abrasion or some other reason, microplastics can shred away from these tools during fishing activities(Chen et al., 2020). So, aquaculture and fisheries are one of the potential sources of secondary microplastics (Andrady, 2011). A study on microplastic pollution due to fishing activities detected571 ± 409 particles/kg sediment in the adjacent suburban rivers of the Beibu Gulf, and this count was even more (735 ± 405 particles/kg sediment) in the adjacent urban river (Xue et al., n.d.). Another investigation was conducted in aquaculture water of Pearl River Estuary where 10.3-60.5 particles/L and 33.0-87.5 particles/L of water sample were extracted in two experimental stations (Ma et al., 2020), which can end up in the river sediment or ocean.

Polymers such as butadiene rubber (BR) and styrene butadiene rubber (SBR) are one of the widely used components of vehicle tires (Waldschläger et al., 2020). While driving, these polymers can wear out due to friction between the road surface and tire (Kole & Löhr, 2017). Thus, wear and tear from vehicle tires are considered as one of the significant sources of secondary microplastics (Ngo et al., 2019). In Japan, approximately 239,762 tons of wear and tear is released from tires each year (Kole & Löhr, 2017), and the emission of microplastics from wear and tear is around 240 kilotons per year(Ngo et al., 2019). Moreover, microplastics from vehicle tires contribute around 3-7% of the total dust, spores, and pollen($PM_{2.5}$ particles) in the air (Kole & Löhr, 2017).

Besides, except the sources mentioned above, construction materials such as pipes, insulating materials, etc., sporting goods such as artificial turfs, goal nets, etc. can also be the potential contributor of secondary microplastics (Waldschläger et al., 2020). However, research on their contribution to microplastic pollution is still in the developing phase.

2.3 Degradation of plastics under aquatic environment

Degradation mainly refers to the decomposition of plastics by chemical alteration (Waldschläger et al., 2020). In other words, degradation incorporates either oxidation or hydrolysis process through which plastic losses its mechanical integrity and molecular weight (Andrady, 2011; Chamas et al., 2020)). It can be induced by various degradation forces such as radiation (photodegradation), heat (thermal degradation), living organism (biological) and water (hydrolytic degradation), etc. (Cassidy & Aminabhavi, 1981). The rate of disintegration of any degradation process is comparatively slower than photodegradation (Andrady, 2011). Thus, in this study, only the process of light-induced degradation, namely photodegradation or photo-oxidation, along with biodegradation will be discussed.

2.3.1 Photodegradation

The mechanism of photodegradation initiates with the absorption of UV-B radiation of sunlight by plastics(Andrady, 2011). UV-B radiation that reaches earth (wavelength 2900-4000 Å) has energy ranges from 72-97 Kcal/mole, which is adequate to disintegrate any chemical bond, with few exceptions such as N-H, O-H, C-H, etc. (Cassidy & Aminabhavi, 1981; Fotopoulou & Karapanagioti, n.d.). The application of sunlight on polymers stimulates a chemical chain reaction in which a hydrogen atom (H•) is removed from an exciting polymer molecule (RH) and produces a free polymer radical (R•) (Chamas et al., 2020).

$$\text{Initiation: RH} \xrightarrow{UV-B} RH^*$$

$$RH^* \rightarrow \text{R•} + \text{H•}$$

This polymer radical (R•) combines with oxygen (O_2) to form a peroxy radical (ROO•) which then reacts with adjacent polymer molecule (RH), extracts hydrogen atom (H•) from it and produces a new polymer radical (R•) as well as a hydroperoxide (ROOH) group (Rånby, 1993).

$$\text{Propagation: R•} + O_2 \rightarrow \text{ROO•}$$

$$\text{ROO•} + \text{RH} \rightarrow ROOH + \text{R•}$$

Hydroperoxide (ROOH) is susceptible to change in the presence of light(Yousif & Haddad, 2013). It breaks down into alkoxy (RO•) and hydroxyl radical (OH•), each of which produces another polymer radical (R•), and thus photodegradation continues through chain propagation (Chamas et al., 2020).

$$\text{Propagation: ROOH} \rightarrow \text{RO•} + \text{OH•}$$

$$2\text{ROOH} \rightarrow \text{RO•} + \text{ROO•} + H_2O$$

$$\text{RO•} + \text{RH} \rightarrow \text{ROH} + \text{R•}$$

Chain propagation terminates when radicals combine together and form non-radical stable products (Rånby, 1993; Yousif & Haddad, 2013).

$$\text{Termination: ROO•} + \text{ROO•} \rightarrow \text{ROOR} + O_2$$

$$\text{R•} + \text{R•} \rightarrow \text{R-R}$$

$$\text{ROO•} + \text{R•} \rightarrow \text{ROOR}$$

β-scission of alkoxy radical (RO•) results in the formation of oxidized groups such as carboxyl, carbonyl, etc., which may promote further chain scission by photolysis of Carbonyl

functional groups (C=O) (Cassidy & Aminabhavi, 1981; Yousif & Haddad, 2013). Carbonyl photolysis advances either through Norrish Type I or Norrish Type II reaction(Rånby, 1993). Norrish Type I reaction refers to photochemically induced homolysis of carbonyl group into two free radical intermediates, whereas Norrish Type II reaction refers to light-induced intramolecular extraction of a γ-hydrogen to produce alkene and enol or enable cyclization of carbonyl compounds to cyclobutanols(Chamas et al., 2020; Scheffer et al., 1986).

As the key role of radiation is to introduce chain initiation reaction, further degradation can proceed at moderate temperature without any exposure to sunlight(Andrady, 2011). So, photodegradation and thermal degradation are indistinguishable under usual conditions (Fotopoulou & Karapanagioti, n.d.). But in the absence of UV radiation, minimum of 100 °C temperature will require to start thermal degradation of Polyethylene (PE) (Chamas et al., 2020). However, studies have found that polyester (PET) and polyamide (PA) are comparatively less persistent and easily degradable than polyacrylonitrile (PAN) under exposure to sunlight (Sait et al., 2021).

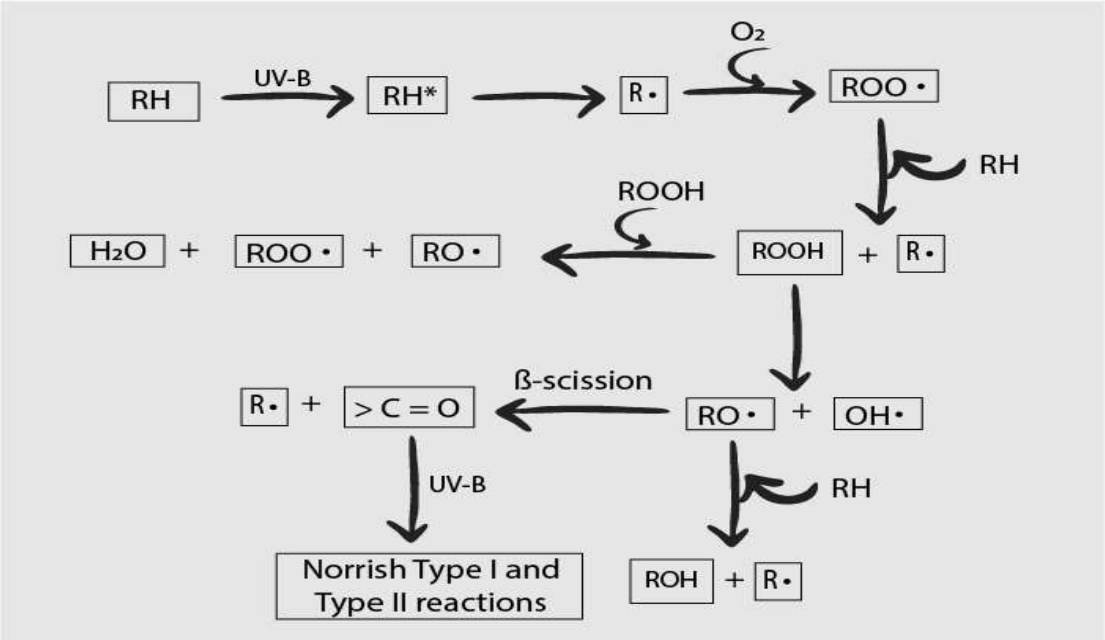

Figure 1: An overview of the photodegradation process of MPs

2.3.2 Biodegradation

After extensive photodegradation and fragmentation by wave, wind, and rain, microplastic particles will undergo biodegradation (Cassidy & Aminabhavi, 1981; Muthukumar & Veerappapillai, n.d.). Biodegradation is a slow process through which polymers convert into biomass and eventually disappear (Andrady, 2011). This process is affected by several factors that include polymer characteristics such as molecular weight, size, shape, surface area, etc., type of organism and nature of their enzyme, characteristics of abiotic exposure such as pH, temperature, moisture, and nature of pre-treatment (Ahmed, 2018; Fotopoulou & Karapanagioti, n.d.; Muthukumar & Veerappapillai, n.d.).The mechanism initiates with the attachment of exoenzymes secreted by microbes to polymer fragments (Ahmed, 2018). The role of exoenzymes is to cleave the polymer chains and convert them into monomers, dimers, or oligomers (Fotopoulou & Karapanagioti, n.d.). Monomers, dimers, or oligomers are light-weight molecules with shorter chains and can easily penetrate bacterial cytoplasm (Muthukumar & Veerappapillai, n.d.). The assimilated molecules are utilized by the microorganisms to produce energy, new cells, and other metabolic products (Cassidy & Aminabhavi, 1981) and converted into water, carbon dioxide (aerobic condition), or methane (anaerobic condition) as the end product (Ahmed, 2018; Muthukumar & Veerappapillai, n.d.).

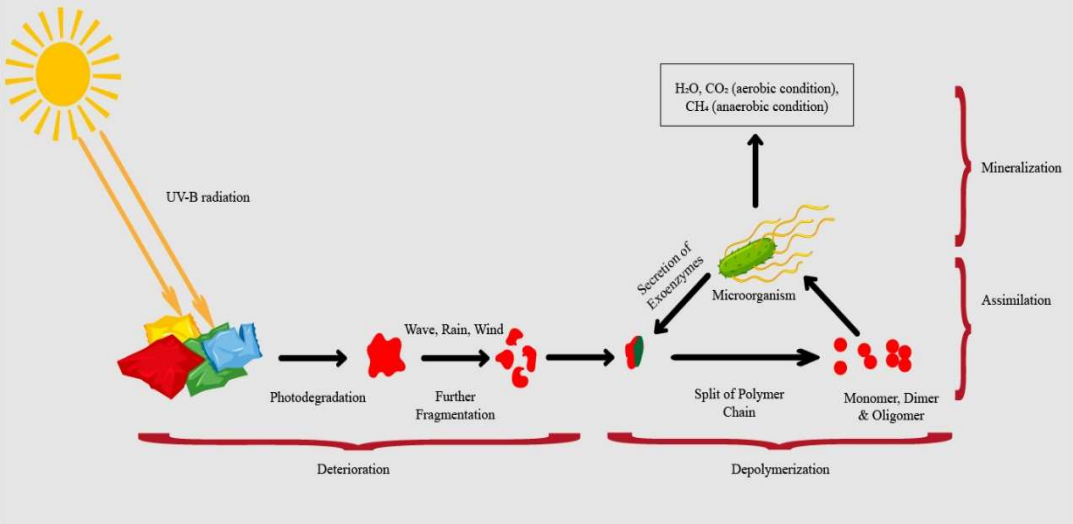

Figure 2: An overview of the degradation process of plastics

2.4 Pathways of MPs to the river sediment

Microplastics do not remain confined to one environmental element; instead, they migrate from one to another(Gong & Xie, 2020). Pathways of microplastics to the river sediment can be described from the following perspective: a) direct discharge of microplastics by land-based activities, b) release of microplastics with treated and untreated wastewater, and c) release of microplastics by water-based activities (Tang et al., 2020)

Land is considered the most significant contributor of microplastics in the aquatic environment (Gong & Xie, 2020; Yang et al., 2021). Some portion of microplastics from waste yards, agricultural fields, roads, and other sources directly move into the river with surface runoff; some portion travels into the subsurface first then reaches the river with subsurface runoff (Gong & Xie, 2020; Yang et al., 2021)and rest enters into the sewage system (Waldschläger et al., 2020). Lack of wastewater treatment facility in municipality results in the discharge of microplastics into the river with sewage wastes. For instance, due to the disposal of untreated sewage wastes at numerous points, a moderate amount of

microplastic particles (96 pieces/kg of dry sample) was detected in the shoreline sediment of the Netravathi river (Amrutha & Warrier, 2020). Furthermore, the sewer system with a proper treatment facility can also be a substantial source of microplastic despite its high removal efficiency (Yurtsever, 2019). Similarly, microplastics can migrate into the river with industrial wastewater also. A study found that a single secondary wastewater treatment plan can introduce 23 billion microplastics into the environment annually(Murphy et al., 2016). Sludge from the wastewater treatment plant can be used as landfill and fertilizer in the agricultural field from which microplastics can enter the aquatic environment by the action of wind and rain (Waldschläger et al., 2020). In addition, some microplastics are directly discharged into the river by water-based activities such as navigation, fisheries, and port activities (Tang et al., 2020). In a river with low flow velocity, microplastics with a density greater than water readily settle down and accumulate in the benthic sediment (Nizzetto et al., 2016). In contrast, in a river with high flow velocity, particles will move with the flow into a low-velocity zone and then settle down (Nizzetto et al., 2016). Particles with a density lower than water usually float and end up into the ocean but can be retained in the river sediment by biofouling and agglomeration (Waldschläger et al., 2020). Biofouling refers to the colonization of microorganisms on the surface of microplastics (Andrady, 2011). The process starts with the formation of a biofilm with algae, spores, and other dissolved matter on the surface of microplastics which enables ease attachment of colonizing microbes (Coyle et al., 2020). The density of particles tends to increase with biofouling which allows the particles to sink when it transcends the density of water (Coyle et al., 2020). Usually, microplastics smaller than 0.2 mm do not end up in the river sediment regardless of density (Nizzetto et al., 2016).

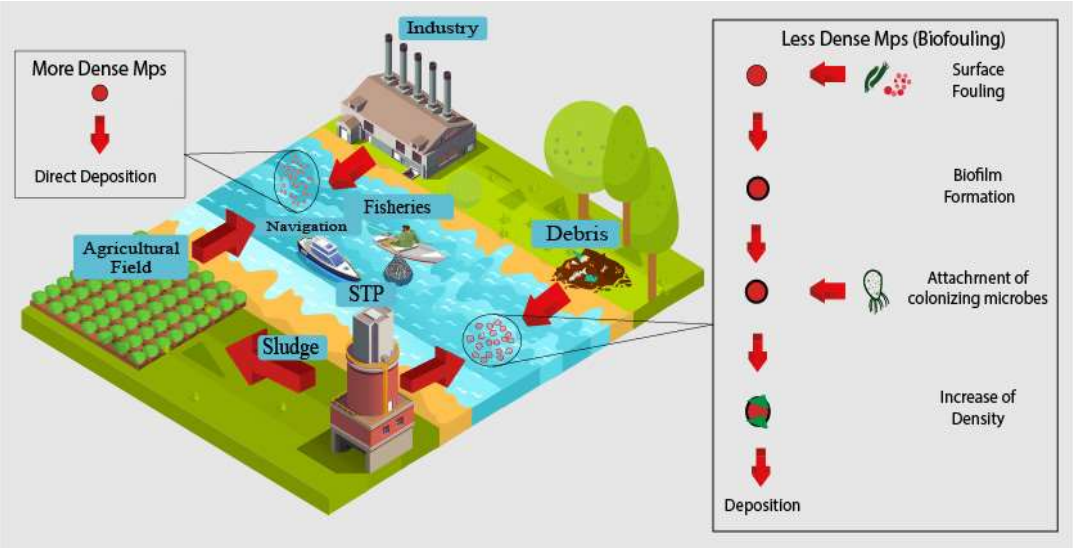

Figure 3:Pathways of MPs to the river sediment

CHAPTER THREE: ABUNDANCE AND CHARACTERISTICS OF MPS

3.1 Factors affecting the occurrence of MPs in river sediment

Abundance of microplastics in river sediment depends on various factors such as- Population density, level of urbanization, and anthropogenic activity of surrounding area; precipitation, wind intensity, tidal current, river width, flow velocity, season, and microplastics properties. Thus, the concentration of microplastics varies significantly around the globe (Jiang et al., 2019).

Since rivers receive household sewage, industrial effluent, and agricultural wastewater, the abundance of microplastics has a positive correlation with population size, urbanization, industrialization, recreational, and other human-induced activities(D. Huang et al., 2020; Nel et al., 2018; Wen et al., 2018; Wu et al., 2020). Though microplastics particles can travel for prolonged distances (Gerolin, 2020), the concentration of microplastics decreases with the distance from the city or industry (Joana C. Prata et al., 2021). However, an effective treatment plant with tertiary technology can eliminate 98% of microplastics pollutants from effluent and prevent WWTP from being a potential source of microplastics(Lin et al., 2018)

Low flow velocity promotes sedimentation of microplastics and thus is negatively correlated with the microplastic concentration in sediment (Tien et al., 2020). This correlation results in low microplastics accumulation during the rainy season(Wu et al., 2020) and high microplastics abundance in comprehensive portion of the river (D. Huang et al., 2020). Subsequently, higher microplastics accumulation is observed during winter than summer due to the decrease in flow rate (Nel et al., 2018; Schmid et al., 2020). Again, Extensive rainfall incorporated with high wind intensity and intense wave action associates the entrance of microplastics from sediment to the water column and reduces microplastics concentration in

sediment (Amrutha & Warrier, 2020). In contrast, there is evidence of increase in microplastics abundance after a typhoon both in water column and sediment(Jun Wang et al., 2019).

Accumulation of microplastics in sediment also depends on microplastics properties like density and surface to volume ratio(Wu et al., 2020). High surface to volume ratio and low density enables polymer to remain in the water as suspension, whereas low surface to volume ratio and high density promotes deposition of polymer in the riverbed(Lin et al., 2018; Liu et al., 2021; Wu et al., 2020).

3.2 Characteristics of MPs

Microplastics can be categorized based on the following characteristics- Shape, size, color, and chemical composition(Fred-Ahmadu et al., 2020). We have reviewed 27 studies to analyze microplastics characteristics and the overview of our investigation is summarized in Table 1.

3.2.1 Shape

Shape of microplastics can be controlled by various factors like source, deterioration process, and retention time (Yang et al., 2021). Fiber, film, pellet, foam, and fragment are the most usual shape of microplastics (Ding et al., 2019; D. Huang et al., 2020; Wu et al., 2020), but some studies included a few additional categories like sheet, sphere (or bead), line and others for the classification of microplastics based on shape (Constant et al., 2020; Fan et al., 2019; Feng et al., 2020).

Fiber is a secondary microplastic, cylindrical in shape, and whose length is significantly higher than its width (D. Huang et al., 2020; Ngo et al., 2019). It usually originates from synthetic clothes during washing and manufacturing process of textile goods, fishing nets, ropes, and sacks(Amrutha & Warrier, 2020; Ngo et al., 2019; Yang et al., 2021). Fibers produced from fishing activity, sometimes defined as lines (Dioses-Salinas et al., 2020) Pellet is a primary microplastic which is spherical or elliptical in shape and usually derived from personal care product such as cosmetics, toothpaste, etc. (D. Huang et al., 2020; Kutralam-Muniasamy et al., 2020; Ngo et al., 2019). Film is a thin, pliable polymer, whereas foam is a soft, light microplastic (Wu et al., 2020). Microplastics with irregular shapes and definite thicknesses are categorized as fragments (D. Huang et al., 2020). Continuous exposure of large plastic debris to erosion, wear, and UV light may produce fragments (Yang et al., 2021). However, Film, foam, and fragments can originate from wrapping or packaging materials, supermarket bags, milk boxes, tires, pavement materials during the mechanical wearing or chemical degradation process (Kutralam-Muniasamy et al., 2020; Ngo et al., 2019; W. Wang et al., 2017). Foam is also derived from the insulating material of buildings (W. Wang et al., 2017)

Interaction with various organisms depends on the shape of microplastics (Kutralam-Muniasamy et al., 2020). The irregular and angular shape of fragments provides a suitable surface for the attachment of microorganisms which accelerates the sedimentation process and increases the removal efficiency of fragments in WWTP(Ngo et al., 2019). However, this phenomenon can severely affect the tissue of microorganisms in the natural environment (Kutralam-Muniasamy et al., 2020).

Fiber is found to be the predominant shape of microplastic in river sediment in most of our reviewed studies (11 out of 27)(Jiang et al., 2019; Liu et al., 2021; Tien et al., 2020). However, in the Nakdong River, South Korea, fragments was detected as the most abundant

shape and contributed to almost 84% of the total microplastics, which trend is similar to some other studies (9 out of 27) (Constant et al., 2020; Eo, 2019; Rodrigues, 2018). In Shanghai, China, the most dominant shape was spheres and accounted for 88.98% of the total number of microplastics observed(Peng et al., 2018). In Pearl river catchment, China, and Brisbane river, Australia, the most common shape of microplastics were sheets and films, respectively (Fan et al., 2019; He et al., 2020).

3.2.2 Size

The probability of being ingested and the pathway of microplastics largely depend on its size (Amrutha & Warrier, 2020; Yang et al., 2021). Due to high specific surface area, biofouling is more likely to occur in small-size microplastics that fasten their deposition in the river bed (Liu et al., 2021; Z. Wang et al., 2018). So, larger microplastics can migrate longer distances compare to smaller ones. In addition, smaller particles are more bioavailable to benthic organisms and can be transmitted to the terrestrial food web (Dioses-Salinas et al., 2020). Different studies have detected microplastics of various size ranges, but small size microplastics were dominant in all studies, which indicates high level of weathering and fragmentation of their initial product (Feng et al., 2020; D. Huang et al., 2020). For example, in the rivers of the Tibet Plateau, 70% of the total microplastics were found to be less than 1mm(Jiang et al., 2019). A similar trend was observed in the middle-lower Yangtze river basin, where microplastics ranges from 0.25-1mm were the most abundant (Su et al., 2018). However, in the Wen-Rui Tang River, microplastics ranges from 20-300 μm were predominant and contributed to 84.6% of the total microplastics (Z. Wang et al., 2018).

3.2.3 Color

Colored microplastics are more likely to be mistaken for food and ingested by organisms(Eo, 2019; He et al., 2020; Wu et al., 2020). Moreover, like shape, color can indicate initial sources of microplastics (Eo, 2019; Yang et al., 2021). For example, Transparent microplastics usually originate from plastic bags, bottles, cups, fishing nets, and other disposable plastic accessories (Di & Wang, 2018; Kutralam-Muniasamy et al., 2020). In contrast, fabric, packaging material, cosmetics, and various colored consumer products can be the potential source of colored microplastics (Di & Wang, 2018; He et al., 2020; Yang et al., 2021). Since the color of microplastics can be bleached out during the sample preparation and even in the natural environment during the photodegradation process, careful approach is required to identify the source of microplastics based on color (Fan et al., 2019; Yang et al., 2021). Furthermore, during sample extraction and purification process, some microplastics can be eroded and result in the underestimation of transparent microplastics (He et al., 2020).

Microplastics are recommended to classify into four colors – Colorless or transparent, black, white, and colored (Yang et al., 2021). However, some studies have also sub-grouped the colored microplastics into yellow, green, blue, red, etc. categories (Jiang et al., 2019; Wen et al., 2018). In most of our reviewed studies, transparent microplastics were found to be predominant (7 out of 27 studies). For example, transparent microplastics contributed to 45.69% of the total number of microplastics in the Tibetan Plateau(Feng et al., 2020). However, white particles were also found to be the most abundant in some studies. For instance, the contribution of white spheres in Shanghai, China were almost 90% (Peng et al., 2018). In contrast, yellow particles were most dominant in the Pearl River and accounted for 36.2% of the entire microplastics (Lin et al., 2018).

3.2.4 Chemical Composition

Chemical composition is one of the most fundamental characteristics of microplastics (Y. Zhang et al., 2020). At present more than 30 types of microplastic polymers have been identified in different studies (Ngo et al., 2019). Among them Polyethylene (PE), Polypropylene (PP), Polystyrene (PS), Poly(ethylene-propylene) Copolymer, Polyethylene Terephthalate (PET), Polyester (PES), Polyvinylchloride (PVC), Vinyl Acetate Copolymer (VC/VAC), Polyamide (PA), Cellulose, etc. are noteworthy. PP, PE, and PET usually originate from packaging material, plastic bags, containers, agricultural films, conduits, cords, automobiles, and domestic accessories, etc. (Kutralam-Muniasamy et al., 2020; Liu et al., 2021; Tien et al., 2020). On the contrary, fabrics, lines and furniture fillers, etc. are the potential source of PA and PES (Tien et al., 2020).

High-density polymers are likely to be deposited in sediment, so concentration is expected to be higher in the sediment than water (Eo, 2019). Despite low density, PE and/or PP were detected as the most abundant polymer type in most of our reviewed studies. For instance, in the sediment of the Haihe River, PE and PP account for 49.3% and 32.9% of the total microplastics, respectively (Liu et al., 2021). Moreover, PP (38%) dominated the types of polymers observed in downstream of West River and followed by PE (27%), PS (16%), PVC (6%), and PET (4%) (D. Huang et al., 2020). Biofouling on the surface of PE and PP that influenced them to sink in the river bed may be the potential cause of this (D. Huang et al., 2020). Other than PP and PE, polymer types varied considerably in different studies. For example, PES (33%) was predominant in the sediment of the Yangtze River basin (Su et al., 2018).

CHAPTER FOUR: MICROPLASTICS ANALYSIS METHODOLOGY

Although several researchers have conducted research on microplastic since two decennaries, standardized methodologies for microplastic analysis have yet to develop (Li et al., 2018; Yang et al., 2021). This paper has reviewed 27 studies to overview sample collection, preparation, and analysis methods of microplastic in river sediment, summarized in Table 2 & Table 3. Overall microplastic analysis methodology can be divided into four steps: Sample collection, sample preparation, sample extraction and purification & identification and quantification.

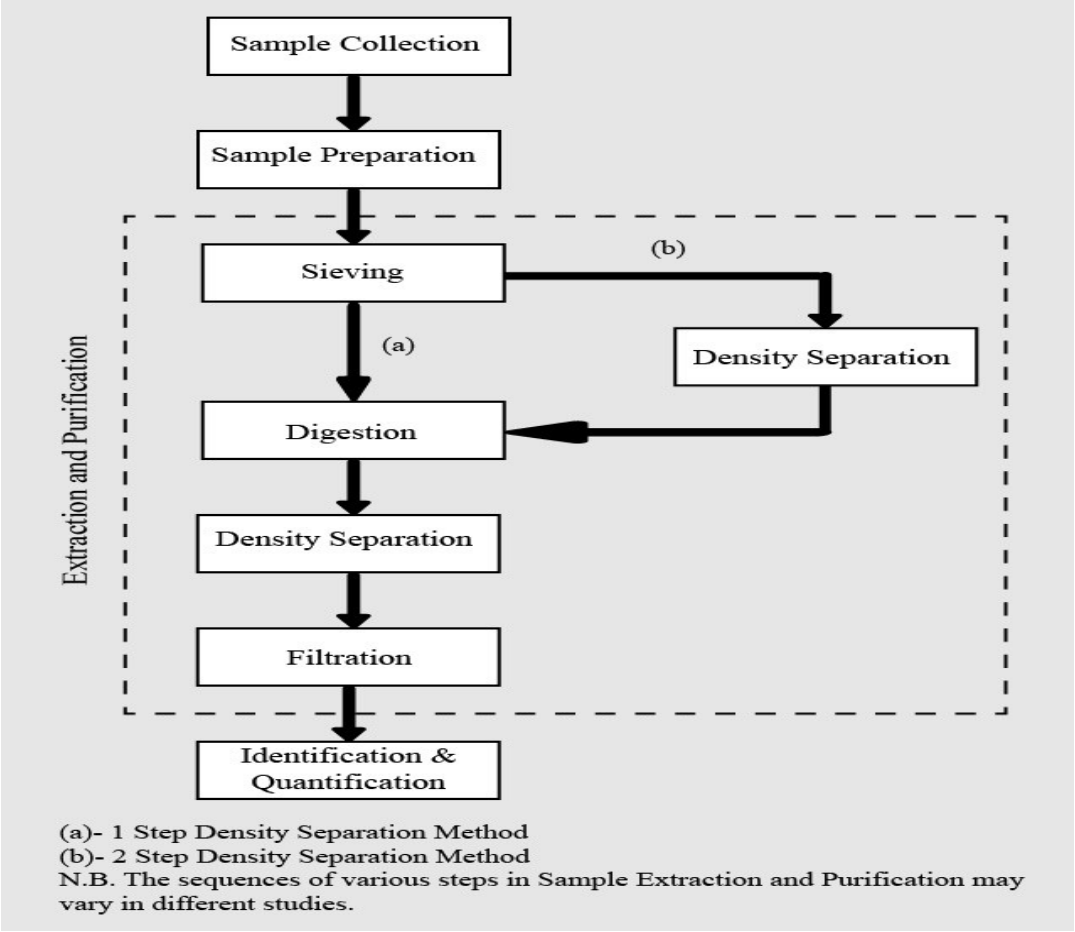

Figure 4: Different steps in microplastics analysis methodology

4.1 Sample Collection

Though sample collection strategy mostly depends on the objectives of the study, it is expected to collect the widest possible number of samples in order to gain an accurate and deep understanding of the distribution and quantity of microplastic particles in sediment (Stock et al., 2019).

The methods of microplastic sampling in freshwater sediment can be categorized into three(Hidalgo-Ruz et al., 2012):

a) Selective Sampling: In this method, microplastics are directly picked from the field samples via visual inspection (Gong & Xie, 2020). This method was not adopted in any of our reviewed studies. However, it can be suitable for samples that contain a copious amount of large microplastic particles (1-5 mm diameter) (Yang et al., 2021).
b) Volume-reduced Sampling: In this method, only the portion of the sample which is necessary for further processing is preserved in order to decrease the volume of the bulk sample(Silva et al., 2018). The sample of interest can be retained by filtering or sieving(Gong & Xie, 2020). This method was adopted in 2 out of 27 river sediment studies reviewed.
c) Bulk Sampling: Entire sample is collected without reducing its size (Silva et al., 2018). Bulk sampling was applied in 25 studies of our reviewed studies.

Shoreline sample offers relatively larger area for sampling in a quick and cost-efficient way, but riverbed sample offers comparatively less disturbed sample as riverbed is less influenced by natural and man-made activity than shoreline(Adomat & Grischek, 2020). Shoreline or riverbed, which should be sampled, largely depends on research perspective, availability of collection tool, and expert availability (Adomat & Grischek, 2020). In most of our reviewed studies, van veen grab sampler (6 out of 27) or stainless-steel shovel (6 out of 27) was used

as sample collection tools. Besides, steel trowel (1 out of 27), Peterson grab sampler (3 out of 27), grab bucket (B-10104) (2 out of 27), box corer (1 out of 27), grasp bucket (1 out of 27), stain-less steel spatula (1 out of 27), stain-less steel spoon (1 out of 27), cole-parmer sediment sampler (1 out of 27), ponar stain-less steel grab sampler (1 out of 27), perspex tubes (1 out of 27), quadrat (1 out of 27) were also used in different studies. Moreover, stain-less steel shovel, spoon, and spatula were used to collect bulk samples from shoreline or riverbank and to collect samples from the middle or center of the river Van Veen grab sampler or Peterson grab sampler were used. Van veen, Petersen, and Ponar grab sampler do not require any winch or crane to operate, unlike box corer sampler. However, box corer offers less variability in penetration depth during sampling of sediment from the bottom of freshwaters and oceans (Brander et al., 2020).

Definition of sampling depth is important for achieving higher accuracy in determining microplastic concentration in sediment samples (Joana Correia Prata et al., 2019). However, Average microplastic concentration can be higher in the top 1-5 cm sample than the top 10 cm sample(Besley et al., 2017). In most of our reviewed studies, sampling depths were defined as the top 5 cm (8 out of 27) or top 10 cm of the sediment (4 of 27). Besides, some of the studies had collected samples from the top 2 cm,15 cm, and even 20 cm of the sediment.

Sampling unit depends on the sample collection tools(European Commission. Joint Research Centre. Institute for Environment and Sustainability. & MSFD Technical Subgroup on Marine Litter., 2013). The laboratory method for microplastics analysis developed by NOAA recommends gravimetric analysis of microplastic in sediment samples (Mausra & Foster, n.d.). However, as the weight of sediment sample is influenced by water content and sediment type, it is suggested by MSDF to use volume as sampling unit instead of weight(European Commission. Joint Research Centre. Institute for Environment and

Sustainability. & MSFD Technical Subgroup on Marine Litter., 2013). Most of the studies (13 out of 27) used weight as the sampling unit, which varies from 200 g to 2000 g. 4 out of 27 studies used area as the sampling unit, which varies from 0.01 m^2 to 0.09 m^2. Eight studies did not specify any sampling unit; one study used area and mass both as their sampling unit.

In order to reduce contamination, the use of plastic equipment should be eschewed during sample collection (Adomat & Grischek, 2020). Hence, Samples are usually stored in aluminum or glass containers. In some cases, samples are stored in polyethylene bags but are folded in aluminum foil. In 8 out of 27 reviewed studies, samples were sealed in glass containers, bottles or jars; nothing was documented in 5 studies and the rest of the studies either used aluminum containers, or bag or wrapped sample in aluminum foil prior to store in a sample box or polyethylene bag. Blank experiment analysis can be initiated to avoid inaccuracy and calculate contamination during sampling and microplastic analysis(D. Huang et al., 2020).

4.2 Sample Preparation

To avoid variability in moisture content of sediment samples, microplastic concentration is suggested to be expressed as dry weight (Van Cauwenberghe et al., 2015). So, residual moisture should be driven off to a constant weight preceding analysis (Yang et al., 2021). Samples can be dried in both oven and air. Interior of oven prevents airborne pollution of sediment sample during the oven drying process(Adomat & Grischek, 2020), but the higher operating temperatures may crack and distort the shape of microplastic(Zobkov & Esiukova, 2017). Nevertheless, in some studies, samples are dried at high temperature by omitting common polymers that are vulnerable to heat distortion(Blair et al., 2019) or eliminating the

effects of heat distortion from consideration (Amrutha & Warrier, 2020; Rodrigues, 2018). In our reviewed studies, drying temperature varies from 40° C to 90°C, and in 4 studies, samples were heated at high temperature of greater than 70°C. In one study, samples were dried in air at room temperature, which may prolong drying time, contaminated the sample with airborne pollutants, and may have residual moisture content even after completion of the drying process (Adomat & Grischek, 2020).

4.3 Extraction & Purification

4.3.1 Sieving

Sieving is a primary extraction process where sediment samples are passed through sieves of various openings to trap microplastic particles and to separate impurities like clay and silt-sized particles from sediment samples (Gong & Xie, 2020). It can be classified into two- dry sieving & wet sieving. Due to electrostatic charges in the surface, fine particles agglomerate together and may retain on sieve during dry sieving. So, dry sieving is not adequate for particles finer than 40 μm. Wet sieving can be efficient for particles up to 20μm but may discard low-density microplastic particles unconsciously (Adomat & Grischek, 2020). Sieving step can be omitted during microplastic analysis in order to include fine-sized microplastic fractions in the study(Z. Wang et al., 2018) or if there is no visible debris in the sample (Di & Wang, 2018).

4.3.2 Digestion

Sample purification or digestion is a pretreatment process for the removal of organic matter from sediment samples in order to avoid disruption of accurate extraction and categorization of microplastics (Adomat & Grischek, 2020; Gong & Xie, 2020). Acid, alkaline, enzyme, 10-30% H_2O_2solutions, Fenton's reagent can be used to treat biological samples.

35%H_2O_2 solution is proved efficient by some studies to remove organic matter from sediment sample(Nuelle et al., 2014), and standardization of digestion method is moderately being developed by usingH_2O_2solution at controlled temperature in a specific digestion period (Y. Zhang et al., 2020). H_2O_2 digestion may result in discoloration and size reduction of polymer particles > 1 mm in size (Nuelle et al., 2014). On the contrary, (Hurley et al., 2018) have found no visible changes for most of the polymer types during H_2O_2oxidation. 12 out of 27 reviewed studies usedH_2O_2 solution for digestion of organic matter from sediment sample.

A mixture of H_2O_2 and Ferrous Sulfate ($FeSO_4.7H_2O$) catalyst, namely Fenton's reagent, can be an alternative to H_2O_2 digestion (Adomat & Grischek, 2020). The reaction rate of Fenton's reagent is more rapid and can more efficiently degrade organic matter that is laborious to remove in traditional H_2O_2digestion (Hurley et al., 2018). Fenton's reaction was applied in 5 out of 27 reviewed studies.

Digestion with acid and alkali of low concentration exhibit low removal efficiency and excess organic compounds may contain residue in the sediment sample at the end of digestion (Nuelle et al., 2014). Moreover, Variability in chemical resistance of different types of microplastic may limit the application of strong acid and alkali digestion (Gong & Xie, 2020). For example, the use of sulfuric acid, nitric acid, or sodium hydroxide may cause the degradation and melting of microplastic particles (Hurley et al., 2018). Potassium hydroxide was found to be used for digestion only in one study out of 27 reviewed studies.

Enzyme digestion is another alternative for the extraction of microplastic from organic-rich samples. More than 97% removal efficiency can be achieved in enzyme digestion using proteinase-K without damaging the morphology of microplastic (Cole et al., 2015). The

applicability of enzyme digestion may be limited to small samples only due to high expense of enzyme (Hurley et al., 2018).

Sand and loosely attached impurities can be extracted from the surface of sediment sample by an ultrasonic bath (Jundong Wang et al., 2017). Though impact identification on microplastic due to ultrasonic cleaning is still in headway, it may lead to aging and degradation of microplastic (Adomat & Grischek, 2020). Integration of deionized water or sodium dodecyl sulfate solution with ultrasonication is another possible method for sample purification but may cause disaggregation of fragile microplastic particles (J. Wang et al., 2017). Only 2 out of 27 reviewed studies used ultrasonication in order to improve the extraction process (Jiang et al., 2019; Jundong Wang et al., 2017).

4.3.3 Density Separation

As the collected sediment samples are mixed with impurities like inorganic clay, separation or extraction of the sample must be performed before analysis(Gong & Xie, 2020). All the studies we reviewed used density separation for microplastic extraction. Using the dissimilarity of density between microplastic and non-microplastic particles is the principle of the density separation method (Gong & Xie, 2020). In this method, saturated or highly dense salt is thoroughly mixed with the sediment sample, which allows the low-density particles like microplastics to float and high-density particles like impurities to settle down (Li et al., 2018). Consequently, microplastics are extracted from the top layer of the solution.

Saturated NaCl solution is a non-lethal, non-abrasive, and economical material, which makes it the most widely used solution for density separation (Yang et al., 2021). The major drawback of this solution is the low extraction efficiency of heavy microplastics like polyethylene (PET) and polyvinyl chloride (PVC) due to its comparatively low density (1.2 $g.cm^{-3}$)(Amrutha & Warrier, 2020). Zinc chloride solution ($ZnCl_2$) (density:

$1.8 g.cm^{-3}$) eliminates the limitation of NaCl solution and allows floatation of all types of polymer (Tien et al., 2020). $ZnCl_2$is a perilous solution and recycling and reusing of this solution should be ensured to circumvent environmental degradation(Li et al., 2018).NaCl was used in 11 out of 27 reviewed studies, and $ZnCl_2$ was also found to be used in 10 studies.

Sodium iodide (NaI) (density: 1.6-1.8 $g.cm^{-3}$) can also separate high-density polymers with better efficiency (Gong & Xie, 2020). Since NaI is costly and hazardous product, primary extraction can be performed with NaCl solution to decrease sample mass prior to secondary extraction with NaI solution (Di & Wang, 2018).

Potassium formate solution (KF) (density: $1.58 g.cm^{-3}$) is relatively less hazardous and non-poisonous to the environment and can be used to extract low-density microplastic as well (Yang et al., 2021). Despite these advantages, only 1 out of 27 studies used KF solution for density separation due to its high expense.

With the exception of density separation, elutriation, and pressurized fluid extraction (PFE) techniques can also be used for microplastic extraction (Li et al., 2018). Microplastic particles of any size can be separated using pressurized fluid extraction method but at optimum PFE conditions microplastic particles can be degraded (Fuller & Gautam, 2016). On the other hand, elutriation can recover microplastics with an efficiency of 93-98% but less effective in extracting microplastic from organic-rich sample (Li et al., 2018).

4.4 Identification & Quantification

Identification is the most important part of microplastic analysis and can be performed by visual and/or spectroscopic inspection. The purpose of visual investigation is to sort out presumed microplastics for further identification based on physical attributes like shape, size,

and color (Y. Zhang et al., 2020). Sorting can be done by the naked eye or in assistance with a microscope (Hidalgo-Ruz et al., 2012).Various types of microscope such as stereoscopic microscope (Jiang et al., 2019; Simon-Sánchez et al., 2019), scanning electron microscope (Wen et al., 2018), metallographic microscope (Ding et al., 2019), fluorescence microscope (Wu et al., 2020), light microscope (He et al., 2020), etc. are used during visual inspection. Among them stereoscopic microscope is considered to be the most used microscope (Yang et al., 2021).

4.4.1 Visual Inspection

Visual sorting may depend on the examiner's perspective, quality of microscope, and condition of sediment sample (Li et al., 2018). So, this type of inspection is open to bias and may result in the misidentification of microplastics (Yang et al., 2021). The accuracy of visual inspection decreases with the decrease of microplastic particle size (Gong & Xie, 2020), and the rate of error can be as higher as 70%(Hidalgo-Ruz et al., 2012).

Selection of plastic particles during visual identification should be based on the following criterion in order to eschew error in visual inspection: free from organic impurities, consistency in thickness, and homogeneity in color across its length (Hidalgo-Ruz et al., 2012). Nevertheless, sometimes irregularity in the edge of colored fiber, bleaching, biological contamination, and design of plastics are taken into deliberation (Simon-Sánchez et al., 2019). It is preferable to examine the microplastics on the filter surface without transferring to any other container to avoid loss (Yang et al., 2021), and the investigation should be performed from the top left to the bottom right in order to avoid duplicate counting (Simon-Sánchez et al., 2019).

4.4.2 Spectrometric analysis

Spectrometric analysis is performed in order to investigate the chemical composition of microplastics (Gong & Xie, 2020). Besides, It can be used to assess visual sorting and correct the particle count determined from visual inspection (Constant et al., 2020). The principle of spectrometric analysis is to detect vibration from agitated samples and compare the produced spectra to the known reference spectra (Li et al., 2018). Fourier transform infrared (FTIR) and Raman spectroscope is the most widely used spectroscope for the analysis of microplastic (Hidalgo-Ruz et al., 2012).

FTIR can function in three modes- Reflection, transmission, and attenuated total reflectance(Yang et al., 2021). FTIR is widely operated by attenuated total reflection (ATR), in which particles are individually identified and detected by microscopy and ATR tip, respectively (Lee & Chae, 2021). Particles smaller than 500 μm may not be detected by ATR(Gong & Xie, 2020). To analyze smaller particles, micro-FTIR is developed by attaching a microscope with FTIR (Ivleva et al., 2017). In transmission mode, translucent samples are preferable than dark or colored particles. Analysis in reflection mode is not dependent on the optical properties of particles, so thick non-transparent particles can be detected in this mode(Gong & Xie, 2020). By using the precision linear mechanism that provides motion in 3 degrees of freedom, individual particles can be analyzed within a specific grid area in focal plane array (FPA) detector mode (Lee & Chae, 2021). FPA detector developed chemical imaging and enables handling of larger data sets (Ivleva et al., 2017). The major advantages of FTIR spectrometric analysis are quick and effective identification of microplastic without the influence of fluorescence and detection of the degree of weathering (Gong & Xie, 2020). The major limitation of FTIR spectrometric analysis is its sensitivity towards moisture(Yang et al., 2021). 19 out of 27 reviewed studies used FTIR for spectroscopic analysis.

Unlike FTIR spectrometry, wet samples can be analyzed in Raman spectroscopy (RM)(Ivleva et al., 2017).RM offers high spatial resolution so particles less than 20 μm can be detected (Gong & Xie, 2020). RM spectrometry is not fit for the samples that are sensitive towards fluorescence action. So, samples need to be free from additives, pigment, cellular, organic, and inorganic impurities (Gong & Xie, 2020; Yang et al., 2021). However, chemical mapping using RM spectrometry is a comparatively time-consuming process (Ivleva et al., 2017). 6 out of 27 reviewed studies used RM for spectroscopic identification.

4.4.3 Miscellaneous

Pyrolysis-GC/MS is another alternative for microplastic analysis where chemical composition of microplastic is determined by analyzing thermally decomposed products (Nuelle et al., 2014). Pyr-GC/MS analysis can only provide information about the type and mass fraction of chemical components (Dümichen et al., 2015). As it causes thermal degradation of sediment sample, amount, shape & size of the sample remains undetermined (Y. Zhang et al., 2020). Sample preparation step can be omitted in this type of analysis (Dümichen et al., 2015; Yang et al., 2021), but samples with a high amount of impurities and samples with smaller particles (<500μm) do not have applicability to Pyr-GC/MS analysis (Yang et al., 2021). Some polymers may exhibit identical degradation outcomes which may cause misidentification of microplastics(Gong & Xie, 2020).

Microplastic surface characteristics and morphology can be investigated by Scanning electron microscope (SEM)(Wu et al., 2020). For field emission SEM analysis, microplastics identified from the spectrometric analysis are wrapped with a thin gold or platinum film and then mapped using SEM. To increase accuracy and avoid the effect of corroded samples, visual inspection should be repeated at least thrice (Jundong Wang et al., 2017). In 5 out of 27 studies SEM analysis was performed.

CHAPTER FIVE: CONCLUSION & RECOMMENDATIONS

Since the production of plastics has an increasing trend, research on microplastics should be more systematic, standardized, and consistent. Firstly, there is no alternative to source identification to understand the temporal and spatial variation of microplastics. Most of the studies we reviewed had indicated some possible sources but not in detail. So, researchers should give more focus on sources of microplastics and conduct a detailed reconnaissance survey of the surrounding area prior to microplastics analysis that will include information on point sources, non-point sources, distance between possible sources and sampling points, extent and level of any anthropogenic activities such as fisheries, navigation, etc., if possible. Otherwise, Like (Feng et al., 2020), they can include justification of their research outcome and quantitative discussion on the contribution of different sources to the microplastic accumulation in different sampling sites. Moreover, Population density, flow rate, and climatic conditions have large influence on microplastic concentration in river sediment. So, this information should also be documented in the reconnaissance survey. Secondly, Indian subcontinent is one of the most densely populated areas in the world, with many textile industries (Ali & Habib, 2012; Raichurkar & Ramachandran, 2015). Hence, Rivers in this zone are the potential hotspot of microplastic pollution. But very few studies on microplastics in river sediment have been performed in this area compared to other parts of the world. Thirdly, from the summary tables, we have provided in this study, extensive variation in sample collection and laboratory analysis technique is clearly observed. Even no sign of consensus is noticed in the classification of microplastics. Hence, standardization and harmonization of analysis methodology and uniform criteria for classification is required to enable relative discussion between different studies. Since research on microplastics is still in infancy, standardization is yet challenging to achieve(Stock et al., 2019). For example, European Commission and National Oceanic and Atmospheric Administration, USA have

already developed separate guidelines for the analysis of microplastics in the laboratory (European Commission. Joint Research Centre. Institute for Environment and Sustainability. & MSFD Technical Subgroup on Marine Litter., 2013; Mausra & Foster, n.d.) but till now very few studies have followed them. In this case, comparative research on performance evaluation of different methodologies should be conducted to assess the dissimilation in the result. Last of all, Hazardous chemicals used in the analysis of microplastics must be recycled. It will not only protect the environment from degradation but also reduce the expense of the research.

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APPENDIX

Table 1: An overview of abundance and characteristics of MPs

No.	Study area	Abundance	Size	Shape	Chemical Composition	Color	Ref
1	Nakdong River, South Korea	mean: 1971 particles/kg dry weight	< 300 µm -81%	Fragments -84%, Fibers -15% and Spheres (1%).	PP- 24.8%, PE- 24.8%, PES- 5.5%, PVC - 5.4%,PS 5.3%, Acrylic - 4.6%, Polydimethylsiloxane- 4.5%, PU- 3.9%, Poly(acrylate-styrene)- 3.7%, Poly(lauryl acrylate) -3.6% and others <3%		(Eo, 2019)
2	Rivers of the Tibet Plateau	range: 50±7 item/kg to 195±64items/kg	<1mm - 70%	Fiber -53.8% to 80.6% and Pellets or Fragments- rest	Polyethylene terephthalate (PET) - most abundant.	Transparent- 30% to 50%, Black- 18% to 30%, White- 4% to 10%, Red- 6% to 18%, Blue- 3% to 32% and Green- 0 to 8%	(Jiang et al., 2019)
3	Têt river, perpigan, france	mean: 258 ± 259 item/ kg		Fragments -54.8%, Fibers -19.5%, Foams -13.0%, Films -7.0% and Beads -5.7%	Fibers: Non-plastic - 40%; Fragments: PE- 45%, PP- 23% ; Films: PE- 29%, PP- 35%; Foams: PS- 50% and Beads: PE- 100%		(Constant et al., 2020)

Table 1: An overview of abundance and characteristics of MPs (Contd.)

No.	Study area	Abundance	Size	Shape	Chemical Composition	Color	Ref
4	Ebro River, spain	range: 1491 ± 272 particles/kg dry weight to 2899±718 particles/kg dry weight	<50 µm: 2.5%, 50-100µm: 4%, 100-200µm: 11.5%, 200-500µm: 30%, 500-1000µm: 18%, 1000-2000µm: 18%, 2000-3000µm: 9% and >3000µm: 7%	Fiber>Fragment>Film>Foam	Polyamide -24%, Polyethylene- 16%, Poly(methyl methacrylate)(acrylic)-12%, Polyester- 12%, Polypropylene- 8% and Polyacrylate- 4%	Colour- 58%, Transparent-20%, Black- 10% and White- 2%	(Simon-Sánchez et al., 2019)
5	Antuã River, Portugal	range: 18 to 629 items/kg dry weight		Fragments- 43.6% (most) and Pellets-1.2% (lowest)	PE- 29.4%, PP- 29.4%, PS- 8.8%, PET- 8.8%, Others- 29.4%	Colour(blue,green) >White>Black>Transparent	(Rodrigues, 2018)
6	Middle-Lower Yangtze River Basin	range: 15 to160 items/kg	0.25-1 mm - Most abundant	Fiber>Fragment>Film>Pellet	Polyester- 33%, Polypropylene- 19% and Polyethylene- 9%	Transparent and blue items- Most abundant	(Su et al., 2018)
7	Xiangjiang River	range: 270.17 ± 48.23 items/kg to 866.59 ± 37.96 items/kg	<0.5mm: 21% to 52%, 0.5-1mm: 12% to 29%, 2-3mm: 5% to 12%, 3-4mm: 3% to 12% and 4-5mm: 2 to 40%	Fragment- 50.82%, Fiber-28.15%,Film-18.14% and Foam<10%	PET- 14.71%, PP-13.24%, PE- 19.12%, PA-10.29%, PS-19.41%, PVC- 7.35%, Non-plastic- 5.88%	Transparent- 16% to 50%, White- 4 to 40%, Red- 2 to 32%, Blue- 4% to 23%, Green- 4% to 23% and Yellow-0 to 8%	(Wen et al., 2018)

Table 1: An overview of abundance and characteristics of MPs (Contd.)

No.	Study area	Abundance	Size	Shape	Chemical Composition	Color	Ref
8	Wei River,china	range: 360 to 1320 items/kg	< 0.5 mm: 40.8% to 68.8%, 0.5-1mm: 8.35% to 24.2%	Fiber- 42.25% to 53.20%, Film- 23.9% to 31.8%, Fragment- 10.2% to 20.3%, Pellet 5.6%-16.1%, Foam- 0.7% to 3.5%			(Ding et al., 2019)
9	Amazon rivers, Brazil	range: 417 to 8178 particles/kg of dry weight (particles 0. 063–5mm) and 0 to 5725 particles/kg of dry weight(particles 0.063–1 mm)	0-1mm: 3 to 70%, 1-2mm: 23 to 72%, 2-3mm: 5 to 28%, 3-4mm: 0 to 7%, 4-5mm: 0 to 11%				(Gerolin, 2020)
10	Maozhou River, china	range: 35 ± 15 to 560 ± 70 item/kg sediments in April; 25 ± 5 to 360 ± 90 item/kg sediment in October	0.1-1mm: 47.5% to 72.9%	Fragment- 89.4%, Foam- 6.7%, Fiber-2.3%, Film- 1.6%	PE- 45%, PS-34.5% and PP- 12.5%	Transparent-38%, White- 28%	(Wu et al., 2020)

Table 1: An overview of abundance and characteristics of MPs (Contd.)

No.	Study area	Abundance	Size	Shape	Chemical Composition	Color	Ref
11	Fengshan River	range: 508 to 3,987 items/kg	Small size: 67% to 96%	Fiber - 61% to 93%	Epoxy resin- 17%, Phenolic resin - 13%, PET- 17%, PE- 8%, PVOH-8%, PI- 7%, PS- 6% and PTFE- 6%		(Tien et al., 2020)
12	Brisbane River sediments, Australia	range: 10 to 520 items/kg	PE: <1mm: 22%, 1-2mm: 20%, 2-3mm: 21%, 3-4mm:19% and 4-5mm:18%; PA: 2-3mm: 4% and 3-4mm:96%;PP: <1mm: 12%, 1-2mm: 23%, 2-3mm: 20%, 3-4mm:13% and 4-5mm:32%; Others: <1mm: 28%, 1-2mm: 50% and 2-3mm: 22%	Film>Fragment>Fiber	PE-70%, PA- 12% and PP- 10%	White - Most abundant	(He et al., 2020)
13	Tibetan Plateau	range: 20−160 items/kg ;mean: 60.8±25.06 items/kg	20-50 µm- 25.83%,50-100 µm: 31.79%, 500 -1000µm 11.26% and >1000 µm -4.65%	Fiber- 42.38%, Fragment- 25.16%, Film- 11.92%, Sphere- 11.26% and Foam 9.27%	PP-32.45%, PE 28.48%, PS 15.23% and PET 13.24%	Transparent- 45.69%, white- 18.54% , black and blue- 15.23% and others - 20.53%	(Feng et al., 2020)

Table 1: An overview of abundance and characteristics of MPs (Contd.)

No.	Study area	Abundance	Size	Shape	Chemical Composition	Color	Ref
14	Wen-Rui Tang River, southeast china	mean: 32947±15342 items/kg	20-300 µm :84.6%, 300-5000 µm: 15.4%	Fragment- 45.9%, Foamp- 29.5%, Pellets- 12.8% and Fibers-11.7%	PE,PP,PES,PS- Most abundant		(Z. Wang et al., 2018)
15	Haihe River	range: 1346 to 11917 items/kg dry weight (dw) average: 4980± 2462 items/kg dw	500-1000 µm: 26.5±12.8% (range: 3.7-50.9%), 200-500 µm:24.7±14.3% (range: 1.9-71.5%) and 1000-2000 µm:23.7±12.7% (range: 1.4-70.4%)	Fibers- 70.9% Fragments- 15.8%, Lines- 5.7%, Films- 4.2% Pellets- 3.3%	PE- 49.3% (LDPE- 90.7% and HDPE- 9.3%), PP- 32.9%, poly(ethylene-propylene) copolymer- 6.4%, PS- 5.9% and cellulose- 5.5%	Black- 47.1%, Green- 22.3%, Red- 17%, Transparent- 7.4%, White- 6.2%	(Liu et al., 2021)
16	Shanghai, China	mean: 802 ± 594 items kg-1 dry weight	<100mm: 31.19%, 100-500mm: 62.15%, 500-1000mm:3.56%, 1000-5000mm: 2.8% and > 5000mm- 0.3%	Spheres- 88.98%, Fiber- 7.55% and Fragments- 3.47%		White spheres - 90%, Blue- 3%, Transparent- 3%, White- 2% and Red 2%	(Peng et al., 2018)
17	WestRiver downstream ,china	range: 2560 to 10240 items/kg	<0.5mm 87-92%, 0.5-1.0mm 6-9% and 1-5mm -2-4%	Fiber 48%-76% Fragment 4%-17% Film 12%-23% Pellet 8-12%	PP - 38%, PE- 27% PS- 16%, PVC- 6% PET 4% Non-microplastics -9%		(D. Huang et al., 2020)
18	Bloukrans River system	mean: 160.1±139.5 items/kg					(Nel et al., 2018)

Table 1: An overview of abundance and characteristics of MPs (Contd.)

No.	Study area	Abundance	Size	Shape	Chemical Composition	Color	Ref
19	Vistula River (Poland)	range: 190 items/kg to 580 items/kg	0.3 – 0.75 mm: Most abundant	fiber- 93% (Most abundant)	PS,PP,PE,Nylon- Most abundant	Black- 24% to 68%, Blue- 5 to 22%, Transparent- 6 to 11%, Red- 0 to 13%, Grey- 0 to 21%, Yellow- 0 to 4%, Pink- 0 to 41%, Green- 0 to 4%	(Sekudewicz et al., 2020)
20	Beijiang River	range: 178±69 items/kg to 544±107 items/kg			PE- 41.7±18.9% to 65.5±11.0%, PP- 17.2±2.6% to 33.3±6.6%, Copolymer- 5.6±0.8% to 18.8±4.3% and others- 5.3±0.8% to 10.3±8.1%		(Jundong Wang et al., 2017)
21	Pearl River along Guangzhou City, China	range: 80 to 9597 items/kg, mean: 1669 items/kg	0.02–1 mm: 65.3%, 1–2 mm: 29.5%, 2–3 mm: 7.6%, 3–4 mm: 3.3% and 4–5 mm:1.6%	Fiber- 54.7%, Fragment- 43.3% and Film- 43.3%	PE- 47.6% and PP- 26.2%	Yellow- 36.2%, White- 26.8% and Black- 11.7%	(Lin et al., 2018)
22	Pearl River catchment, China	mean: 685 ± 342 items/kg dry weight	<0.1mm- 45.0 ± 4.3%, >1mm- 64.5 ± 7.0%	Sheets- 70.0±4.2% (Most abundant)	PP- 2% to 39%, PE- 3% to 40% and PP-PE copolymers- 0% to 6%	White/transparent- 51 ± 7% (Most abundant)	(Fan et al., 2019)

Table 1: An overview of abundance and characteristics of MPs (Contd.)

No.	Study area	Abundance	Size	Shape	Chemical Composition	Color	Ref
23	Netravathi River,India	average: 96 items/kg of dry weight	1-5 mm: 34.6% and 1-0.3 mm: 65.4%	Fragments- 44.4 %, Fibres- 34.6 %, Films- 8.7 % and Foams & Pellets- < 1%	PE- 56.99 %, PET- 23.43 % and PP- 4.20 %	White- 32.2 %, Transparent- 29.0 % and others (black, red, blue, brown, green and yellow)- < 10 %	(Amrutha & Warrier, 2020)
24	Qinhuai River	range: 163-563 items kg-1 wet sediments	In 1st Layer: 4-5mm: 40.5%, In 3rd Layer: -2-4mm: 41.9%, In 5th Layer: < 2mm: 63.5%	Fragment- 51.3%, Fiber 45.5%	PE-48%, PP- 32%, PMMA- 11%, PU- 5%	Transparent - 35.9%, Green- 20.5%	(Niu et al., 2021)
25	Bloukrans River, South Africa	Summer: 6.3 ± 4.3 particles kg−1 (mean), Winter:160.1 ± 139.5 particles kg−1 (mean)	-	-	-		(Nel et al., 2018)
26	Tributaries of the River Thames	range: 18.5 ± 4.2 to 66 ± 7.7 particles per 100g	1-2mm: 10.2 ± 3.1 to 41.9 ± 3.4 particles per 100g, 2-4mm: 8.1 ± 5.3 to 24.1 ± 5 particles per 100g	Fragment- 49.3%, Fiber- 47.4% & Film- 3.3%	PET - 14 particles, PP- 5 particles, PAS- 5 particles, PE- 2 particles, PS- 1 particle, PVC- 1 particle, Others- 6 particles		(Horton et al., 2017)
27	Qin River	range: 0 to 97 items·kg−1 dry weight	1-5 mm: 76.0% & 0.03-1 mm: 24.0%	Fibre- 30.9%, Sheet- 62.8% & Fragment- 6.3%	PP- 55.3%, PET- 21.3%, PE- 17.0%	Black- 1.5%, White- 30.0%, Blue- 27.6%, Green- 18.3%, Red- 18.5%, Yellow- 3.5% & Others- 0.6%	(L. Zhang et al., 2020)

Table 2: An overview of sample collection techniques of microplastics in the river sediment

No.	Sediment Type	Collection Tool	Sampling Method	Depth	Area	Mass/Volume	Ref
1	Riverbed	Van Veen grab	Bulk	2 cm	-	945 gm	(Eo, 2019)
2	Between the shoreline and water edge	stainless steel shovel.	Bulk	2 cm	0.04 m2	200 gm	(Jiang et al., 2019)
3	River shore	steel trowel	Volume-reduced	1 cm	-	-	(Constant et al., 2020)
4	Riverbed	ven veen grab	Bulk	10 cm	-	-	(Simon-Sánchez et al., 2019)
5	Shoreline	Van Veen grab	Bulk	12 cm	0.051 m2	-	(Rodrigues, 2018)
6	Riverbed	Peterson sampler	Bulk	10 cm	-	2000 gm	(Su et al., 2018)
7	Shoreline	shovel	Bulk	5 cm	-	1000g	(Wen et al., 2018)
8	Riverbed	grab (B-10104, Ravene)	Bulk	-	-	-	(Ding et al., 2019)
9	Riverbed	van Veen sampler	Bulk	5–10 cm	-	200 gm	(Gerolin, 2020)
10	Riverbed	Box corer	Bulk	20 cm	-	1000 gm	(Wu et al., 2020)
11	Riverbank	Cole–Parmer sediment sampler	Bulk	15 cm	-	500 gm	(Tien et al., 2020)
12	Riverbed	Ponar stainless-steel grab sampler	Bulk	0-3 cm	-	-	(He et al., 2020)
13	Shoreline	stainless steel spatula	Bulk	0-5cm	-	-	(Feng et al., 2020)

Table 2: An overview of sample collection techniques of microplastics in the river sediment (Contd.)

No.	Sediment Type	Collection Tool	Sampling Method	Depth	Area	Mass/Volume	Ref
14	Riverbed	Peterson grab sampler	Volume-reduced	0-15 cm	-	500 gm	(Z. Wang et al., 2018)
15	Midpoint,shore and riverbank	Peterson grab sampler	Bulk	5cm	305mm*305mm	-	(Liu et al., 2021)
16	Riverbank	shovel	Bulk	-	-	500 gm	(Peng et al., 2018)
17	Riverbed	grab bucket (B-10104, Ravenep	Bulk	0-10 cm	-	5000 gm	(D. Huang et al., 2020)
18	Riverbed		Bulk	5cm	-	2000 gm	(Nel et al., 2018)
19	Riverbank	stainless-steel shovel	Bulk	4-5cm	100 cm2	-	(Sekudewicz et al., 2020)
20	Shoreline	stainless-steel shovel	Bulk	2cm	20cm*20 cm	-	(Jundong Wang et al., 2017)
21	Riverbed	Van Veen grab sampler	Bulk	5cm	-	2kg	(Lin et al., 2018)
22	Riverbed	grasp bucket	Bulk	-	-	-	(Fan et al., 2019)
23	Shoreline	stainless-steel spoon	Bulk	0-5cm	-	1-2kg	(Amrutha & Warrier, 2020)
24	Riverbed	Perspex tubes	Bulk	50 cm			(Niu et al., 2021)
25	Riverbed	Quadrat	Bulk	5 cm		2 kg	(Nel et al., 2018)
26	Shoreline	Stainless steel scoop	Bulk				(Horton et al., 2017)
27	Middle and both sides of the bow	Grab dredge & stainless steel shovel	Bulk	5 cm			(L. Zhang et al., 2020)

Table 3:An overview of Sample Analysis techniques of microplastics in the river sediment

No.	Sampling Device	Container	Drying Temp. (oC)	Size Selection	Digestion	Density Selection	Filtration	Visual Inspection	Identification	Ref.
1	Van Veen grab	Amber glass bottle	60	20 µm metal sieve	Fenton's reagent	Lithium metangstate	Polycarbonate filter paper (5 mm, 47 mm Ø)		FT-IR	(Eo, 2019)
2	Stainless steel shovel.	Aluminium foil and sample box	70	2 mm stainless sieve	Wet peroxide oxide	Zinc Chloride	0.22-mm pore size GF/C filter	Stereoscopic microscope	Raman spectroscope	(Jiang et al., 2019)
3	Steel trowel	Glass bottle		A column of sieves (5, 2.5, 1 mm; 500, 315, 63 µm)		A hypersaline solution	Whatman® filter papers	Dissecting stereo-microscope	FT-IR	(Constant et al., 2020)
4	Ven Veen grab	Glass container		2mm, 63 µm sieve	Wet Peroxide Oxide	Saturated NaCl solution	Glass-fiber filter (GF/F; 47 mm ø, 0.7 µm pore size)	Stereomicroscope	µFT-IR	(Simon-Sánchez et al., 2019)
5	Van Veen grab	Aluminium foil and sediment box	90	0.055 mm sieve	Fenton's reagent	Zinc Chloride	0.45 µm clean membrane filter	Stereo microscope Optika	ATR–FTIR	(Rodrigues, 2018)

Table 3: An overview of Sample Analysis techniques of microplastics in the river sediment (Contd.)

No.	Sampling Device	Container	Drying Temp. (oC)	Size Selection	Digestion	Density Selection	Filtration	Visual Inspection	Identification	Ref.
6	Peterson sampler	Glass bottle, aluminum pot and aluminum foil bag	65	20 micrometer nylon filter	Hydrogen peroxide	Saturated sodium chloride solution	20 micrometer nylon filter	Microscope	µFT-IR	(Su et al., 2018)
7	Shovel	Aluminum foil and bag	65		Fenton's reagent	Zinc chloride granules	Vacuum-filtered onto a GF/C filter	Scanning electron microscope	Micro-Raman spectroscopy	(Wen et al., 2018)
8	Grab (B-10104, Ravene)		70		30% Hydrogen peroxide	NaCl solution	0.45 µm filter paper	Metallographic microscope	SEM	(Ding et al., 2019)
9	Van Veen sampler		50	63-µm stainless steel mesh	30% Hydrogen peroxide	ZnCl2 solution	Filter-paper (pore: 18 µm)	Motorized stereomicroscope	Software AxioVision	(Gerolin, 2020)
10	Box corer	Aluminum foil bags			Wet Hydrogen peroxide	ZnCl2 solution	0.45 µm GF/C glass microfiber filter membranes	Fluorescence microscopy	FT-IR, µ-FT-IR & SEM	(Wu et al., 2020)

Table 3: An overview of Sample Analysis techniques of microplastics in the river sediment (Contd.)

No.	Sampling Device	Container	Drying Temp. (oC)	Size Selection	Digestion	Density Selection	Filtration	Visual Inspection	Identification	Ref.
11	Cole–Parmer sediment sampler	Glass bottles	50	mesh sieves (50–297 µm and 297–5000 µm)	35% Hydrogen peroxide	Zinc chloride solution	Filter membranes (47 mm diameter and 0.8 µm pore size)	Dissecting microscope	FT-IR	(Tien et al., 2020)
12	Ponar stainless-steel grab sampler	Glass jars				Zinc chloride (ZnCl2)	Acuum filtration (0.45 µm membrane filter)	Light microscope	FT-IR	(He et al., 2020)
13	stainless steel spatula	Aluminum foil bag	70	2 mm stainless sieve	30% Hydrogen peroxide	Saturated NaCl	GF/C filters (0.45µm pore size, 47mm diameter)	Stereoscopic microscope	Raman spectroscope	(Feng et al., 2020)
14	Peterson grab sampler		60	5 mm stainless steel mesh	30% Hydrogen peroxide	Zncl2 solution	4 micrometer polycarbonate membrane filter	Fluorescence stereo microscope	µFT-IR	(Z. Wang et al., 2018)
15	Peterson grab sampler	Aluminum boxes	air-drying		30% Hydrogen peroxide	Saturated NaCl solution	Whatman GF/C glass fiber filter (pore size = 1.2 micrometer)	Stereo microscope	µ-FT-IR	(Liu et al., 2021)

Table 3: An overview of Sample Analysis techniques of microplastics in the river sediment (Contd.)

No.	Sampling Device	Container	Drying Temp. (oC)	Size Selection	Digestion	Density Selection	Filtration	Visual Inspection	Identification	Ref.
16	Shovel	Tin cup or aluminum foil	70			Saline solution of NaCl	Filter paper (Whatman GF/B, 4 ¼ 1 mm)	Stereo microscope	m-FTIR	(Peng et al., 2018)
17	Grab bucket (B-10104, Ravenep		70		30% Hydrogen peroxide	Saturated salt solution	0.45 µm filter paper	Metallographic microscope	AT-FTIR	(D. Huang et al., 2020)
18			50	2 mm mesh steel sieve		Hyper-saturated saline solution	63 µm mesh	Olympus dissecting microscope		(Nel et al., 2018)
19	Stainless-steel shovel	Glass container	40			NaCl solution	Metal sieves (5, 0.75 and 0.30 mm) & filtered	Stereo microscope	Raman/FT-IR & SEM	(Sekudewicz et al., 2020)
20	Stainless-steel shovel	Aluminium foil bag	50			Saturated NaCl	Glass microfiber filter (Whatman GF/B, diameter 47mm, pore size 1µm)	Digital handheld microscope	µ-FTIR, SEM & ICP-MS	(Jundong Wang et al., 2017)
21	Van Veen grab sampler	Aluminium foil bag	60		10% KOH	Saturated NaCl solution	20 µm membrane filter	Stereo light microscope	µ-FTIR	(Lin et al., 2018)

Table 3: An overview of Sample Analysis techniques of microplastics in the river sediment (Contd.)

No.	Sampling Device	Container	Drying Temp. (oC)	Size Selection	Digestion	Density Selection	Filtration	Visual Inspection	Identification	Ref.
22	Grasp bucket	Wrapped with aluminum foils, and sealed in ziploc bags		1 mm, 0.45 mm and 0.1 mm mesh sieves		Potassium formate (KF) solution	8 µm cellulose nitrate membrane filter	Stereomicroscope	µ-FTIR & Raman spectroscopy	(Fan et al., 2019)
23	Stainless-steel spoon	Aluminium container, and aluminium foil.	90	0.3 mm and 5 mm sieves	Fenton's reagent	Zinc chloride solution	0.3 mm sieve	Stereozoom Microscope	FT-IR attenuated total reflectance (ATR) unit	(Amrutha & Warrier, 2020)
24	Perspex tubes	Glass Container	90	metal mesh screens (0.3-5.0 mm)	Fenton's reagent	saturated NaCl solution	Whatman GF/B glass microfiber filter (pore size 1.0 µm)	stereo microscope	Fourier-transform infrared spectroscopy attenuated total reflectance (FTIR-ATR) & SEM	(Niu et al., 2021)

Table 3: An overview of Sample Analysis techniques of microplastics in the river sediment (Contd.)

No.	Sampling Device	Container	Drying Temp. (oC)	Size Selection	Digestion	Density Selection	Filtration	Visual Inspection	Identification	Ref.
25	Quadrat	Ziplock bag	50	2 mm mesh steel sieve		hyper-saturated saline solution	63 µm mesh	Olympus dissecting microscope		(Nel et al., 2018)
26	Stainless steel scoop	Glass Kilner jar	80	1–2 mm and 2–4 mm sieve		ZnCl2 solution	1.2 µm Whatman GF/C glass microfibre filter papers	binocular light microscope	Raman spectroscopy	(Horton et al., 2017)
27	Grab dredge & stainless steel shovel	aluminum foil sample bags	60		hydrogen peroxide	saturated sodium chloride solution	0.45 µm filter membranes	Magnifying glass (10×) & Vertical optical microscope	micro-Fourier transform infrared (FTIR) spectrometer	(L. Zhang et al., 2020)