Identification, Quantification and Removal of Microplastics from Various Sources

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DOCTOR OF PHILOSOPHY

Submitted by

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This thesis is dedicated to my Parents, Family Members and Mentors, who have motivated me to achieve my goal

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DECLARATION

I do hereby declare that the content embodied in this thesis entitled "***Identification, Quantification and Removal of Microplastics from Various Sources***" is the result of investigations carried out by me at the Department of Chemical Engineering, Indian Institute of Technology Guwahati, Guwahati, India, under the guidance of **Prof. Kaustubha Mohanty and Prof. Senthilmurughan S.** The result documented in this thesis are not submitted to any other university or institute for the award of any degree or diploma. In keeping with the general practice of reporting scientific observations, due acknowledgements have been made wherever the work described is based on the findings of other investigators.

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CERTIFICATE

This is to certify that **Mr. Naveenkumar Ashok Yaranal** (Reg. No. 176107022) has been working under our supervision since July 2017. We here by forward his thesis entitled **"*Identification, Quantification and Removal of Microplastics from Various Sources*"** to be submitted for the award of the degree of Doctor of Philosophy to IIT Guwahati. We certify that he has fulfilled all the requirements according to the rules of this institute and the investigations embodied in his thesis have not been submitted elsewhere for a degree or diploma.

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Abstract

Microplastics in the environment pose a significant threat to the entire ecosystem. The frequent utility of plastic in daily life, inadequate disposal, and improper waste management leads to a wide distribution of microplastic in atmospheric, terrestrial, and aquatic environments. Household, industrial, tyre wear and tear, construction, incineration, plastic litter, landfill, and agricultural activities are the major sources of microplastics in the environment. Microplastics are associated with various monomers and plastic additives. On the other hand, it becomes the carrier of toxic and hazardous chemicals from the surrounding environment. Microplastics enter the human body through the air, food, and drinking water (tap and bottled water). Taking all these issues into consideration, the main objectives of this work are divided into four sections. The first section deals with microplastics in Brahmaputra River water, tap water, and household purified water. The second section deals with the identification of microplastics in sea water and beach sediments. The third section deals with the identification of microplastics in Indian edible salts and the removal of microplastics to produce microplastic-free salt. The fourth section deals with the quantification of microplastics and removal from the laundry outlets using the electrocoagulation method.

Firstly, the work is focused on determining abundance, chemical composition, and selective elemental composition to assess the toxic potential of microplastics in Brahmaputra River water, tap water, and household purified water in the Guwahati city of Assam, India. Approximately 0.30 ± 0.08 to 2.56 ± 0.13 and 0.20 ± 0.03 to 0.52 ± 0.09 microplastic particles/L were determined in the river and tap water, respectively. There were no significant differences in microplastic concentration based on the water supply system (tap water) or

season. Approximately 55 - 60 % of all microplastics observed in river water were smaller than 1 mm in size, and with increasing size, the percentage contribution of various microplastic ranges reduced. Most of the microplastics found in the river water were fibers (58 %), colorless (49 %), polyethylene (30 %), polyester (20 %), and polypropylene (13 %). The tap water had 56 % fibers, 53 % colorless, and 31 % polyethylene, followed by 15 % polypropylene and 15 % pigmented polymer. An attempt was made to remove microplastics from tap water using hollow fiber ultrafiltration (HF-UF) membrane. The result showed that ultrafiltration was 100 % effective at eliminating microplastics (≥ 20 μm) from tap water.

The occurrence and distribution of microplastics in the marine water and beach sediments are collected from Karnataka state. The results revealed that marine water and sediments from Indian beaches has been polluted with microplastics. The total mean microplastic abundance in sea water from all considered locations is 8.2 ± 3 m^3. The highest microplastic concentration in beach sediments was found in Malpe beach (1002 ± 174 MP kg^{-1}), followed by Panambur beach (931 ± 156 MP kg^{-1}), which can be connected with a population, tourists, and industry around the shores. Based on morphology, the occurrence of fragment-shaped microplastics was dominant, followed by fibres. Polyethylene, polypropylene, and polystyrene microplastics were abundantly observed in all selected locations. The effects of long-term exposure to microplastics in the environment were visualized from FESEM images which showed the cracking and roughness on the surface of plastic particles. According to present study, microplastic concentration in the beach sediment is connected with the population, tourists, and industry around the shores.

Further, the work focuses on the presence of microplastics in edible salts, creating a potential health hazard for humans. The occurrence of microplastics in edible salts and their extraction procedure is limited. A facile and cost-effective protocol for the extraction and separation of

microplastics from the edible salt sample was developed. The visual assessment was performed to identify the shape, size, number, and colour of microplastic particles using light and fluorescence microscopy. The composition of the sample was analyzed by micro-Raman spectroscopy. A wide range of microplastics was found: 1400 ~ 1900 particles/kg in refined sea salt, 1900 ~ 2300 particles in unrefined sea salts, and 200 ~ 400 particles in rock salts. A relatively high number of microplastics were found in sea salts rather than rock salts. Sheet-type microplastics with size 1 ~ 4 μm were observed in rock salts. The most common microplastics were polyethylene, polypropylene, polyethylene terephthalate, nylon, and polystyrene. Additionally, microplastics were effectively removed from synthetic seawater with the help of a microfiltration membrane, which has the capability of arresting the transfer of microplastic particles into edible salts.

The last study focused on releasing and removing contaminants from laundry wastewater, particularly microplastics, and surfactants. The electrocoagulation method was used to remove the pollutants from laundry wastewater. According to the results, a reference load of 2 kg of synthetic materials releases 92,700 to 1,14,300 synthetic microfibers. The removal efficiency of microfibers, surfactants, and COD is higher at neutral pH. The percentage removal efficiency of microfibers, surfactants, and COD was 97.9 %, 91.2 %, and 86.3 %, respectively, at an operating time of 25 min, with a current density of 300 A/m^2 with optimum power consumption. The total operation cost of laundry wastewater treatment by electrocoagulation was 0.53 US $ /m^3 /44.12 INR/m^3.

Table of Contents

List of Figures

List of Tables

Abbreviations

ANOVA	Analysis of variance
ATR-FTIR	Attenuated total reflection-Fourier transform infrared
COD	Chemical oxygen demand,
CV	Control valve
DWTPs	Drinking water treatment plant
EDCs	Endocrine Disrupting Chemicals
FESEM	Field emission scanning electron microscope
µ-FTIR	Microscope attached with FTIR
µ-Raman	Microscope attached with Raman
HF-UF	Hollow fibre – ultrafiltration
HF-MF	Hollow fibre – microfiltration
IC	Ion chromatography
MPs	microplastics
MFs	Microfibers
NPs	Nanoplastics
NR	Nile red dye
PP-HF	Polypropylene hallow fibres
PA	Polyamide
PE	Polyethylene
PET	Polyethylene terephthalate

PCTE	Polycarbonate track etching
PES	Polyester
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinyl chloride
PU	Polyurethane
POPs	Persistent organic pollutants
ROS	Reactive oxygen species
WWTPs	Wastewater treatment plants

Symbol and Units

ºC	Degree Celsius
g	Gram
g/L or gL^{-1}	Gram per litre
Particles/kg	Microplastics per kilogram
Particles /m^3	Microplastics per cubic meter
MFs/kg	Microfibers per kilogram
MFs/m^2	Microfibers per square meter
MPs/y/person	Microplastics per year and per person
mL	Millilitre
µL	Microlitre
Particles kg^{-1}	Milligram per kilogram
mV	Millivolt
L^{-1}	Per litre
rpm	Rotation per minute

Chapter 1

1. Introduction

Preface: *In the early stage of this chapter, the numerous advantages of plastics, yearly worldwide plastics production, and plastic waste disposal in the various environment are briefly discussed. In addition, a brief discussion on microplastics sources, pathways and accumulation of microplastics into various environment is elaborated. Humans are now directly or indirectly exposed to microplastics due to the presence of microplastics in the air, food and food products (such as seafood, salt, honey, and etc.), and drinking water. The average human exposure to microplastics around the world and potential effects on human health were discussed. Finally, the study's motivation is also discussed in this chapter.*

1.1. Background

The widespread use of plastic may be attributed to their numerous attractive properties. Plastics are affordable, flexible, durable, lightweight, waterproof, easy to clean and sterilize, and act as insulators, among other things (Bergmann et al., 2015; Lebreton and Andrady, 2019). Plastics are commonly used in packaging, automotive, household leisure, electrical and electronic parts, building and construction materials, sports products, and the agricultural sector (Elizalde-Velázquez and Gómez-Oliván, 2021). Plastic production has grown exponentially, with large-scale production starting in the 1950s around 1.5 million tons per year to 368 million tons in the 2019s. Due to improper plastic waste disposal, a significant number of plastics eventually end up in the marine ecosystem via household materials, industrial discharge, wastewater treatment plants (WWTPs), incineration, landfills, and rivers. In 2015, five billion metric tonnes of plastic garbage were disposed of landfilled or scattered in nature, which is expected to increase to 12 billion metric tonnes by 2050 (Henry et al., 2019; Lee et al., 2021). According to the report, about 4.8 to 12.7 million metric tons of plastic garbage enter the marine environment each year (Iñiguez et al., 2017; Veerasingam et al., 2016), with nearly 1.15 – 2.41 million metric tons of plastic entering from the river system (Kim et al., 2018; Lebreton and Andrady, 2019). This has resulted in accumulation of plastic pollution in oceans worldwide (Aslam et al., 2020; Crawford and Quinn, 2016; Henry et al., 2019). This accumulation of large plastic litter in the marine environment have been extensively documented (Ajith et al., 2020; Du et al., 2020). The average quantity of plastic debris on the surface of the ocean is around 18,000 items per km^2 and more higher amount of plastics are observed in North Atlantic and Pacific Oceans (Ma et al., 2019b). This could be due to plastics being easily transported over greater distances by ocean currents, hydrodynamic processes, and atmospheric currents (Rochman et al., 2015; See et al., 2020). In addition, degradation of plastics due to physical,

chemical, and microbiological processes also lead to break down of plastic waste into microplastic and nanoplastic particles (Golwala et al., 2021; Lambert and Wagner, 2016).

Introduction to microplastics: Microplastics are plastic particles with a diameter between 0.1 mm and 5 mm. Typically, nanoplastics are defined as plastic particles smaller than 0.1 mm. Due to limitations in analytical techniques, quantification of nanoplastics are not well defined. Further, this work is mainly focused on microplastics. Microplastics, based on their origin, are classified into primary and secondary categories (Blair et al., 2019; Boucher and Friot, 2017; Herbort et al., 2018).

Primary microplastics: These are intentionally produced small plastic particles or fibres that are directly released into the environment. There sources include (i) cosmetic and cleaning products such as soaps, make-up foundation, body scrubs, lotions, toothpaste, peeling, baby products, nail polish, sunscreen, insect repellents, (ii) industrial process, (iii) synthetics clothes (iv) pre-production plastics products (Carr et al., 2016; Veerasingam et al., 2020). The industry originated feedstocks used in plastic manufacturing, plastic resin powder/pellet spillage during air-blasting, 3D printer ink (Rios Mendoza et al., 2018), and textile fibers also lead to a high level of plastics and microplastic generation in the environment (Talvitie et al., 2017). Textiles release microplastics in the form of synthetic fibres to the atmosphere during the process of production to end use (De Falco et al., 2018; Henry et al., 2019; Stanton et al., 2019).

Secondary microplastics: Secondary microplastics formed as a result of breakdown of bigger plastic items such as bottles, bags, toys, construction materials, paints, packaging

materials, electronics items, and vehicle tyre (Carr et al., 2016; Da Costa et al., 2016). Secondary microplastics are also generated during the collection and disposal of municipal solid waste and as the result of anthropogenic actions such as littering (Du et al., 2020; Horton et al., 2017b). Considering the enormous volume of macroplastics entering the environment, secondary microplastics are seen as a major contributor to microplastic contamination in the environment. Plastic items are weathered as they travel through the environment, creating microplastics as a result of mechanical fragmentation, chemical, and biological degradation. These factors lead to continuous temporal changes in the physical and chemical characteristics of plastics (Cai et al., 2018; Oliveira et al., 2020; Singh and Sharma, 2008; Zbyszewski et al., 2014). Photodegradation of the polymer matrix causes bond breakage and brittleness in plastics, leading them to disintegrate. Among others, Cai et al. (2018) studied the impact of UV exposure for three polymers types of polystyrene (PS), polypropylene (PP), and polyethylene (PE). Their exposure studies have revealed that the pellet surface homogenous texture changed drastically over months of exposure, where cracks and flakes were common patterns of this degradation process.

These primary and secondary category microplastics are ubiquitous in the atmospheric, terrestrial, and aquatic environment. Abundance of primary microplastics may be due to inadequate waste water treatment and waste management before disposal. Secondary microplastics, on the other hand, are mainly created as a result of direct disposal of plastic waste in the outdoor environment (Ajith et al., 2020)(Golwala et al., 2021).

Microplastics in aquatic environment (particularly in coastal and marine environments) can cause a variety of health and environmental problems (Sruthy and Ramasamy, 2017). The

recent microplastic study has raised awareness of microplastic threats to fish, crabs, seashells, crustaceans, and other aquatic species/environments (Li et al., 2015; Weber et al., 2020; Wright and Kelly, 2017). Microplastics may also accumulate in marine species and make their way up the food chain, eventually reaching higher trophic levels, such as humans. Recent investigations show microplastics have been observed in human lungs, digestive tract, placenta, blood, breast milk, and feces (Pauly et al., 1998; Ragusa et al., 2021; Schwabl et al., 2019; Toussaint et al., 2019). The humans are directly or indirectly exposed to microplastics. Direct exposure of pollutions to organisms occurs through different mediums, including water and air. Indirect exposure, on the other hand, occurs through the food chain or in conjunction with other chemicals (additives, heavy metals, and POPs). In general, direct exposure causes significant toxicity in the near term, whereas indirect exposure can cause long-term organ damage (Du et al., 2020). Microplastics found in table salt, marine food, and drinking water can enter the human body via the digestive tract, but microplastics found in the atmosphere can expose both the respiratory and gastrointestinal systems (Amato-Lourenço et al., 2021; Huang et al., 2022; Pauly et al., 1998; Stapleton, 2021). A schematic diagram of sources, pathways, and toxic effects of microplastic is shown in **Fig. 1**. However, evidence on human exposure to microplastics is limited. Knowing all microplastic sources, pathways, and abundance in atmospheric, terrestrial, and aquatic environments is essential to understand better and control microplastic pollution. The following section reviewed the possible sources, pathways, and abundance of microplastics in Atmospheric, terrestrial and aquatic environments.

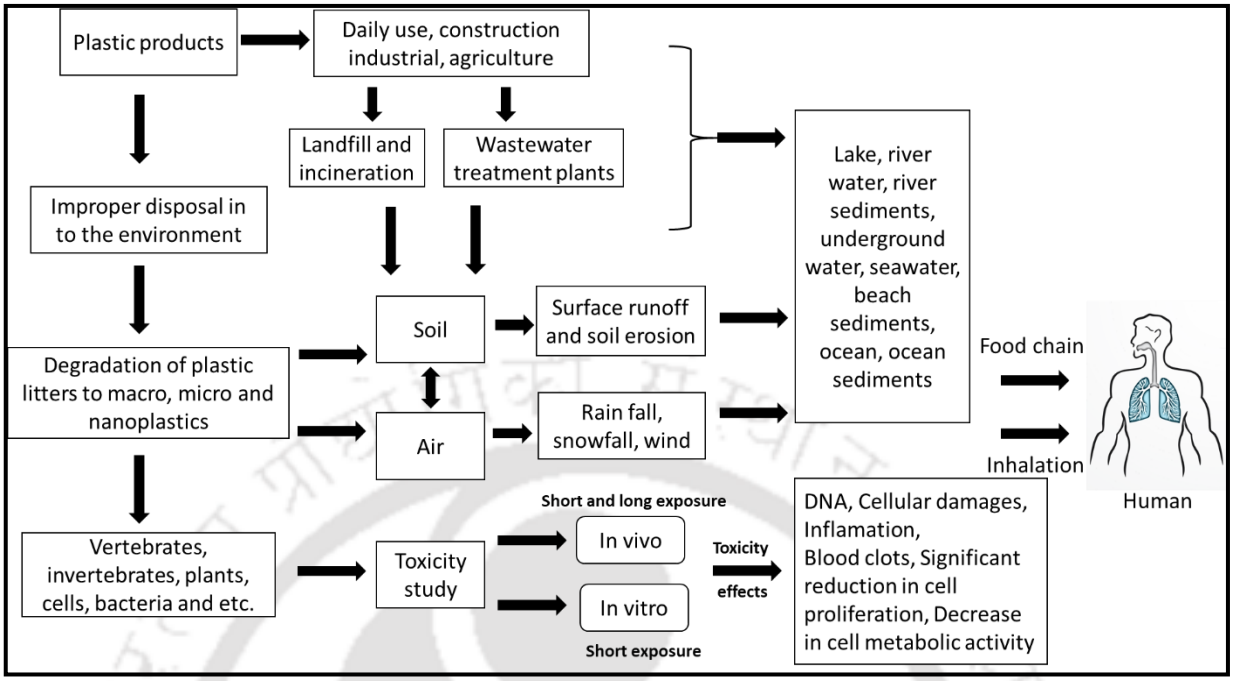


Figure 1. Schematic diagram of sources, pathways, and toxic effects of microplastics.

1.2. Sources, Abundance, Pathways and accumulation of microplastics into various environment

The plastic industry has outgrown exponentially, and the use of plastics has increased in recent years. Historically the global production of plastic materials has increased by around 8 - 10 % each year (Crawford and Quinn, 2016; Peixoto et al., 2019). It has been predicted that the usage will double in the next 20 years (Perren et al., 2018). On the other side, the unregulated disposal practices of plastics have resulted in aquatic, terrestrial, and now atmospheric environments. All three environments are interconnected through diverse pathways and sources that can affect the microplastic accumulation and flux in these matrices. The microplastic sources, transport pathways, and accumulation among different environments are depicted in **Fig. 2**.

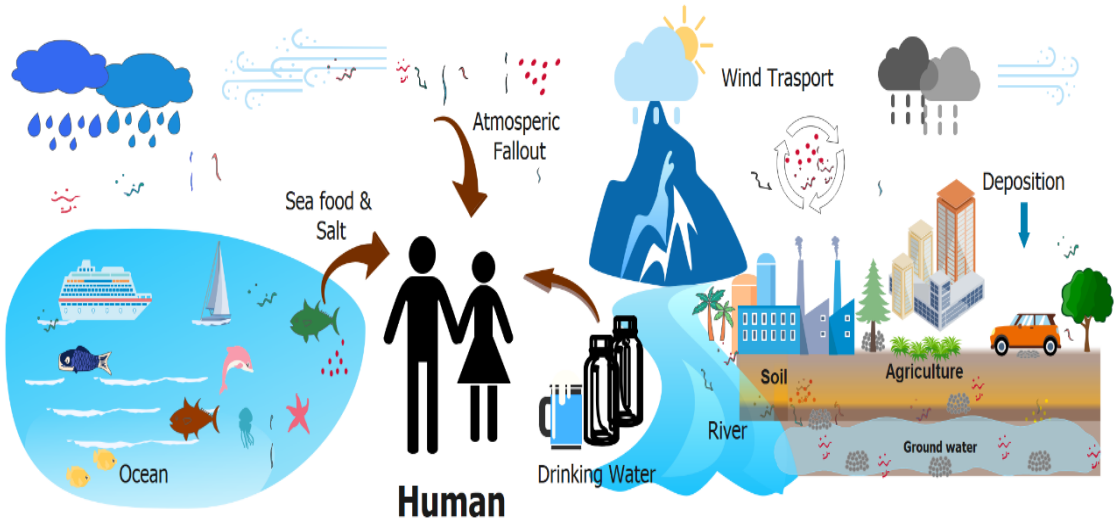


Figure 2. Sources, pathways and accumulation of microplastic in atmospheric, terrestrial, and aquatic environments.

1.2.1. Atmospheric environment

Sources: Plastic fragments from household goods, landfills, incineration (mostly due to incomplete incineration), industrial emissions, particle resuspension, and sewage sludge used as soil fertilizer are all potential sources of airborne microplastics (Dris et al., 2016; Henry et al., 2019; Lassen et al., 2015; Sujathan et al., 2017). Furthermore, incomplete garbage incineration releases a huge quantity of microplastic particles into the environment. Synthetic textiles are the primary source of microplastics in the atmosphere (Gasperi et al., 2018). Grinding, cutting, and chopping in synthetic textiles can also produce a lot of microscopic fibers. Additionally, small fibers easily tear from clothes during wearing, cleaning, and drying (Henry et al., 2019). Another source of airborne microplastics is busy traffic in the cities (G. Chen et al., 2020).

Abundance: To our knowledge, Greater Paris was the first region to study the presence of microplastic particles in the air (Dris et al., 2015). Dris et al. (2015) collected the total

atmospheric fallout in a dense urban area with an average of 118 particles m^{-2} day^{-1} (more number microplastics were observed in the rainy season), and Dris et al. (2016) collected the microplastics with the rain sampler and result revealed that more numbers of microplastics in the urban area (high population density) compared with the sub-urban (less dense) environment. Further, Dris et al. (2017) carried out sampling in indoor (private apartment and office) and outdoor environments (just outside the apartment). Results revealed that indoor air (1-60 particles/m^3) contains a higher microplastic concentration than outdoor air (0.3-1.5 particles/m^3). Catarino et al. (2018), in Edinburg, observed the household MFs fallout during a meal with a concentration and deposition of 5 ± 3.3 to 10 ± 4.2 particles/m^3 and 1666 to 1672 particles m^{-2} day^{-1}, respectively. Vianello et al. (2019) investigated indoor air sampling in 3 apartments in Denmark, and microplastic particles concentrations were observed between 1.7 and 16.2 particles/m^3 (81 % of particles were polyester). Liu et al. (2019a) and Cai et al. (2017) analysed airborne microplastics in Shanghai and Dongguan city, China, respectively. In Shanghai city, the total atmospheric fallout concentrations range was 0 - 4.18 particles/m^3. In Dongguan City, the total atmospheric fallout concentrations range was 175 to 313 particles m^{-2} day^{-1}. Kaya et al. (2018), in Sakarya province at a university campus, Turkey, collected the microplastics in the air using a vacuum pump, and sorted them through a stereomicroscope, found the microplastics in the range of 259 - 12,895 particles/L. Abbasi et al. (2019), Su et al. (2020), Yukioka et al. (2020) and Patchaiyappan et al. (2021) identified the presence of microplastics in suspended dust. The airborne microplastics were identified in the West Pacific Ocean and North Atlantic Ocean atmosphere by Liu et al. (2019) and Trainic et al. (2020). The deposition of the microfibers (MFs) on snow and mosses from the atmosphere was also confirmed by Bergmann et al. (2019) and Roblin and Aherne (2020). These findings, therefore,

confirm the ubiquitous existence of microplastics in the global ecosystem. The concentrations and characteristics of microplastics in available atmospheric data is summarised in **Table 1**.

Pathways and accumulation: The climatic and seasonal conditions play an essential role in the deposition and concentration of the microplastic in the atmosphere. The density and buoyancy affect the vertical distribution of microplastic in the atmosphere. Microplastics in the urban environment are often associated with extensive human activity. Some potential sources of microplastics in the atmosphere are landfills, construction waste, industrial pollutants, urban dust, agricultural practices, and plastic items form dump yard (G. Chen et al., 2020; Qiu et al., 2020; Y. Zhang et al., 2020). Microplastics were easily dispersed by wind and might stay for a long period in the atmosphere, transferring them to remote places (Trainic et al., 2020). Liu et al. (2019a, 2019b) revealed a more significant concentration of microplastics in land-based (atmospheric air) sampling than in sea air. Dris et al. (2016) found a similar finding in their investigation that total atmospheric fallout was higher in a dense urban area than in a less dense suburban area. Rain snow and wind have been proven to influence microplastic deposition in terrestrial and marine environments (Allen et al., 2019; Gasperi et al., 2018).

Table 1. Worldwide abundance and characteristics of atmospheric microplastics.

Study area and air type	Identification	Sample preparation method	Shape	Size (µm)	Composition	Microplastics abundance	Reference
Paris, France (urban outdoor)	Stereomicroscope	Filtration	Fibres, fragments	100- 1,000	N/A	29 - 280 particles m^{-2} day^{-1}; Average:118 particles m^{-2} day^{-1}	(Dris et al., 2015)
Paris, France (indoor and outdoor)	Stereomicroscope, µ-FTIR	Vacuum pump + density separation ($ZnCl_2$) + filtration (1.6 GF)	Fibres	50 – 4,850	RY, PE, PA, PP, PET	Indoor: 1 - 60 fibres m^{-3} (1586-11,130 fibres m^{-2} day^{-1}); Outdoor: 0.3 - 1.5 fibres m^{-3}	(Dris et al., 2017)
Edinburgh, UK (indoor home)	Fluorescence microscope, µ-FTIR	Density separation (NaCl) + filtration	Fibres	≥ 500	PET, PUR	5±3.3 - 10±4.2 particles m^{-3}	(Catarino et al., 2018)
Aarhus, Denmark (indoor)	µ-FTIR	Filtration	Fibres	4 - 400	PES, PE, nylon	1.7 – 16.2 particles m^{-3}	(Vianello et al., 2019)
Dongguan, China (urban outdoor)	Stereomicroscope, µ-FTIR, SEM	Filtration (1 µm, GF)	Fibres, fragments, foams, films	200 – 4,200	PE (14%), PP (9%), PS (4%)	175 - 313 particles m^{-2} day^{-1}	(Cai et al., 2017)
Shanghai, China (urban - outdoor)	Stereomicroscope, µ-FTIR	Filtration (1.6 µm, GF)	Fibres, fragment, granule	23 - 9,555	PE, PET, PES, PAN, RY	0 - 4.18 particles m^{-3}	(Liu et al., 2019a)
Shanghai, China	Microscope, µ-FTIR	Filtration	Fibres, fragments, and microbeads	< 5,000	PET, PE, PP, PA and PS	0.05 to 0.07 particles m^{-3}	(Liu et al., 2019b)
Hamburg, Germany (metropolitan area)	Fluorescence microscope, µ-Raman spectroscopy	Digestion (NaClO) + filtration	Fragments and fibres	< 63 – 5,000	PE, PVA, and PET	136.5 and 512 particles m^{-2} day^{-1}	(Klein and Fischer, 2019)
Pyrenees mountains, France (remote mountain area)	Stereomicroscope, µ-FTIR	Filtration	Fragments, films, fibres	25 - 2,600	PS, PE, PP, PVC, PET	365 ± 69 particles m^{-2} day^{-1}	(Allen et al., 2019)

Study area and air type	Identification	Sample preparation method	Shape	Size (µm)	Composition	Microplastics abundance	Reference
Snow from Europe and artic (Alps, N. Germany, Artic)	Stereomicroscope, µ-FTIR	Filtration (0.2 µm, aluminum oxide)	Fibres	11 - 5000	PE, rubber, PA, PS and polyester	0.19 - 154 × 10^3 N L^{-1}	(Bergmann et al., 2019)
Asaluyeh County, Iran (suspended dust, urban dust-outdoor)	Fluorescence microscope, SEM-EDS	Digestion (30% H_2O_2) + density separation (NaI) + filtration	Spherules, films, fragments, fibres	2 - 1,000	N/A	900 MPs and 250 micro rubbers in 15 gm dust sample. Average: 0.3 - 1.1 particles m^{-3}	(Abbasi et al., 2019)
Victoria, Australia (roadside dust from rural and urban area)	Stereomicroscope, µ-FTIR	Dust collected (sweeping) + screening + digestion (30% H_2O_2) + density separation (NaCl) + filtration	Fibre, fragment, film and pellet	20 – 4,700	Polyester and PP	20.6 to 529.3 items kg^{-1}	(Su et al., 2020)
Kusatsu, Japan, Da Nang, Vietnam, and Kathmandu, Nepal (road dust)	Stereomicroscope, ATR-FTIR	Digestion (30% H_2O_2) + density separation (NaI) + filtration	Fragments, film, fibres, and granules	100 - 5000	Rubbers, PE, PP, PS and PET	Kusatsu: 2.0 ± 1.6 pieces m^{-2}; Da Nang: 19.7 ± 13.7; Kathmandu: 12.5 ± 10.1 pieces m^{-2}	(Yukioka et al., 2020)
Chennai, India (suspended dust - outdoor)	Fluorescence microscope, µ-Raman, SEM-EDS	Digestion (0.05 M Fe + 30 % H_2O_2) + density separation (NaI) + filtration	Fragments	> 50 - < 5000	PVC, PE, PTFE	227.94 ± 91.37 particles/100g dust sample	(Patchaiyappan et al., 2021)
Ireland (mosses from lakes catchment)	Stereomicroscope, Raman spectroscopy	Digestion (0.05 M Fe + 30 % H_2O_2) + filtration	Fibres	< 4,310		24 g^{-1} dry weight of mosses	(Roblin and Aherne, 2020)
West Pacific Ocean (sea air)	Stereomicroscope, µ-FTIR	Filtration (GF)	Fibres, fragment, granule, microbeads	20 - 500	PET, PE-PP, PS, EP, others	0-1.37 particles m^{-3}	(Liu et al., 2019c)
North Atlantic Ocean (sea air)	Stereomicroscope, µ-Raman	Filtration	N/A	1-100	PP, PE, PP, poly-silicone	N/A	(Trainic et al., 2020)

1.2.2. Terrestrial environment

Sources: The presence of a wide range of microplastic debris in the terrestrial environment (parks, gardens, wetlands, industrial and agricultural soil) is primarily due to excessive use and their improper disposal (J. Li et al., 2020). On the one hand, microplastics enters the agricultural soil through sewage sludge, digested residues, irrigation with polluted waters, polymer-based fertilizer and pesticides, atmospheric deposition, littering, plastics items from landfills, and surface runoff (Da Costa et al., 2016; Sujathan et al., 2017; Talvitie et al., 2017). Microplastics in agricultural soils, on the other hand, are created by the degradation of plastic materials utilized in agricultural practices (Liu et al., 2018; Rehm et al., 2021).

Abundance: Microplastics presence and dispersion in soils have not been well investigated; however, the current data **(Table 2)** demonstrated that microplastic contamination exists in soil matrices globally. The presence of microplastics in soil affects the growth, reproduction, and immune system of organisms (W. Li et al., 2020). According to Liu et al. 2018, microplastics can occur in top soil as well as subsurface soils; however, research on microplastic pollution in the soil is minimal. Some of the researchers, such as Fuller and Gautam (2016), conducted the first survey of the concentration and distribution of microplastics in industrial soil. Furthermore, microplastics have been identified in parks, gardens, wetlands (Yang et al., 2021b), industrial farmlands (Fuller and Gautam, 2016), and floodplain soils (Scheurer and Bigalke, 2018). The average microplastics found in industrial soils of Sydney were 300 - 67,500 mg kg^{-1} and 5.5 mg kg^{-1} or 593 items kg^{-1} of microplastics in floodplain soil of Switzerland (Fuller and Gautam, 2016; Scheurer and Bigalke, 2018). Many researchers have identified the presence of microplastics in farmland soils of China and reported a high

concentration ranging from 50 - 12,560 particles/kg (Y. Chen et al., 2020; Liu et al., 2018; Wang et al., 2017; B. Zhou et al., 2020; Zhou et al., 2016) as show in **Table 2**. Low and high numbers of microplastics were found in the agricultural soil of Germany (0.34 - 0.36 particles/kg) and Spain (190 – 5,190 particles/kg), respectively (Piehl et al., 2018; Van den Berg et al., 2020). Microplastic accumulation in the soil with sewage sludge and mulching applications was confirmed by Piehl et al. (2018) and B. Zhou et al. (2020). The study of Van den Berg et al. (2020) also showed that farmlands without sludge applications had an average of 2,030 microplastics kg^{-1} while farmland with sludge application had an average of 5,190 microplastics kg^{-1}. B. Zhou et al. (2020) observed higher amount of microplastics in mulching soil than non-mulching soil, with an average of 571 pieces kg^{-1} and 263 pieces kg^{-1}, respectively. The above studies revealed that microplastic content increased with mulching and sewage sludge applications in the farmland. Plastics are likely to break down further and degrade into the finer particle, and thus increasing microplastic pollution. The variation in abundance of microplastics mainly depends on the continuing damage and degradation of the film mulch over time due to biotic and abiotic factors. Many factors, such as cultivation, fertilization, and sampling sites, are the main reason for the diversified abundance of microplastics in different regions. Based on review data, it is evident that microplastics in the terrestrial environment is rapidly increasing. Eco friendly materials, techniques and fertilizers in agricultural practices can reduce the microplastic presence. Easy standard sampling procedure should be developed and adopted for identification and quantification of microplastics in terrestrial environment.

Pathways and accumulation: Due to non-degradability and various other properties, microplastics may remain in the environment for a longer period, affecting biodiversity and the

environment, including the growth and reproduction of plants and organisms (Kumar et al., 2020; W. Li et al., 2020). Arable soils can be polluted with microplastics via various pathways. Due to its remarkable benefits, PE and polyvinyl chloride (PVC) composed mulch films have become extensively used in global agricultural production. Plastic mulch is widely used in agriculture practices and covers around 20 million hectares of farmlands worldwide, where China alone accounts for nearly 90 % of it (Yang et al., 2021b; Y. Zhou et al., 2020). In 2016, globally, 4 million tons of plastic film was used in agriculture and predicted to rise to 5.6 million tons per year by 2030. The removal of the mulching film from the agriculture field is a time-consuming and labor-intensive task; as a result, certain parts of it are left on farms, either purposely or inadvertently. Different organisms, including earthworms, can aid in the production of microplastics and nanoplastics from larger plastic fragments (Kumar et al., 2020; Piehl et al., 2018). The current overall understanding of possible microplastic sources in agricultural soils is limited, owing to the difficulty of detecting microplastics in the soil (Möller et al., 2020).

According to J. Li et al. (2020), agricultural soil is highly porous media; thus, particles (0.1 – 6.0 μm) can easily move vertically along with the soil. Based on the different studies on estimates of microplastics in the soil, the agriculture soil is considered as a major microplastic sink. It is sometimes debated whether the accumulation of microplastics in the soil leads to a permanent or whether it is lost again through leaching into groundwater or surface runoff and erosion. Because agricultural soils are particularly prone to erosion, the possibility of microplastics being transported by surface runoff is of major importance (Y. Chen et al., 2020; Rehm et al., 2021). The highest plastic transport was recorded in the Rhône River a few days after rainfall events, demonstrating that surface runoff may have a significant impact on microplastics intake to water bodies when compared to other processes (Castro-Jiménez et al.,

2019). The studies of Piehl et al. (2018) and Zhou et al. (2020) found that frequent uses of plastic material in the farmland are major contributors to plastic pollution in the agricultural fields. In addition, careless disposal and displacement of plastics by wind were the primary routes for input to croplands.

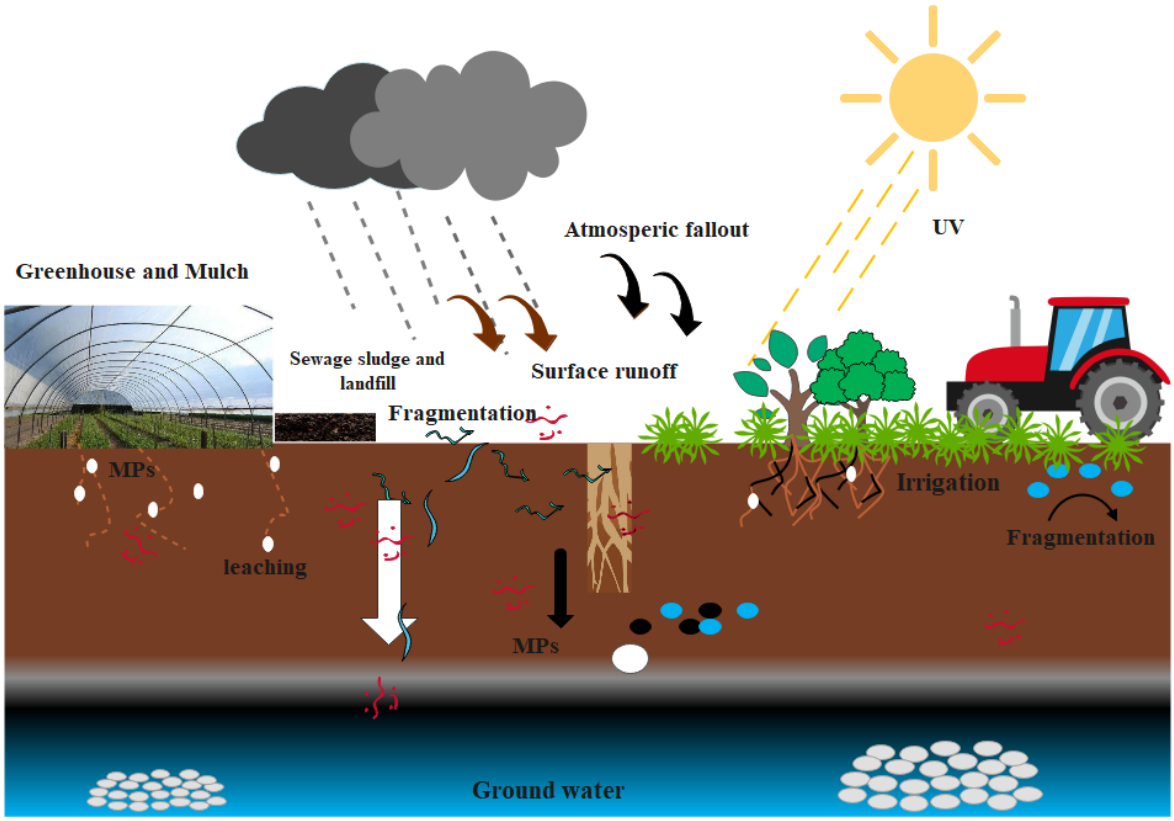


Figure 3: Sources, accumulation and transport of microplastics in soils.

Table 2. Worldwide abundance and characteristics of terrestrial microplastics.

Study area	Identification	Shape	Size (µm)	Sample preparation	Microplastics abundance	Composition	Reference
Sydney, Australia (soil from industrial area)	ATR-FT-IR	N/A	< 1mm	Sieve	300 – 67, 500 mg kg^{-1}	PVC, and PE	(Fuller and Gautam, 2016)
Shandong, China (coastal soil)	Stereomicroscope, SEM-EDS	Granules, fragment fibres, films	< 4.7 mm	Density separation (NaI) + filtration	634 particles/kg	N/A	(Zhou et al., 2016)
Switzerland (floodplain)	Microscope, FT-IR	N/A	< 1 mm	Digestion (HNO_3) + density separation (NaCl, $ZnCl_2$) + filtration (0.45 µm)	55.5 mg kg^{-1} or 593 particles kg^{-1}	PE, PS, and PP	(Scheurer and Bigalke, 2018)
Southeast Germany (farmland soil)	Stereomicroscope, ATR-FT-IR	Films and fragments	< 5mm	Digestion (H_2O_2) + filtration	0.34 ± 0.36 particles/kg	PE, PS, and PP	(Piehl et al., 2018)
Shanghai, China (farmland soil)	µ-FT-IR	Fibres, fragments, films, and pellets	< 5 mm	Digestion (H_2O_2) + density separation (NaCl) + filtration (20 µm)	Top soil: 78 ± 12.91; Deep soil: 62.50±12.97 items kg^{-1}	PP and PE	(Liu et al., 2018)
Nanjing and Wuxi, China (farmland soil)	µ-FT-IR	Fibres, fragments	< 5 mm	Digestion (H_2O_2 + H_2SO_4) + density separation (NaCl, ZnCl2) + filtration (2 µm)	420–1,290 MP particles/kg	PE and PP	(Li et al., 2019)
Wuhan, China (vegetable farmland soil)	Stereomicroscope, µ-Raman spectroscopy	Fibres and microbeads	< 5 mm	Density separation ($ZnCl_2$) + filtration (0.45 µm)	320 to 12,560 particles/kg	PA, and PP	(Y. Chen et al., 2020)
Shihezi City, Xinjiang, China (farmland soil)	Microscope, SEM ATR-FT-IR	Film	< 5 mm	Flotation + filtration	40.35 mg kg^{-1}	PE and others	(W. Li et al., 2020)
Hangzhou Bay, China (farmland soil)	Stereomicroscope, µ-FT-IR	Fragments, films, fibres	< 5 mm	Density separation (NaCl) + filtration	Mulching soils: 571 particles/kg; Non-mulching soils: 263 pieces kg^{-1}; Irrigation water: 3.9 – 17 particles/kg	PE, PP, PA polyester, rayon, and acrylic	(B. Zhou et al., 2020)

Study area	Identification	Shape	Size (µm)	Sample preparation	Microplastics abundance	Composition	Reference
USA (wetland)	Microscope, ATR-FT-IR	Fibres and fragments	< 5 mm	Digestion (H_2O_2) + filtration	1,270 ± 150 particles/kg Or 23200 ± 2500 particles m^{-2}	PS, PE, and synthetic rubber	(Helcoski et al., 2020)
Valencia, Spain (sewage sludge and soil)	Microscope, µ-FT-IR	Fragment, fibres, film and foam	< 5 mm	Density separation (NaI) + filtration	Sewage sludge: 18,000 ± 15,940 and 32,070 ± 19,080 particles/kg; Soil without sludge: 930 ± 740 and 1,100 ± 570 particles/kg; Soil with sludge: 2,130 ± 950 and 3,060 ± 1,680 particles/kg	PP, and PVC	(Van den Berg et al., 2020)
Mellipilla, Chile (crop lands and pastures)	Microscope, µ-FT-IR	Film, fibres and fragments	< 5 mm	Density separation (NaCl, $ZnCl_2$) + filtration	Crop land: 306 ± 360 particles/kg^{-1}; Pastures: 184 ± 266 particles/kg	Acrylates, PU, PE, PP and PA	(Corradini et al., 2021)

1.2.3. Aquatic environment

Sources: Microplastic enters the freshwater via different pathways, significantly through drainage systems from domestic and industrial waste discharge, textile industry, WWTPs effluent, incomplete incineration, and secondary source microplastics (municipal solid waste, land filling, and plastic disposal near the rives e.g., food wrappers, bags, bottles, and disposable styrofoam food containers) (Horton et al., 2017b; Koelmans et al., 2019; Wilkinson et al., 2017).

Plastic trash enters the marine ecosystem from continental sources, primarily through rivers, industrial and municipal effluents, and runoff from coastal sediments and neighbouring farms. The other part is caused by direct inputs such as offshore industrial operations (e.g., aquaculture, oil and gas production), shipping, degradation of fishing nets, and trash generated through marine activities such as tourism (Karthik et al., 2018). Furthermore, Cai et al. (2017) suggested that certain microplastics in the aquatic system might be caused by air fallout.

Abundance: The microplastics detected around the world, including lakes, rivers, sediments, beaches, and seawater, are presented in **Table 3**. The microplastics concentration in surface water viz. 379 - 12,611 particles/m^3 in fresh water of China (Di and Wang, 2018; Lin et al., 2018), 58 – 1,265 particles/m^3in Antuã River of Portugal (Rodrigues et al., 2018), 2.68 - 3.36 particles/m^3 in lake of Italy (Fischer et al., 2016), 67 - 11,532 particles/m^3 in Meuse and Dommel rivers of Netherland (Mintenig et al., 2020), and 3.52 to 32.05 particles/m^3 in Carpathian basin of Europe (Bordós et al., 2019) was reported. While the concentration of microplastic in the sediment of the same water system was observed in the range of 25 - 7924 particles/m^3 in China (Di and Wang, 2018; Lin et al., 2018), 18 - 629 particles/m^3 in Portugal (Rodrigues et al., 2018), 234 particles/m^3 in Italy (Fischer et al., 2016), and 0.46 to 1.62

particles/m^3 in Europe (Bordós et al., 2019). Recent researchers in India Selvan et al. (2021) identified the presence of microplastics in groundwater. Moreover, J. Wang et al. (2017) reported that the microplastic concentrations in sediment are much higher than that in freshwater. Microplastics abundance in freshwater and sediment is generally higher in China when compared to other countries **(Table 3)**; the rise in concentration is attributed to the rapid economic development of the country. Studies in China, Korea, Netherland, Italy, Turkey, and Tunisia reported the highest microplastics pollution in seawater and sediments. Coastal areas recorded the maximum concentrations of microplastics (Kang et al., 2015; Lots et al., 2017; Nel and Froneman, 2015). The concentration of microplastics in the ocean varied with different regions. For instance, the average abundance of seawater was around 1.15 particles/m^3 in the Atlantic Ocean (Kanhai et al., 2017); 1.0×10^4 particles km^{-2} in the North-western Pacific Ocean (Pan et al., 2019); 2 - 17 particles/L in sea ice from Arctic Central Basin and 0 - 18 particles/m^3 in seawater beneath ice floes of Arctic Ocean (Kanhai et al., 2020).

Pathways and accumulation: The microplastics present in cosmetics, cleaning products, and MFs from cloth can enter the aquatic environment through industrial or domestic drainage systems. According to De Falco et al. (2018), the primary microplastics infiltrate the aquatic environment through WWTPs in urban areas. Surface runoff from agricultural fields and urban areas is another significant source of microplastics in surface water (WHO, 2019). Additionally, data suggests that tyres and road markings may contribute to microplastic contamination, with storm-water runoff as a major pathway for plastic particles to reach surface waterways. Microplastics may also be deposited on the terrestrial environment surface, where they may be re-suspended into the air by the wind or transferred into aquatic environments by stormwater/surface runoff (Abbasi et al., 2019; Prata, 2018). As a result, the interchange of

microplastics between air, terrestrial, and aquatic ecosystems constitute the circulation of microplastics in the environment. According to research calculations, every year, approximately 4.8 to 12.7 million metric tons of plastics waste ends up in the marine ecosystem (Veerasingam et al., 2020) in that nearly 1.15 to 2.41 million metric tons of plastics reaches through rivers (Kim et al., 2018; Lebreton and Andrady, 2019). Land-based plastic debris contributes to 80 % of all plastic garbage in the ocean (Issac and Kandasubramanian, 2021; Y. Li et al., 2020). Estuaries and coastal lagoons are where the bulk of aquaculture species are cultivated. The significant use of plastic materials in the fishing and aquaculture sectors is the primary cause of microplastic pollution in the ocean (Du et al., 2020; Thiele et al., 2021). According to Eriksen et al. (2014), the oceans already contain more than five trillion particles of plastic weighing more than 2,50,000 tones. These microplastics are a hundred times lower on the sea surface than predicted, lending credence to the notion that most microplastics sink and collect in the core region of the ocean. Some have been found frozen in the Arctic ocean ice, which has become a global sink for microplastics (Kanhai et al., 2020). Microplastics with densities lower than seawater float at the surface layer or are suspended in the water column with neutral density, whereas microplastics with densities higher than seawater are concentrated in marine benthic regions (Ajith et al., 2020; Y. Li et al., 2020). Some of the factors, such as turbulence and biofouling, increase the density of microplastics.

Table 3. Worldwide abundance and characteristics of aquatic microplastics.

Study area and sample type	Identification methods	Sample preparation method	Size (µm)	Composition	Microplastics abundance	Reference
China, Three Gorges Reservoir	Microscope, µ-Raman spectroscopy	Digestion (30% H_2O_2) + filtration (0.45 µm, GF)	< 5000	PC, PE, PP, PS, PVC	Surface water: 1,597– 12,611 particles/m^3; Sediment: 25 – 300 particles/kg	(Di and Wang, 2018)
China, Pearl River	Stereo light microscope, µ-FTIR	Digestion (30% H_2O_2) + filtration	20 - 5000	PP, PE, PET	Surface water: 379–7,924 particles/m^3; Sediment: 80 – 9,597 particles/kg	(Lin et al., 2018)
Portugal, Antuã River	Stereomicroscope, ATR-FTIR	Digestion (Fe + 30% H_2O_2) + density separation ($ZnCl_2$), filtration (0.45 µm)	55 - 5000	PP, PE, PS, PET	Surface water: 58 - 1,265 particles/m^3; Sediment: 18–629 particles/kg	(Rodrigues et al., 2018)
Italy, Lake Chiusi,	UV- microscope, SEM	Digestion (HCl) + density separation (NaCl), filtration (0.45 µm)	< 5000	N/A	Surface water: 2.68–3.36 particles/m^3; Sediment: 234 particles/kg	(Fischer et al., 2016)
Europe, Carpathian basin	Microscope, ATR-FTIR	Density separation (NaCl), filtration	200-5000	PE, PP, PTFE, PS	Surface water: 3.52 – 32.05 particles/m^3; Sediment: 0.46 – 1.62 particles/kg	(Bordós et al., 2019)
Netherland, Meuse and Dommel river	Stereomicroscope, µ-FTIR, ATR-FTIR	Digestion (H_2O_2), density separation ($ZnCl_2$), vacuum filtration	<25 - 1000	PE, PP, and EPDM	Surface water: 67 – 11,532 particles/m^3	(Mintenig et al., 2020)
India, Ground, surface water	Stereomicroscope, ATR-FTIR, AFM, ICP-OES	Digestion (Fe + 30% H_2O_2) + filtration	300 - 5000	PP, PA, PE, and PVC	Ground water: 10.1×10^{-3} particles/L, Surface water: $19.9 \times 10^{-3} \times 10^{-3}$ particles/m^3	(Selvam et al., 2021)
Germany, Drinking water treatment plants	Microscope, µ-Raman spectroscopy	Vacuum filtration	5 - 1000	PE, PET, PP, PA	197 particles/m^3(raw water), 1–102 particles/m^3 (DWTP), 6 - 74 particles/m^3 (Tap water)	(Pittroff et al., 2021)
Korea, Geoje island, Seawater	Microscope, FTIR	N/A	<5000, and >5000	PE, PP, PES, ALK, PS	0.62– 15,560 particles/m^3	(Kang et al., 2015)
European, beach sediments (13 different countries)	Microscope, Raman spectroscopy	Density separation + filtration	< 5000	PES, PE, PP	72±24 -1,512 ±187 particles/kg	(Lots et al., 2017)
South Africa, South-eastern coastline seawater and beach sediments	Microscope	Vacuum filtration	< 5000	N/A	Seawater: 275.9 – 1,215 particles/m^3; beach Sediment: 688.9–3,308 particles m^{-2}	(Nel and Froneman, 2015)

Study area and sample type	Identification methods	Sample preparation method	Size (µm)	Composition	Microplastics abundance	Reference
China, Maowei Seawater	Microscope, µ-FTIR	Vacuum filtration	< 5000	PP, PE, PES, PA, PS	1,200–10,100 particles/m^3	(Zhu et al., 2019)
North-western Pacific	Microscope, µ-Raman spectroscopy, SEM-EDX	Digestion (Fe + H_2O_2), density separation (NaCl) + filtration (0.2 µm)	< 5000	PE, PP, PA	1.0×10^4 particles km^{-2}	(Pan et al., 2019)
India, beach sediment	Microscope, µ-Raman spectroscopy, FESEM-EDX	Digestion (H_2O_2) + density separation (NaCl) + filtration (0.2 µm)	< 5000	PE and PP	664 ± 114 particles/kg	Present study
Atlantic Ocean	Microscope, FTIR	Filtration (GF)	250 - 5000	PES, PA, acrylic	Seawater: 1.15 ± 1.45 particles/m^3	(Kanhai et al., 2017)
Arctic Ocean, sea ice and seawater beneath ice floes	Microscope, FTIR	Filtration (GF)	100-5000	PVC, PA, polyester	Sea ice: 2 – 17 particles/L; Ice floes: 0 - 18 particles/m^3	(Kanhai et al., 2020)

1.3. Routes of exposure to human

Inhalation: Air

Air is an essential component of human survival. Breathing polluted air, on the other hand, is dangerous and lethal. Among the numerous pollutants in the air, airborne microplastics are newly identified by researchers. Dris et al. (2017) and Liu (2019) detected microplastic particles in the form of fibers and fragments in different regions like indoor, outdoor, and workplace atmospheres. Liu et al. (2019) and Trainic et al. (2020) identified the airborne microplastics in the West Pacific and North Atlantic Ocean (remote areas), respectively. As a result, these data demonstrate that microplastics are pervasive throughout the global environment because of their low density and small size, allowing for easy dispersion via wind. According to several observational studies, humans breathe in microplastics as they are prevalent in the atmosphere (Amato-Lourenço et al., 2021; Huang et al., 2022; Pauly et al., 1998).

Ingestion: Food, food products, and water

Microplastic has also been found in aquaculture/seafood sold for human consumption, including fish, bivalves (such as mussels and oysters), and crustaceans (such as brown shrimp) (Curren et al., 2020; Karami et al., 2018; Rochman et al., 2015; Thiele et al., 2021; Wootton et al., 2021). Meso-microplastic contamination has been observed in canned sardines, sprats, edible salts, and honey. Plastic particles in drinking water were originally reported by Orb Media in 2017. Later microplastics have been widely detected in surface and groundwater in urban, suburban, and remote areas (Eerkes-Medrano et al., 2019; Koelmans et al., 2019; Zhang et al., 2017). Microplastics present in the water from these sources may enter the drinking water supply for human consumption.

1.4. Average global exposure of microplastics to human

Air: The concentration of microplastics in the atmosphere might possibly be utilized to evaluate human's exposure to microplastics, particularly through dust consumption. In 1998 Pauly et al. examined 114 human lungs where fibres were present in 99 specimens (87 %). Amato-Lourenço et al. (2021) also detected microplastics (37 MPs) in 13 out of 20 human lung tissues during the autopsies. PE and PP were the frequently seen polymers (<17 µm)(Amato-Lourenço et al., 2021). Simplistic modeling has suggested that roughly 7,665 microplastic particles are ingested yearly by a person in Shanghai (assuming an adult consumes 15 m^3 of air each day) (Liu et al., 2019a). In Iran, children and adults are expected to expose or consume 3,223 and 1,063 MPs each year, respectively (Dehghani et al., 2017). In France and Turkey, per person, yearly inhalation exposure was 53,700 particles, with an average stated air concentration of 9. particles/m^3 (Cox et al., 2019; Kannan and Vimalkumar, 2021). The greatest exposure concentration (16.2 particles/m^3) was observed in Denmark, where the average male inhalation exposure is 88,695 MPs (272 MPs over 24 hours) (Vianello et al., 2019). Catarino et al. (2018) investigated the extent of household dust fibres that out during the preparing and eating of a food on the surface of a normal plate with a radius of 12.5 cm in Edinburg. This finding demonstrated that a significant amount of microplastics (13,731 and 68,415 MPs/y/person) are ingested through food. With predictable rates of indoor dust intake (2.2 - 41 mg/d), younger children are especially at risk of consumption of microplastics through dust and airborne fibres (Catarino et al., 2018; Dris et al., 2017). Urban dust dispersion can include up to 33% microplastics and identify as a possible source of microplastics for human exposure (Dris et al., 2017). A recent study on 22 patients suffering from respiratory diseases also revealed the presence of 21 types of microplastic (20-500µm) in human sputum. The identified

microplastic was in the size of 20-500 µm dominated by polyurethane (PU), polyester (PES), chlorinated polyethylene, and alkyd varnish (Huang et al., 2022).

Food and food products: Consumption of seafood is one of the pathways for human microplastic exposure. Plastic particles are frequently detected in high concentrations in the digestive systems of organisms such as bivalves and fish. Some studies, for example, have assessed annual human exposure to plastic particles from mussels, which are thought to be the most contaminated food. Van Cauwenberghe and Janssen (2014) and Li et al. (2015) estimated 11,000 microplastic particles per year per capita in European countries and >11,000 particles of microplastics per year per capita in China. According to Catarino et al. (2018) studies, in Belgium, France, and Spain, where shellfish consumption rates are greater (3.08 kg/y/capita), there are 4,620 microplastic particles per year per capita compared to 123 microplastic particles per year per person in the UK. Furthermore, Rochman et al. (2015) and Wootton et al. (2021) detected microplastics in commercially marketed wild-caught fish from Indonesia (28 % found in gastrointestinal tract, 55 % found in gut content), California, USA (25 % found in gastrointestinal tract, 67 % found in gut content), Australia (61.6 % seen in the gastrointestinal tract), and Fiji (35.3 % found in the gastrointestinal tract). Karami et al. (2017, 2018) purchased canned sardines and sprats' fish from 13 countries over 4 continents and observed 1 - 5 number of microplastics presence in dried canned fish. According to Karami, humans may absorb a maximum of 246 anthropogenic particles from dried fish every year. The annual consumption of microplastics from salt was estimated as a result of global analysis. Men and women consume roughly 5 g of salt every day, for a total intake of 1.825 kg per year. The existing research estimates that people consume 35 – 6,172 MPs/year from salt alone (Kim et al., 2018; Lee et al., 2021; Renzi and Blašković, 2018). With the recent rapid surge in plastic use, plastic

pollution may be expected to rise everywhere, especially in sea and lake salts. The risks to human health from microplastic ingestion via fish, bivalves, and crustaceans are low compared to exposure levels from home dust intake.

Water: According to the data currently available, drinking water is most common way route of oral ingestion of microplastics (Cox et al., 2019; Mortensen et al., 2021). Using the drinking water data (microplastics in treated and tap water data) and assuming that a child drinks 1 L of water per day, drinking water itself could lead to an annual intake of 0 - 2.60 x 10^5 MPs (Kirstein et al., 2021; Pittroff et al., 2021; Pivokonsky et al., 2020, 2018; Tong et al., 2020). An adult who drinks 3 L of water each day might consume between 0 – 7.83 x 10^5 particles/year. According to the findings of Kosuth et al. (2018), roughly 21,900 MPs are consumed yearly by Indian adults in New Delhi. Microplastics concentrations in mineral water bottled are much higher; 6292 ± 10,521 particles/L in glass bottles, 2,649 ± 2,857 particles/L in single-use plastic bottles, and 23,594 ± 25,518 particles/L in reusable bottles, which potentially resulting in severe inadvertent microplastic exposure (Oßmann et al., 2018a).

1.5. Possible impacts of microplastics on human health

Plastic products and fragments break down in the environment into macro-, micro-, and nanoplastic particles, which can adulterate the food chain by leaching chemicals and hazardous components (Ajith et al., 2020; Lebreton and Andrady, 2019). A recent study discovered microplastics in humans, confirming that plastic particles may pass in the human body via several pathways (Pauly et al., 1998; Rist et al., 2018; Schwabl et al., 2019), as shown in **Fig. 4**. Microplastics most enter the human body by ingestion, inhalation, and skin contact (G. Chen et al., 2020; Cox et al., 2019; Smith et al., 2018). Microplastics contaminated food products

and microplastics dust deposited in food are sources of gastrointestinal exposure, whereas suspended microplastics in the air for respiratory issues (Sana et al., 2020).

Depending on their size, microplastic/nanoplastic particles can enter our stomach. These plastics are either expelled, become lodged in the stomach and intestinal walls, or freely circulate in bodily fluids such as blood, reaching numerous organs and tissues (Rist et al., 2018; Schwabl et al., 2019; Q. Zhang et al., 2020). The study conducted by Schwabl et al. (2019) also revealed the existence of microplastic particles in the feces of individuals from eight different nations. Endocrine disruptor chemicals (EDCs), such as bisphenol A (Fu et al., 2018), are reported in tissue samples, human blood, breast milk and urine (Rist et al., 2019, 2018; Talsness et al., 2009) which possibly disruptor humans endocrine system (Ma et al., 2019b; Verma and Subbiah, 2019; Zimmermann et al., 2019). It is indeed uncertain how much exposure to airborne microplastics endangers public health. Although the visible fibres are allegedly too big to be inhaled, however, exposure may happen by dust intake, predominantly in young infants (Gasperi et al., 2018; Wright and Kelly, 2017). The diameter of the fibre is important for its breathability, while the length is important for its persistence and toxicity. Particles with tiny fibres can enter the lungs and finally penetrate deep inside the lung. Fibres can accumulate in terminal bronchioles, alveolar ducts, and alveoli, causing fibrosis, persistent inflammation and granulomas. Pauly et al. (1998) found polymeric and cellulosic fibres in human lung tissue (cancer patients). Among those, the inhaled fibres were more (97%) in malignant lung specimens. According to Greim et al. (2001), the contact between cells and particles/fibres can promote inflammation, which in turn causes cell growth and secondary genotoxicity owing to the constant generation of reactive oxygen species (ROS). ROS overproduction causes oxidative stress, which causes persistent inflammation and contributes to the pathophysiology

of lung illnesses. While other research (associated to the textile sector) have found a higher cancer risk as a result of exposure to synthetic fibre after 10–20 years. Workers in the flocking, plastic processing and synthetic textile industries are more likely to be exposed to MFs (Prata, 2018). These bio-resistive and bio-permanent polymer fibres may contribute to the risk of lung cancer. The severity of tissue damage is generally proportional to the amount of microplastics breathed over time (G. Chen et al., 2020). Other pathways of exposure to airborne microplastics could emerge through the skin (dermal). According to Stapleton (2021), using personal special care products and being exposed to aerosolized particles are likely additional sources of skin exposure to nanosized particles. Micro/nanoplastics will penetrate the healthy dermal barrier and enter the body if a skin is damaged by minor rips, rashes, sunburn. Additionally, it has been demonstrated that the mechanical wear of medical devices inserted into the body, such as cosmetic implants, dental caps and PE articulating spacers used in shoulder, knee or hip replacements, enables the production and translocation of micro- and nanoplastics within the system (Stapleton, 2021). According to Prata et al. (2018), greater exposure occurs in indoor surroundings since most people spend the majority of their time (70-90 percent) indoors. Vianello et al. (2019) reported that in their recent simulated experiment using a breathing thermal manikin to determine people's exposure to indoor airborne microplastics, the manikin ingested a total of 272 microplastics over the course of 24 hours.

In vitro studies on the toxicity of microplastics to human health are possible (Ballesteros et al., 2020; Çobanoğlu et al., 2021; Gopinath et al., 2019; Schirinzi et al., 2017). Ballesteros et al. (2020) found that PS nanoparticle exposure increased DNA damage in human monocytes and polymorphonuclear cells. The research by Obanolu et al., (2021) on PE microplastics revealed that even modest levels of concentration of microplastics raised the amount of genetic

instability, which may lead to illnesses, most notably cancer. According to Gopinath et al. (2019) research on the interaction of nanoplastics with blood plasma, blood proteins are absorbed on the surface of nanoplastics and form plastic-protein complexes (13 to 600 nm). The aggregation of these plastic-protein complexes might obstruct the flow of bodily fluids. Nano-plastics can potentially disrupt mitotic activity and induce cytotoxicity in human and animal cells. Human cells (alveolar A549) exposed to PS microplastics (1 and 10 μm dia) demonstrated a substantial reduction in cell proliferation, a decrease in cell metabolic activity, and a change in cell architecture and behavior (loss in cell to cell contact)(Goodman et al., 2021). Exposure to two human cell lines (T98G and HeLa, epithelial and cerebral cells, respectively) to PE and PS microplastic particles showed oxidative stress as one of the cell-level causes of cytotoxicity. Other biological responses that might occur include genotoxicity, apoptosis, and necrosis, all of which could result in tissue damage (Galloway, 2015; Grafmueller et al., 2015; Schirinzi et al., 2017).

The transfer and deposition of micro and nanoplastic particles also affects the reproductive system. From the original site of exposure, micro and nanoplastics (PS) have moved to the testes, ovaries, and placenta. According to studies, ovarian micro PS uptake decreased follicular growth and brought on oxidative stress, which aided in the development of ovarian fibrosis (Stapleton, 2021). Rescently, Ragusa et al. (2021) observed the man-made pigmented polymers in human placenta (~70% of women had microplastics) following real-world exposure during pregnancy. According to Galloway (2015), the first study to detect the real-world nanoplastic translocation in the human placenta. Both research studies demonstrate the possibility of exposure in human and systematic bioaccumulation, even though it was unknown how these particles reached the maternal system via ingestion or breathing pathways.

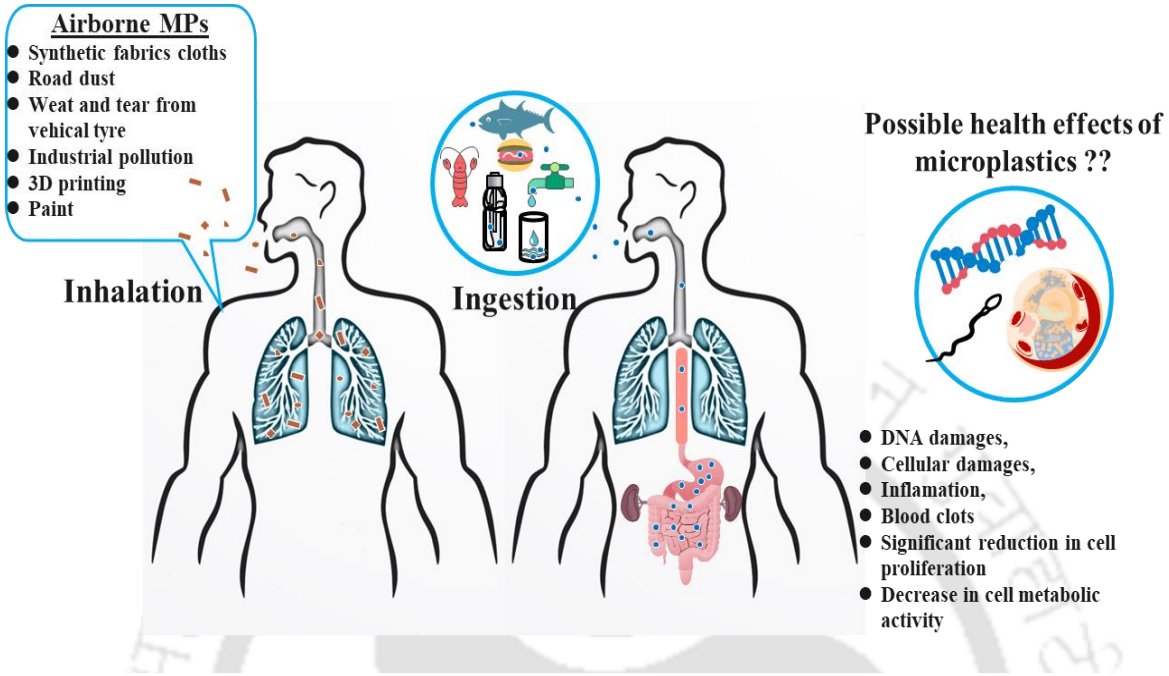


Figure 4: Exposure pathways and possible health effects of microplastics to humans.

1.6. Motivation

Microplastics are ubiquitous in the environment, however, knowledge of sampling methodology and concentration of microplastics in atmospheric, terrestrial, and aquatic regions is minimal. Therefore, the inclusion of alternative standard sampling methodology and more studies may facilitate the comparison of microplastics distribution in various environments. The development of new technologies and resources for the alternative materials to replace and mitigate plastic items is necessary. Further, the implementation of effective plans and technologies for solid and sludge waste management towards microplastics fate, degradation, and utilization is equally essential. The findings raise concerns about the consumption of microplastics by humans through air, water and aquatic food and food products, in addition to potential consequences for human health. In view of these issues, study was prompted by the

need to: 1) identify the sources of microplastics in salt, drinking water and marine environment, 2) develop new technologies to prevent microplastic pollution in the environment. The studies in the subsequent sections will provide detailed data on microplastics in different environments, which may be helpful in the futuristic plan for sustainable management and protection of environment and healthy ecosystem.

Chapter 2

2. Literature review and Objectives

Preface: *This chapter is aimed to provide a detailed literature survey about the presence of microplastics in freshwater, tap water, marine environment, and in edible salts. Furthermore, an in-depth literature study was carried out on laundry wastewater, and removal of microplastics and surfactant pollutants from the same using electrocoagulation method for the reutilization of treated water. From the outcome of the literature review, the prominent research gaps were identified to frame the objectives of the present research work at the end of this chapter.*

Plastics are prepared by adding several chemicals such as Bisphenol A, phthalates, and phenol, which are categorized under (EDCs) and these type of microplastics were found in human blood, tissues and in organs (Rist et al., 2018). These microplastics directly affect the human reproductive and immune system and also increase the incidence of cancer (Ma et al., 2019a; Verma and Subbiah, 2019; Zimmermann et al., 2019). Various reports from different countries suggested that maximum microplastics can reach humans through drinking water and edible salts, and seafood (Jan Kole et al., 2017; Kühn et al., 2015; Oßmann et al., 2018b; Wright and Kelly, 2017). The microplastics pollution in freshwater and marine ecosystem from Indian subcontinent remains largely unexplored (Sruthy and Ramasamy, 2017). Therefore, it is very important to know the microplastics concentration in freshwater and marine environment. Because, human health is directly linked to the quality of drinking water, marine food and sea salt.

2.1. Presence of microplastics in fresh water and tap water

The detrimental effects of microplastics on freshwater and marine life have recently raised awareness of microplastic pollution in water resources, particularly drinking water supplies (Sruthy and Ramasamy, 2017). These concern has spread to the water supply, and drinking water as microplastics are persistent, buoyant, and capable of absorbing other toxic pollutants such as cadmium, mercury, and polychlorinatetabd biphenyls (Richard et al., 2019; Selvam et al., 2021; Tu et al., 2020). Microplastics have been increasingly documented globally in numerous environmental compartments. L. Yang, Luo, et al. (2021) studied the Koshi River in Nepal for the presence of microplastics, and their findings showed that urbanisation, agriculture, transportation, and tourism were the main contributors. Detained works about microplastics sources and pathways were explained in **Section 1.2.3**. The existence of

microplastics contamination in freshwater from various countries is showed in **Table 4.** However, despite the fact that European rivers have significant plastic pollution levels, Asia is the region that contributes the majority of the world's marine microplastics. According to a recently published worldwide model, 1.15 to 2.41 million tonnes of plastic have already entering the seas through the river line system each year. This estimate was made using geographic data on waste management, population density, and hydrology. Asian's China Rivers (Xiang Jiang River and Yangtze river) and Philippines Rivers are considered to be among the major sources of plastic waste in the oceans (Bellasi et al., 2020; Osorio et al., 2021; Shen et al., 2021b; Zhao et al., 2014). There are very few studies examining microplastics in freshwater and sediments of Indian rivers (Ajay et al., 2021; Lechthaler et al., 2021; Napper et al., 2021; Singh et al., 2021). Ajay et al. (2021), was the first author to check the microplastics and phthalate esters levels in the water and sediment of the inland aquatic system in Indian Himalaya lake. This lake water had average concentration of 2,000 to 64,000 particles/m^3 and in sediments 180 ± 143 particles/kg. According to a study by Napper et al. (2021), the Ganges may discharge up to 1-3 billion microplastics daily into the Bay of Bengal (Indian Ocean). The yearly microplastic particles input via the Adyar River into the Bay of Bengal was estimated by Lechthaler et al. (2021) to be 11.6 trillion, which is alarming and substantially greater than the input number for the Ganges as reported by Napper et al. (2021). A huge amount of emphasis is placed on knowing the load of microplastics transported by the surface-water system to oceans by the fact that approximately 70 % of Indian rivers flow into the Bay of Bengal, which also includes the Brahmaputra River as one of the major rivers. But there is limited research on microplastic pollution in the Brahmaputra River. Currently, no published data are available on the combination of microplastic contamination in Brahmaputra River and tap water. The main barriers to microplastics entering the human body are drinking water

treatment plants (DWTPs). Now, it is interesting to note that just a small number of studies examining the presence of microplastics in DWTPs (raw water/fresh water and tap water/drinking water) have been carried out globally, with a typical removal efficiency of 50 - 80 % (Dalmau-Soler et al., 2021; Mintenig et al., 2019; Pittroff et al., 2021; Pivokonsky et al., 2020, 2018; Shen et al., 2021b). The remaining microplastic particles reach the human body through tap water. As reported by Kirstein et al. (2021), Kosuth et al. (2018), Pittroff et al. (2021), Strand et al. (2018), the concentration of microplastics in tap water was found in Italy, Germany, Sweden, Denmark, Lebanon, and USA in the range of 910 – 9, 240 particles/m^3, whereas studies showed that concentration of microplastics in Tap water in Delhi, India was found to be 6,240 particles/m^3 (Kosuth et al., 2018). Microplastics in drinking water pose implications on human health. To avoid this, it is necessary to remove microplastic particles from tap water. However, to the best of our knowledge, no reports on the removal of microplastics in tap water are available.

Table 4. Worldwide concentration of microplastics in freshwater.

Type of sample	Location	MPs concentration	Reference
Ground water (well)	Punnakayal, Tamil Nadu, India	0 – 4,300 particles/m^3	(Selvam et al., 2021)
Ground water supply chain	Oldenburg, Germany	0 – 30 particles/m^3	(Mintenig et al., 2019)
Lake water	Renuka lake Himachal Pradesh, India	2,000 to 64,000 particles/m^3	(Ajay et al., 2021)
Lake water	Red Hills Lake, Tamil Nadu, India	5,900 particles/m^3	(Gopinath et al., 2020)
River and reservoir	Czech Republic	1473 ± 34 - 3605 ± 497 $\times 10^3$ particles/m^3	(Pivokonsky et al., 2018)
River water	Meuse and Dommel River, Belgium, Netherland	67-11,500 particles/m^3	(Mintenig et al., 2020)
River water	Koshi River, Nepal	227 ± 85 particles/m^3	(Yang et al., 2021a)
River water	West River, China	2,990 to 9,870 particles/m^3	(Huang et al., 2021)
River water	Five Rivers, Philippines	1.58 to 57.66 particles/m^3	(Osorio et al., 2021)
River water	Xiang Jiang River, China	2,173-3,998 $\times 10^3$ particles/m^3	(Shen et al., 2021b)
River water	Llobregat River basin, Catalonia, Spain	3,600 particles/m^3	(Dalmau-Soler et al., 2021)
River water	Chao Phraya and Maeklong River, Thailand	400 – 2,400 particles/m^3	(Chanpiwat and Damrongsiri, 2021)

Type of sample	Location	MPs concentration	Reference
River water	Ganga River in India and Bhangladesh	38 ± 4 particles/m^3	(Napper et al., 2021)
River water	Adyar River, Tamil Nadu, India	330 particles/m^3	(Lechthaler et al., 2021)
	Kosasthalaiyar River, Tamil Nadu, India	670 particles/m^3	
	Muthirappuzhayar River, Kerala, India	200 particles/m^3	
DWTPs (after treatment)	Czech Republic	$338 \pm 76 \times 10^3$ to $628 \pm 28 \times 10^3$ particles/m^3	(Pivokonsky et al., 2018)
Tap water	Denmark	580 particles/m^3	(Strand et al., 2018)
Tap water	Different cities of China	440 ± 275 particles/m^3	(Tong et al., 2020)
Tap water	Bangkok, Thailand	430 – 1,520 particles/m^3	(Chanpiwat and Damrongsiri, 2021)
Tap water	Baden-Wurttemberg, Germany	6 -74 particles/m^3	(Pittroff et al., 2021)
Tap water	Hunan province, China	267 - 404 $\times 10^3$ particles/m^3	(Shen et al., 2021b)
Tap water	Delhi, India	6,240 particles/m^3	(Kosuth et al., 2018)

2.2. Presence of microplastics in marine environment

Reliable estimates of the amount of plastic waste that enters the aquatic environment cannot be obtained, but the amounts are nevertheless quite substantial (Derraik, 2002). Globally, plastic pollution was increased 8.8 million tones of plastic waste entering the oceans from coastal communities every year. India has over 7,500 km of a long coastline, 9 coastal states it includes more than 60 districts, and one-third of the population lives in coastal areas which dumps nearly 0.6 million tons of plastic waste into the ocean (Jayasiri et al., 2013; Sathish et al., 2019). For the last four decades, the deposition of both macro and microplastics has consistently increased on marine water, seashores and sediments (Nanthini Devi et al., 2023). The accumulation of microplastics along the Indian coastline has not yet attracted the amount of publicity it deserves, considering the widespread knowledge of the harmful existence of plastics. There have been very few studies on microplastics related to India's coastal environment as shown in **Table 5**. Most of the studies are emphasized on microplastics in beach sediments. However, it is envisaged from **Table 5**, that Robin et al. (2020), is the only author who has reported the study on microplastics in marine water (1.25 ± 0.88 particles/m^3). More number of microplastics are observed in the entire southeast coast of India. It has been noted that east coast of Tamil Nadu has a larger accumulation of plastic debris than west coast of Kerala, perhaps as a result of the vast beach geomorphological settings of the terrain, which play a vital effect in the dispersal of plastic waste. This is because many rivers which flow from west to east carry a lot of plastic waste to the east coast. Additionally, the southeastern coast is sandier, broader, and

predominant industry of fishing, which is surrounded by well-irrigated paddy land and numerous ageing textile industries. (Neelavannan et al., 2022).

Sea food and sea salt is an important component in the human diet, and the abundance of microplastics in the sea food and sea salt poses a serious threat to the human health. Various studies have demonstrated the presence of microplastics in the edible salt, fish, shrimps, and prawns, and through which microplastics enter the human systems as a result of biomagnifications (Ajith et al., 2020; Li et al., 2021). Therefore, it is very important to know the microplastics concentration in costal environment. As per our knowledge there are no studies related to microplastics in the Karnataka coastal region. Therefore, an attempt was made on investigating microplastics with respect to distribution and characterization in Karnataka, Indian coastal environments.

Table 5. Microplastics concentration along the Indian coastal environment.

Seawater/Beach sediment and location	Number of microplastics (n)	MPs Size	Polymer %							Reference
			PE	PP	PA	PS	PET	PVC	Others	
Tamil Nadu, southeast coast of India	46.6 ± 37.2 particles /m^2	0.3 – 4.75 mm	46	19.50	10.49	17.5	-	-	6.69	(Karthik et al., 2018)
Rameswaram Coral Island, Gulf of Mannar, Southeast coast of India	403 particles/kg	1.01- 4.75 mm	17.9	39.7	14.6	15.6	-	12.2	-	(Vidyasakar et al., 2018)
Chennai, southeast coast of India	309±184 particles/kg	0.5 – 3 mm	81	16	-	3	-	-	-	(Sathish et al., 2019)
Mumbai, Tuticorin, Dhanush kodi coast, India	220 ± 50, 181 ± 60 and 45 ± 12 particles/kg	36 µm to 5 mm	43	12.3	-	17	17.3	1.33	11	(Tiwari et al., 2019)
Puducherry coast, India	720 ± 191 particles/kg	0.3 – 5 mm	-	-	-	-	-	-	-	(Dowarah and Devipriya, 2019)
Kerala, southwest coast of India	1.25 ± 0.88 particles /m^3 and 40.7 ± 33.2 particles /m^2	0.3 – 4.75 mm	43	22.54	11.27	9.22	3.07	0.61	10.25	(Robin et al., 2020)

Seawater/Beach sediment and location	Number of microplastics (n)	MPs Size	Polymer %							Reference
			PE	PP	PA	PS	PET	PVC	Others	
Silver beach, Tamil Nadu, Southern India	204 particles/kg	-	14	-	7	-	-	79	-	(Vidyasakar et al., 2020)
Andaman coastal, India	414.35 ± 87.4 particles /kg	0.3- 5 mm	-	-	-	-	-	-	-	(Patchaiyappan et al., 2020)
Andaman and Nicobar Archipelago, India	1900 particles/kg	0.45 µm – 2 mm	28.39	46.40	-	8.92	3.88	2.09	10.32	(S. et al., 2020)

2.3. Presence of microplastics in edible salt

India is the third-largest producer of sea salt. Approximately 24 million tonnes of raw salt are produced in Indian each year, with 20% exported mostly to China, Japan, Indonesia, and the United States. Various authors across the globe reported the presence of microplastics in edible salts as shown in **Table 6**. The presence of these plastic particles in salt is mainly due to contaminated seawater (Karthik et al., 2018; Lee et al., 2019, 2021; Seth and Shriwastav, 2018), and some microplastics may have reached during salt production through airborne (Kosuth et al., 2018; Seth and Shriwastav, 2018; Yang et al., 2015). Iñiguez et al. (2017), Kosuth et al. (2018), Gündoğdu, (2018) and Selvam et al. (2020), observed the more dominance of fibers type microplastics in edible salts. This may be because of laundry water from domestic and textile industries or Municipal wastewater effluents. It is essential to understand where the fibers in table salt actually come from. According to Iñiguez et al. (2017) and Rist et al. (2018) the estimated consumption of microplastics through salt was 510 and 37 ~ 1000 particles per year respectively, which pose a higher risk to the human health. However, the level of contamination of Indian sea salts with microplastics is not known. Further, to the best of our knowledge, no information on potential ways to prevent contamination of the produced salt exists. This is a crucial gap since polluted seawater is still used for salt manufacturing across the world. It is critical to clean the water used for salt manufacturing in order to potentially reduce the substantial quantities of microplastics. We evaluate the level of microplastic pollution in Indian sea salts in this work and provide a feasible preventative approach.

Table 6. Comparison with previous literatures.

References	Number of particles /kg		Composition, %								
	Sea salt	Well/Rock salt/	PE	PET	PP	PVC	Nylon	PU	PS	Pigment	Unidentified
Yang et al. 2015	550 - 681	7-204	8.5	16.3	4.7	0.8	-	-	-	-	-
Iñiguez et al. 2017	50 - 280	115-185	3.3	83.3	6.7	-	-	-	-	-	-
Karami et al. 2017	1- 10	1-10	13.8	2.8	16.6	-	1.4	-	2.8	23.6	29.1
Kim et.al 2018	0 - 1674	0-148	22.9	26.9	20	-	-	-	-	-	15.4
Kosuth et al., 2018	46.7 - 806	113 to 367	-	-	-	-	-	-	-	-	-
Gündoğdu, 2018	16-84	9-16	12.5	18.8	18.8	12.5	12.5	25	-	-	-
Seth and Shriwastav, 2018	56 ± 49 - 103 ± 39	-	22	61	-	-	16	-	-	-	-
Renzi and Blašković, 2018	1,600 - 19,800	-	-	-	-	-	-	-	-	-	-
Lee et al., 2019	0 - 10	-	34.9	2.3	39.5	-	-	-	14	-	6.6
Lee et al.,2020	98 - 2,395	-	25	4	60	3	-	3	-	-	-

2.4. Laundry wastewater

Domestic laundry wastewater is a kind of greywater. The report estimates that there are 590 million washing machines in 38 countries, and the average yearly water use of each washing machine is approximately 10 m^3 (Luo et al., 2022; Pakula and Stamminger, 2010). If such a huge volume of laundry effluent is released straight into the environment without treatment, it will cause an enormous impact on the ecosystem. Direct discharge of laundry wastewater may not only cause eutrophication but may also impair the functioning of WWTPs. The majority of laundry effluent enters the wastewater treatment facility directly via municipal wastewater piping, which, if reused after treatment, can conserve water resources. Laundry activities generate a large amount of wastewater high in lint, dye, oils, fats, suspended particles, surfactants, and microplastics (Kruszelnicka et al., 2019; Oktiawan et al., 2021; Santiago et al., 2021). Surfactants and microplastics are two common contaminants in laundry effluent that can affect the ecosystem. Surfactants have been shown in studies to not only harm the morphology and physiology of plants but also to significantly reduce sludge dewaterability (Badmus et al., 2021; Kaleta and Elektorowicz, 2013; Luo et al., 2022). The substantial majority of research data suggests that a significant source of microplastics (in the form of microfibers: size is less than 5 mm) in the freshwater system is from laundry outlets (Belzagui et al., 2019; De Falco et al., 2019, 2018; Galvão et al., 2020). **Table 7** shows the various literatus on synthetic microfibers observed in laundry wastewater. For instance, De Falco et al. (2018), Belzagui et al. (2019), and Galvão et al. (2020) determined that laundry outlets released MFs at rates of 1.75×10^5 - 5.6×10^5 MFs/kg, 12×10^5 MFs/kg and 30×10^5 MFs/kg of cloth loads respectively. A significant accumulation of microplastics and surfactants along with their degraded products in aquatic environments resulted in frequent exposure to living species,

causing an ecological imbalance (Elizalde-Velázquez and Gómez-Oliván, 2021; Tripathi et al., 2013). Till date, the majority of research has focused on surfactant concentration, microplastic sources, distributions, estimates, and hazardous consequences (Belzagui et al., 2019; De Falco et al., 2019; Galvão et al., 2020; Li et al., 2022; Luo et al., 2022; Patil et al., 2020; Ramcharan and Bissessur, 2017). Globally, the removal of microplastics and surfactants from laundry outlets is a significant problem as well as a challenging task. There is an immediate need to take steps to eliminate these contaminants from laundry effluent.

Several literatures on the treatment of laundry wastewater based on various methodologies have been reported shows in **Table 8**. Most often used methods include adsorption, chemical coagulation, biological, membrane filtration, electrocoagulation, and sand filtration or a combination of these techniques (Patil et al., 2020; Santiago et al., 2021; Šostar-Turk et al., 2005). In spite of satisfactory results, each process has its own drawbacks. For example, adsorption is a pH-dependent process that requires longer treatment time, decreases adsorption capacity as the number of cycles increases, and requires high energy for regeneration. Similarly, the chemical coagulation approach necessitates the use of several chemicals. It cannot form flocs across a larger pH range. The pH must be regulated during the process, with the addition of acids and various coagulants. During the coagulation-precipitation process, a significant number of secondary pollutants, such as chloride and sulphates, are produced, as well as a large amount of sludge is also produced, which causes serious environmental issues (Das et al., 2021a). In biological treatment, a large amount of space and prolonged time is required for effluent treatment (Ramcharan and Bissessur, 2017). When it comes to membrane filtration, it causes fouling which has a negative influence and reduces permeate flow. In this method, equipment and cleaning costs are expensive (Deepti et al., 2021; Šostar-Turk et al.,

2005). The sand filtration technique is an easy and alluring option for wastewater treatment. However, it is reported that microplastics can easily escape from sand filtration media in the presence of surfactants (Jiang et al., 2021). As a result, there is an urgent need for more reliable and cost-effective technologies that use fewer chemicals, consume less energy, and have a high capacity for pollutant removal. Electrocoagulation has gained widespread acceptance for pollutant removal from industrial and municipal wastewater. The reason could include the simple equipment design, ease of operation, rapid reaction rate, integration with various treatment techniques, and low operating costs (Dimoglo et al., 2019; Priya and Jeyanthi, 2019).

Various researchers have also studied this process's effectiveness in removing microplastics and surfactants from multiple sources. Shen et al. (2022) reported the removal of microplastics from synthetic wastewater. The findings indicate that aluminium anodes performed better than iron anodes in terms of removal rate, which was over 90%. Perren et al. (2018) reported the removal of microbeads from synthetic wastewater using electrocoagulation with removal efficiency ranging from 90 to 99%. Similarly to this, Elkhatib et al. (2021) collected final effluent from municipal wastewater and incorporated the synthesized microplastics. The finding indicated that 96.5% of microplastics were removed using the aluminum electrode from electrocoagulation. Oktiawan et al. (2021), the study focused on the removal of surfactants using electrocoagulation. Study results revealed that the efficiency of removal of surfactants was higher in aluminum electrodes when compared to iron electrodes. From the literature, it is envisaged that electrocoagulation with the aluminum electrode is best suited for eliminating microplastics and surfactants from wastewater (Elkhatib et al., 2021; Oktiawan et al., 2021; Shen et al., 2022). Therefore, to the best of our knowledge, the

electrocoagulation technique has not been much investigated for the treatment of laundry wastewater especially focusing on MFs and surfactants.

Table 7. Various literatus on synthetic microfibers observed in laundry wastewater.

Type of fabric	New clothes	Synthetic MFs	MFs length	Surfactant concentration	Reference
Commercial garments (PES, PP)	Yes	12×10^5 to 35.40×10^5 MFs/kg	20 to 2,000 µm	n.d	(De Falco et al., 2018)
Commercial garments (polyester)	Yes	6.40×10^5 to 15×10^5 MFs/kg	Avg 360 to 660 µm	n.d	(De Falco et al., 2019)
Textile garments (Polyester, polyester-elastane and polyamide-elastane)	Yes	30,000 to 4,65, 000 MFs/m^2	20 to 5,000 µm (Avg 0.2 to 0.4 mm)	n.d	(Belzagui et al., 2019)
Household clothes and linens (Cotton, PES, PA, viscose, elastane, acrylic)	No	30×10^5 MFs/kg	0.17 mm (50 to > 500 µm)	n.d	(Galvão et al., 2020)

Type of fabric	New clothes	Synthetic MFs	MFs length	Surfactant concentration	Reference
PES, PA, and poly acetate fabrics	Yes	74,816 ± 10,656 MFs/m^2	5 to 4000 µm (avg PES: 499.49 ± 505.65 µm, PA: 1056.53 ± 761.42 µm, acetate: 1128.00 ± 750.72 µm	n.d	(Yang et al., 2019)
Household clothes	No	4,400 to 10,800 MFs/L	6 to 4000 µm	8 ± 2 to 800 ± 50 (mg/L)	(Luo et al., 2022)

Avg., average length; n.d, not determined

Table 8. Removal of microfibers and surfactant in various wastewater using the electrocoagulation process.

Type of wastewater	Pollutants	Treatment method	Electrode	Removal efficiency	References
Domestic wastewater (PE, PVC)	Microplastics	Electrocoagulation-electroflotation and membrane filtration	Aluminum and iron	Microplastics 100 % (Electrocoagulation and membrane)	(Akarsu et al., 2021)
Effluent from the local wastewater treatment plant	Polyester microplastics (25 mg/L) were added	Electrocoagulation	Aluminum	Microplastics 96.5 %	(Elkhatib et al., 2021)
Synthetic wastewater	Microplastics (PE, PMMA, CA, PP)	Electrocoagulation	Aluminum and From the literature iron	For Al: PE 93.2%, PMMA 91.7%, CA 98.2%, PP 98.4%. For Fe: PE 71.6%, PMMA 58.6%, CA 85.4%, PP 82.7%.	(Shen et al., 2022)

Type of wastewater	Pollutants	Treatment method	Electrode	Removal efficiency	References
Secondary effluent of the sewage treatment plant	PE, microplastics (25 mg/L) were added	Electrocoagulation	Aluminum	Surfactants 97.5%	(Xu et al., 2022)
Laundry wastewater	Surfactants	Electrocoagulation	Aluminum	Surfactants 80%	(Ramcharan and Bissessur, 2017)
Laundry wastewater	Surfactants	Electrocoagulation/ electroflotation	Aluminum	Surfactants 90%	(Dimoglo et al., 2019)
Laundry wastewater	Surfactants	Electrocoagulation	Aluminum and iron (Al-Al, Fe-Fe, Al-Fe, and Fe-Al	72.89% in Al-Al, 54.33% in Fe-Fe, 62.70% in Al-Fe, and 49.01% in Fe-Al	(Oktiawan et al., 2021)

2.5. Research Gaps

1. Microplastics in drinking water pose implications on human health. Now, it is interesting to note that just a limited number of studies examining the presence of microplastics in raw water/fresh water and tap water/drinking water have been carried out. Currently, no published data are available on the combination of microplastic contamination in Brahmaputra River and tap water.

2. Various studies have demonstrated the presence of microplastics in the edible salt, fish, shrimps, and prawns, and through which microplastics enter the human systems as a result of biomagnifications. Therefore, it is very important to know the microplastics concentration in costal environment. There were no studies reported related to microplastics with respect to distribution and characterization in Karnataka coastal region.

3. Further, the level of contamination of Indian sea salts with microplastics is not known. This is a crucial gap since polluted seawater is still used for salt manufacturing across the world. It is essential to clean the water used for salt manufacturing in order to potentially reduce the substantial quantities of microplastics.

4. Surfactants and microplastics are two common contaminants in laundry effluent that can affect the ecosystem. The substantial majority of research data suggests that a significant source of microfibers in the freshwater system is from laundry outlets. However, studies for the treatment of laundry wastewater especially focusing on microfibers and surfactants are scant. Therefore, it is necessary to develop an effcient treatment technique for the removal of microfibers from the laundry wastewater.

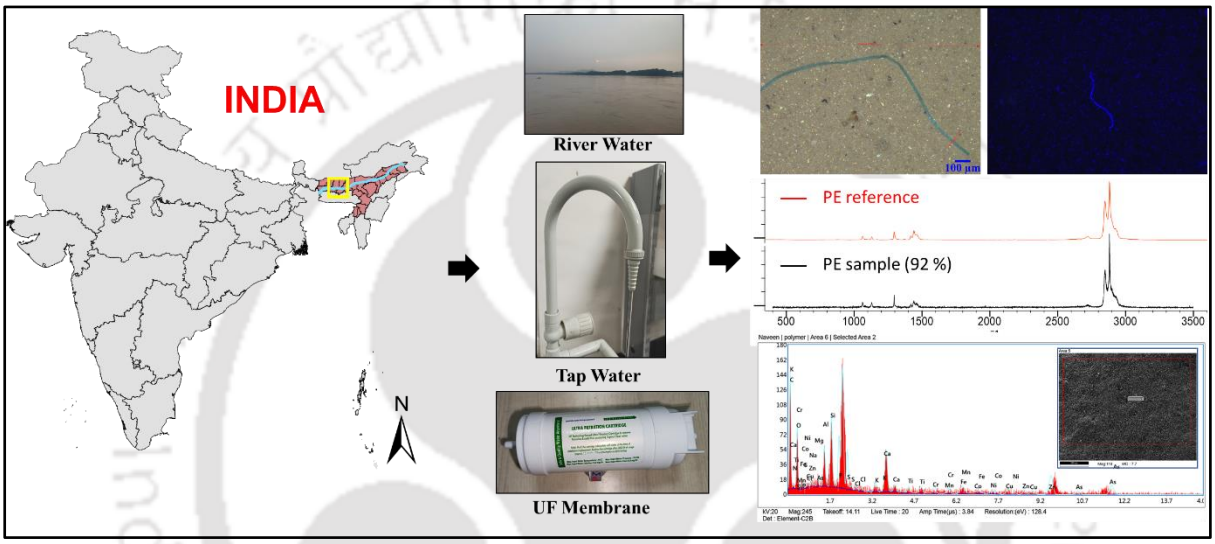
2.6. Objectives

From the above research gaps, the following objectives are derived for doctoral thesis:

1. Presence and detection of microplastics (qualitative study) in:
    1.1 Fresh water (Brahmaputra River water and tap water)
    1.2 Indian seawater and beach sediments
    1.3 Edible salts
    1.4 Laundry effluent
2. Develop a proper protocol for quantitative detection of microplastics in aquatic streams, beach sediments, edible salts, and laundry effluent.
3. Remediation technique to remove microplastics:
    3.1. Separation of microplastics from seawater water by hollow fiber microfiltration membranes for the production of microplastic free salt
    3.2. Separation of microplastics from drinking water with ultrafiltration membranes
    3.3.Separation of microplastics and surfactants from laundry effluent by using electrocoagulation method.

Chapter 3

3. Microplastics from the river to tap water: occurrence and removal from tap water



***Preface**: In this chapter, Analysis of water samples from a river, a tap, and a domestic purifier was done to determine the microplastics abundance, chemical composition, and selective elemental composition. In addition, the removal of microplastics from tap water was evaluated using commercially available hollow fibre ultrafiltration (HF-UF) membranes. The overarching goal of this chapter was to assess the quality of drinking water in terms of microplastic pollution in order to ensure human safety.*

3.1. Materials and methodology

3.1.1. Study area and sample collection

The Brahmaputra River originates in Tibet and is the longest river in the Himalayan region. The Brahmaputra river enters states such as Arunachal Pradesh and Assam in India, then continues to flow in Bangladesh (Tsering et al., 2021). It is an essential river for local transportation, agriculture, and drinking purposes. The maximum depth of the river is 135 m (440 ft, at Sadiya, Assam), while its average depth is 30 m (100 ft). One of India's most rapidly expanding towns, Guwahati, is situated between the Brahmaputra River banks. Guwahati had a population of approximately 9,62,334 in 2011 (Census 2011); it comes under the Kamrup district. The river water samples were collected in seven locations: four near Guwahati city (R1, R2, R3, and R4) and three in North Guwahati (R5, R6, and R7). River water sample location and site characteristics are shown in **Table 9**. Three river water samples were collected near the inlet of DWTPs in the Kamrup district at R2, R4, and R6. Tap water samples were collected from six locations: T1 and T2 (DWTP1 outlet), T3 and T4 (DWTP2 outlet), and T5 and T6 (DWTP3 outlet). The DWTPs are not named for reasons of confidentiality. The water treatment plant selected for present study employs technologies such as aeration, coagulation and flocculation, sedimentation, sand filtration, and then chlorination treatment. The locations of 13 sampling points in the study area were obtained by using a handheld GPS instrument. The ArcGIS 10.5 software and google map were used to map the geographic location and sampling sites (**Fig. 5**).

Table 9. River water sample location and site characteristics.

River samples	Location	Latitude and longitude	Site Description
R1	Cunchali picnic spot	26°12'24.0"N 91°47'59.3"E	Picnic spot
R2	Uzan bazar Ghat	26°11'42.8"N 91°45'18.9"E	High population density area, fisheries activity, traffic area
R3	Bharalumukh/Fancy Bazar	26°10'30.5"N 91°43'46.2"E	High population density area, market, and dense traffic area
R4	Pandu Ghat	26°10'19.9"N 91°41'08.5"E	Moderately populated fishing and port area
R5	Madhyam khanda	26°12'04.3"N 91°44'07.1"E	Less populated area
R6	Brahmaputra Ghat	26°10'53.4"N 91°41'48.0"E	Less populated area
R7	Amingaon	26°10'51.1"N 91°40'05.5"E	Moderately populated, industrial and fishing activity area

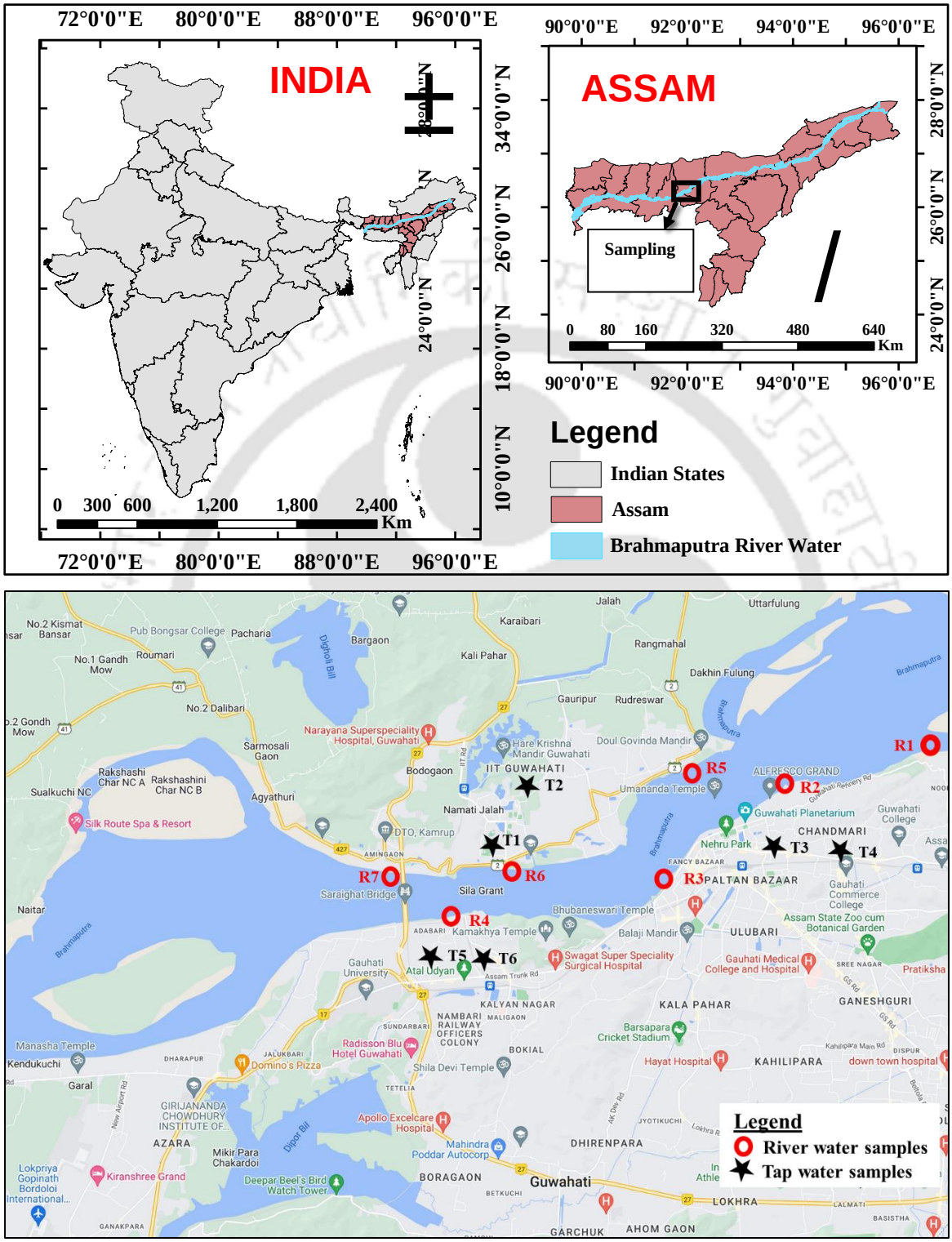


Figure 5. Overview map of all river, tap water sampling sites in Kamrup district, Assam,

3.1.2. Sample processing

River water samples were collected in March and April 2022. Tap water samples were collected in the dry season (months of March and April) and wet season (months of June and July) in 2022. Water samples (river and tap water) were run through metal sieves (5, 3.3, 2, and 1 mm), rinsed with distilled water (DW) on the surface of the sieves, and separated into different fractions in the laboratory. To avoid contamination, all successive sample preparation steps in the laboratory were carried out in a laminar flow box. Plastic particles larger than 5 mm in size were discarded. The samples were digested with hydrogen peroxide (H_2O_2, 30%) to remove the organic matter deposited to the microplastics surface. The solution was then filtered through nylon/PCTE membranes (0.2 µm pore size, 47 mm dia) using a vacuum filtration setup **(Fig. 6)**. The filtered samples were dried at 40 °C for two days and stored in glass Petri dishes. Further analysis was carried out. Three procedural blanks were used during the extraction and

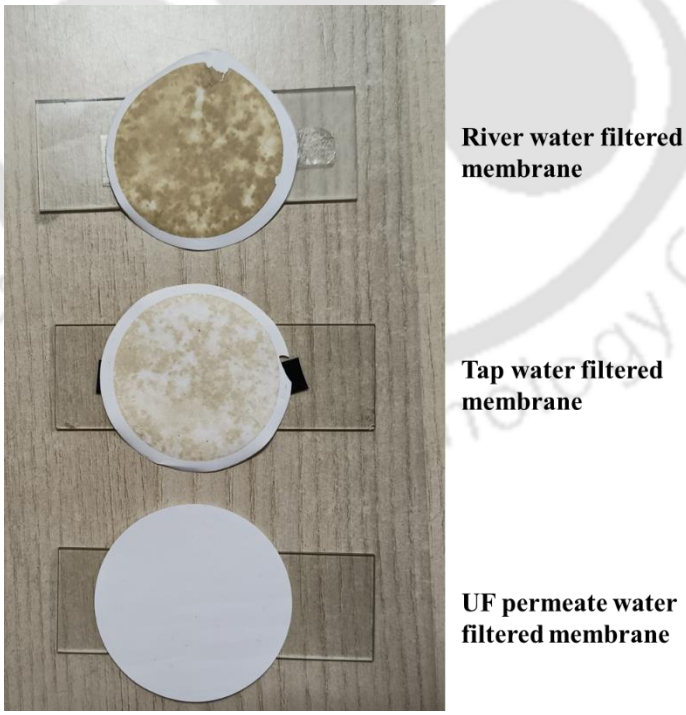


Figure 6. Representative images of river, tap, UF permeate water filtered nylon/PCTE membranes.

identification stages to cross-check the contamination of plastic materials, and the results were negative, with no microplastics in the blanks. To see the adsorbed particles on the microplastics, ≥ 4 mm microplastics were chosen and split into two pieces for the FESEM-EDX analysis. One sample analysed without digestion and another with digestion (H_2O_2, 30%).

3.1.3. Removal of microplastics using an ultrafiltration membrane system

Removal of microplastics from tap water was carried out using hollow fibre ultrafiltration (HF-UF) membranes (Make: Eureka Forbes ltd. Model: Aquaguard Superb-UF membrane commercially available in India, UF membrane dimension 11.5 x 5 x 5 cm, pore size 0.02 to 0.1 µm). The schematic flow diagram of the HF-UF membrane setup is shown in **Fig. 7**. The membranes were soaked in distilled water the day before the experiments, and a pure water permeability test was performed. The experiment was carried out at room temperature, and the highest number of microplastic contaminated tap water was passed through HF-UF membrane. To keep the feed pressure at 14 psi, a pump was used, which was connected to the ultrafiltration membrane via a control valve (CV1) in cross-flow mode operation. The feed flow rate and pressure were adjusted using the pump speed controller and control valve (CV2). The permeate from the membrane module was collected, and flow rate was measured using a flow meter (Ascorp, intelligent flow totalizer). Repeated experiments were conducted to assess the membrane performance and microplastic removal efficiency. The collected permeate solution was filtered through a polycarbonate track etching (PCTE)/nylon membrane using a vacuum filtration setup, and membranes were further characterized using an optical and fluorescence microscope.

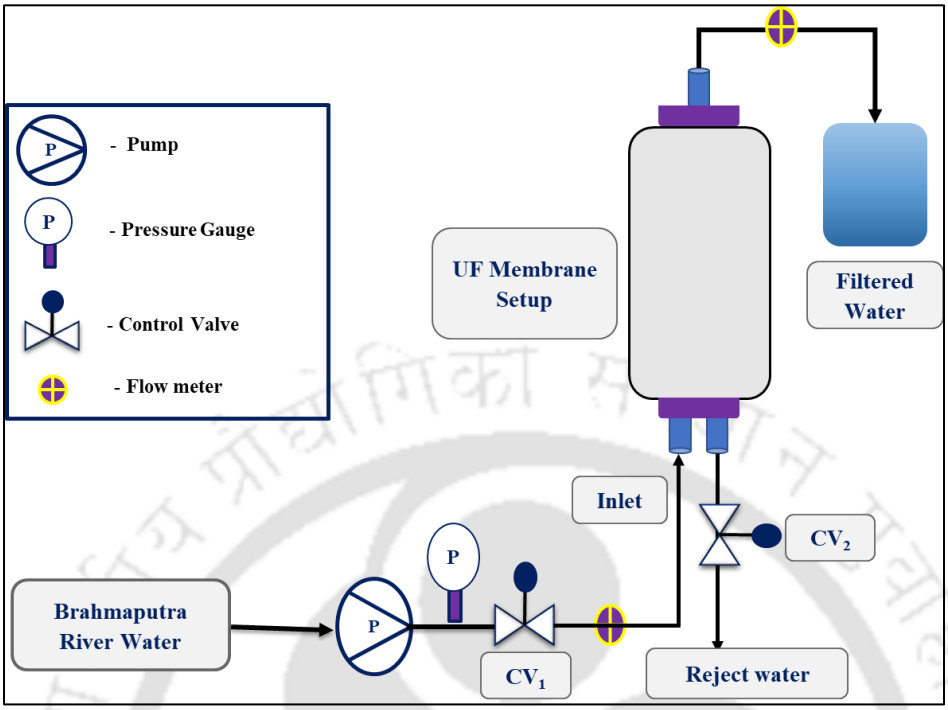


Figure 7. Experimental setup for removal of microplastics from tap water.

3.1.4. Microplastics identification and composition

Microplastics were identified and counted using an optical and fluorescence microscope (Axio Scope.A1, Carl Zeiss, Germany). Microplastics were visually estimated to classify their colour, size, and shape based on their physical characteristics. White and transparent microplastic particles were difficult to distinguish in both bright and dark field mode images. This type of problem is mitigated with the use of fluorescence microscopy. microplastics easily absorb the Nile Red dye and give fluorescence images when exposed to red, green, blue, or UV light. Preparation of Nile Red dye stock solution and working solution carried out.

Microplastics composition has been analyzed using micro-Raman spectroscopy (Horiba Scientific, LabRam HR, Lab Spec 6 software). For the Raman spectroscopy, a 20, 50, and 100x objective lens (NA 0.55) and 5 mW laser source with a grating 1,800 lines per mm were used.

Spectra were scanned from 400 cm^{-1} to 3500 cm^{-1} with an acquisition and accumulation time of 10 s and 2 s, respectively. The KnowItAll software from Bio-Rad has been used to identify and process the polymers spectrum. Using a field emission scanning electron microscope (FESEM, Carl Zeiss, Sigma 300) coupled with energy-dispersive X-ray spectra (EDX), the surface morphology and adhesion of any other elements to the microplastic were analyzed. To prevent charging, microplastics samples were coated with gold. With a working distance of 6 to 8 mm and an accelerated voltage of 5 kV, high-magnified images of microplastics were captured. EDX analysis was carried out at a 20 kV accelerated voltage using an Oxford Instruments system and Aztec V2.1 software.

3.1.5. Data analysis

One-way analysis of variance (ANOVA) was used to statistically analyze differences in microplastic concentrations between locations and environmental factors. If the p-value was less than 0.05, differences were deemed statistically significant. MATLAB and Microsoft Excel (2019) were used to create the graphical representation of the data on the abundance of microplastics and their physical characteristics.

3.2. Results and discussion

3.2.1. Abundance of microplastics

Microplastic particles were identified using optical microscopy, as shown in **Fig. 8a) and b)**. It was found that microplastics concentrations varied from 0.20 ± 0.03 to 0.52 ± 0.09 particles /L in tap water, whereas river water had microplastics concentrations ranging from 0.30 ± 0.08 to 2.56 ± 0.13 particles/L. Distribution of microplastics along the selected location is shown in **Fig. 9**. The microplastic abundance in river water was higher at sampling site R2 and R3 (centre of the city), whereas the lowest value was observed at sampling site R6 (less populated area).

The reason for the higher abundance could be due to the breakdown of large plastic materials which entered the river through urbanisation (massive effect from municipal sewage), air, and surface runoff. In the case of tap water, the highest and lowest value was observed at T4 and T2 site, respectively. The primary sources of these microplastics in fresh water are urban drainage systems, surface/storm-water runoff, WWTPs effluent, agriculture, wind, fisheries, industry, as well as the breakdown of plastic items due to weathering process (Sekudewicz et al., 2021; Wang et al., 2021). Dry deposition caused by wind movement is another route for the spread of microplastic (Amato-Lourenço et al., 2021; Stanton et al., 2019). Larger plastic debris and the by-products of their degradation could enter aquatic habitats by wind dispersal (Gasperi et al., 2018; Jan Kole et al., 2017).

ANOVA study indicates a significant variation in the concentration of microplastics at different sampling sites. The F value in the river was 65.56, and the associated p-value was 1.91×10^{-9} ($\alpha = 0.05$), whereas the F value in the tap water was 7, and the related p-value was 2.80×10^{-3}, indicating that the two places are different from one another.

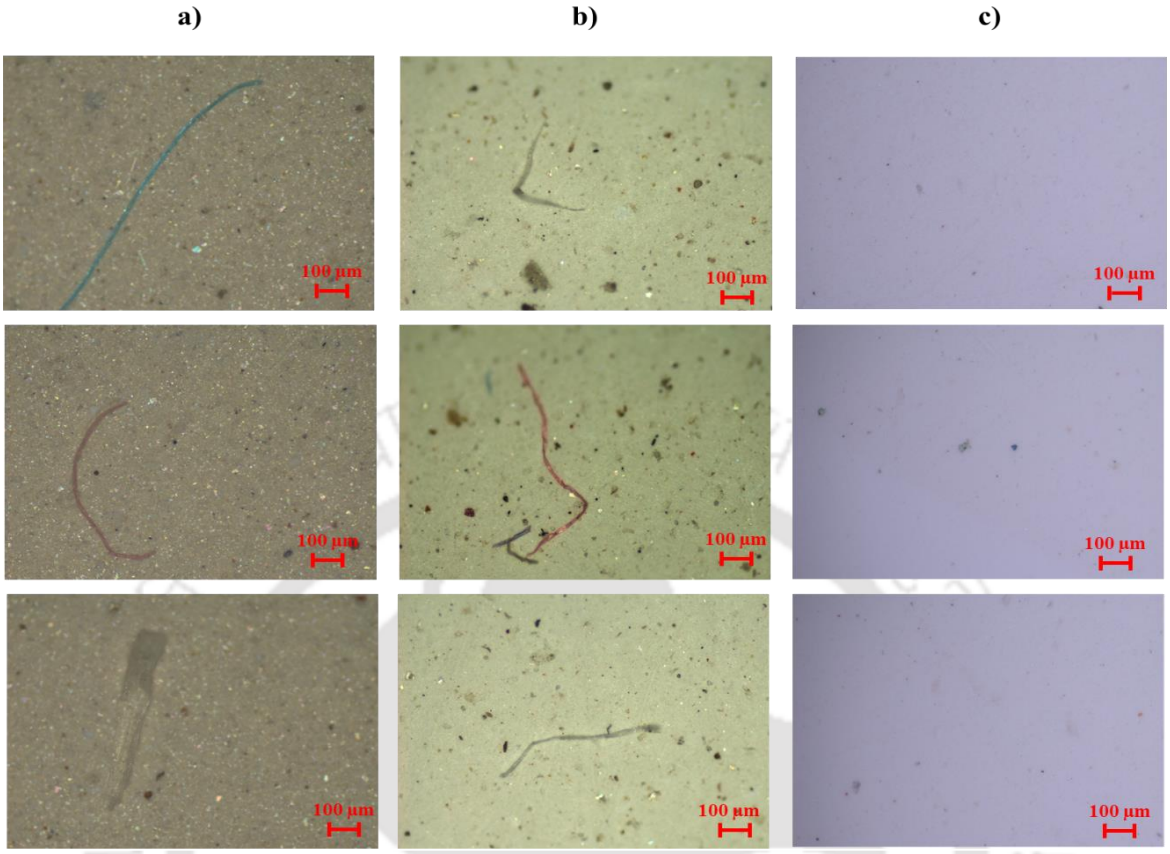


Figure 8. Microscopic images of a) microplastics in river water, b) tap water and c) UF-HF permeate.

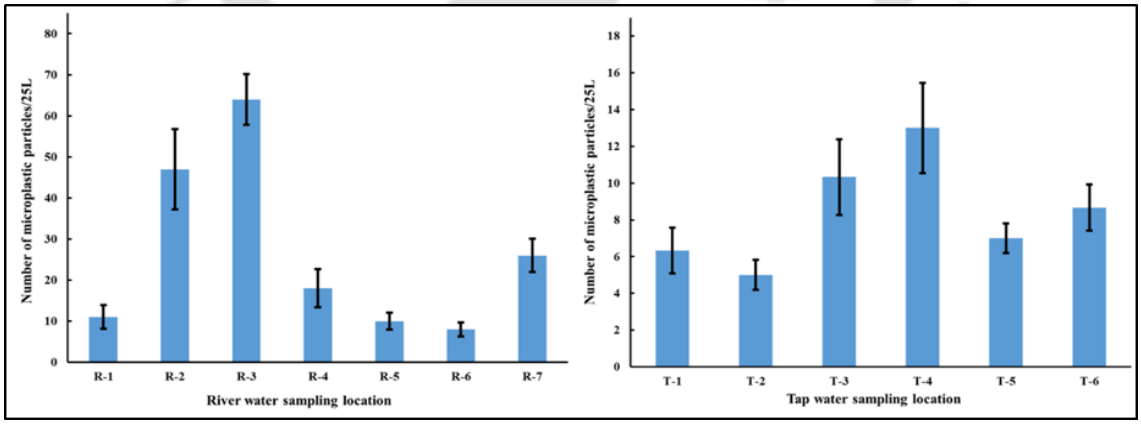


Figure 9. Distribution of microplastics along the selected location a) river water, b) tap water samples.

3.2.2. Morphological characteristics

Microplastics on the filtered membrane were chosen for morphological examination under an optical microscope. Later, microplastic particles were categorized according to their colour, size, and shape. The most prevalent colour particles were white, transparent, blue, brown, and black as shown in **Fig. 10**. Plastic carry bags and packing materials are the main sources of transparent and white microplastics. Also, as a result of the photoaging process, the colour of the plastic changes, particularly in terms of lightness. Plastics progressively transform to light colours when exposed to sunlight for a prolonged period of time (Zhao et al., 2022). Blue-coloured microplastics were also most typically found, which might be attributed to polyester fibres from clothes, fishing nets, and the use of various plastic packaging materials. The presence of black microplastics in river water might have been released into the environment during transportation due to tyre abrasion on road surfaces as part of normal wear and tear. In contrast, black microplastics may have been released from the storage tank and water pipes in tap water. microplastics were classified into four sizes (0.1 - 1 mm, 1.0 - 2 mm, 2 - 3.3, and 3.3 - 5.0 mm) as shown in **Fig. 11**. The size distribution of microplastic particles in all examined samples shows clearly that concentrations of particles under <1 mm are significant. These results matched several previous investigations in which smaller-sized microplastics predominated (Gopinath et al., 2020; Huang et al., 2021; Singh et al., 2021). Because of the higher degree of degradation and weathering of larger plastics, the factor contributed to a higher abundance of smaller microplastic particles (Chanpiwat and Damrongsiri, 2021). According to the size distribution data, the most prevalent size was (<1 mm), contributing for more than 55% in river water and 70% in tap water. Another important observation from this study was that microplastics were predominantly found in three morphologies: fibers (59 %), fragments (28 %), and sheets (13 %) in river water. Similarly, fibers (56%), fragments (33%), and sheets

(11%) were found in tap water samples **Fig. 12**. The most prevalent type of microplastics were fibers and size ranges between 0.1 to 5 mm. The content of smaller microplastics in the river water samples is presumably due to the fact that plastic breaks down into smaller parts or because lighter particles are more easily washed from the sediments. According to Sekudewicz et al. (2021), fibers float easily in the water column, whereas micro beds agglomerate more easily and settle at the bottom of the river and are not uniformly distributed. microplastics, both coloured and uncoloured, pose a serious threat as they are consumed by plankton, fish, and other organisms that are unable to distinguish them from prey. Because microplastics are abundant and zooplankton consume a large amount of it. This also raises serious concerns about the higher tropic level organisms and the consequences for the biota. Thus, the biotic components, particularly those studied on fish and even humans for this freshwater system, necessitate extensive research to determine the effect of microplastic's bio toxicity and health hazards to the organism.

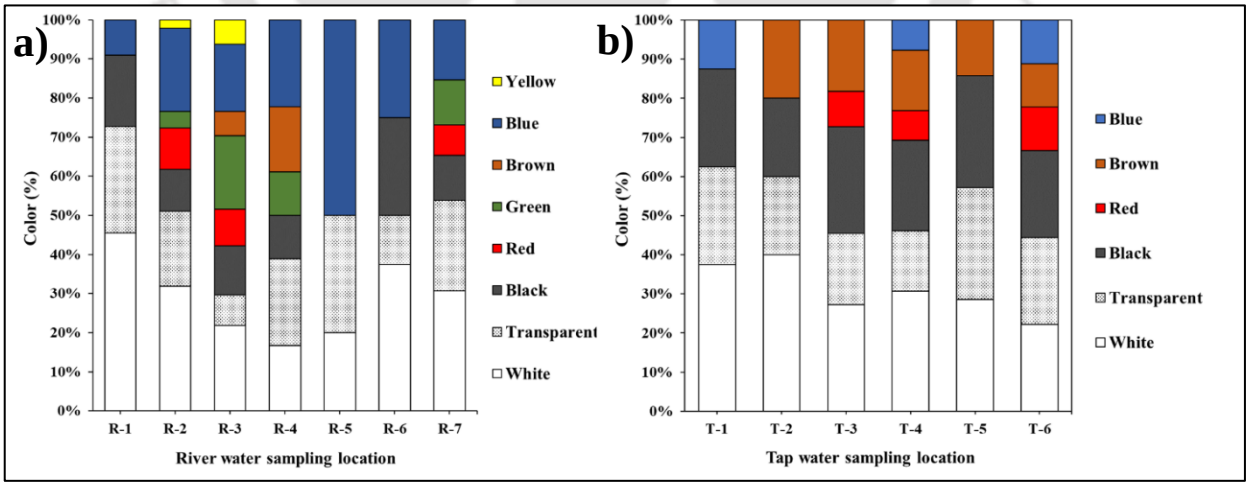


Figure 10. Colour distribution of the microplastics along the selected location a) river water, b) tap water.

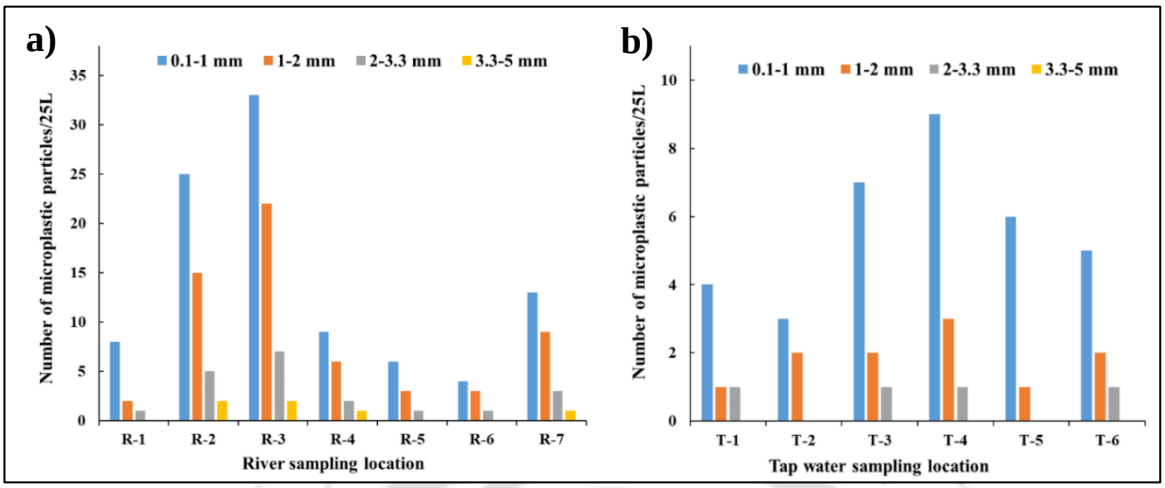


Figure 11. Size distribution of microplastics in a) river water and b) tap water samples.

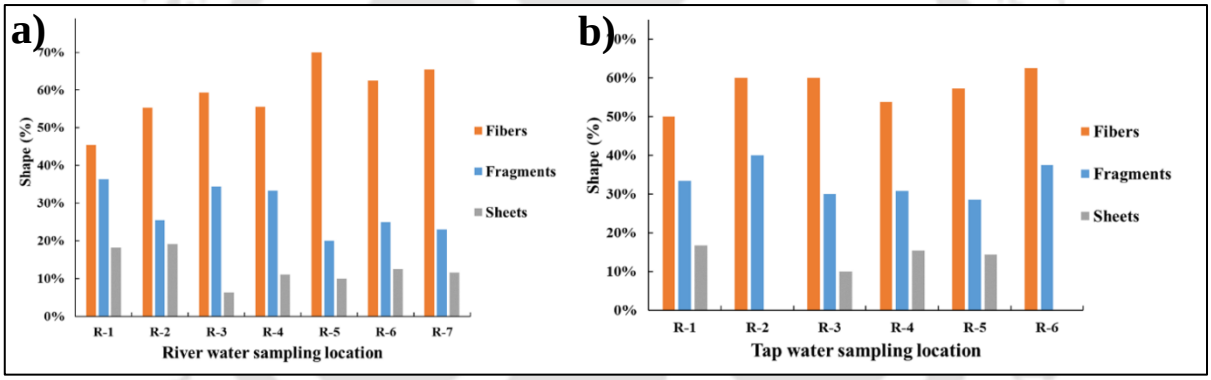


Figure 12. Morphological distribution of microplastics in a) river water, and b) tap water.

3.2.3. Chemical composition of microplastics

Microplastics from river (50 numbers) and tap (25 numbers) water samples were randomly selected. These samples compositional study was identified using Raman spectroscopy, as shown in **Fig. 13.** The results found that PE, PP, PES, PS, and pigmented polymers were the most common polymers in river water. Whereas PE, PP, PVC, and pigmented polymers were more dominant in tap water samples. Further, PE and PP were found to be the most detected

polymers, and their presence is a result of processes including packing, labelling, and construction. Moreover, the presence of PES is mainly attributed to the washing of synthetic clothes, which is also explained by (Dalmau-Soler et al., 2021). From, it is seen that PE-Ivory black (carbon) and PVC polymers were seen only in tap water samples. Which confirms that water pipes and storage tanks of the supply system are frequently leaches microplastics. The presence of PVC microplastics in tap water was also observed in earlier studies (Mukotaka et al., 2021; Shen et al., 2021b).

Field emission scanning electron spectroscopy (FESEM) coupled with EDX was used to analyze the surface of microplastics. Through analysis, it was found that several metals, including Al, As, Ca, Cu, Co, Fe, Mg, Mn, Na, K, P, Si, Ti, and Zn, were present on their surface and may be absorbed by the microplastics due to their larger surface area. Since gold was utilized as a surface coating, its prominent X-ray peak (Au) is seen in all spectra. According to Richard et al. (2019) and Tu et al. (2020) research, microorganisms can easily colonize the surfaces of microplastics and are capable of forming biofilm, to which metals and other pollutants have adhered. present studies also confirm that concentrations of metals are directly related to biofilm accumulation. After organic matter degradation, metal concentration was drastically reduced (**Table 10**), and some metal particles remained adhered to microplastics near surface grooves and cracks. All three samples have high concentrations of Al, Ca, Fe, and Si, which can be seen in **Fig. 14** and **Table 10.** Clay minerals, in particular, have strong sorption capabilities, which may be the reason for the metal's presence on these fine-grained particles. Similar elements were noticed on the surface of microplastics in Poland's Vistula River (Sekudewicz et al., 2021), Red Hills Lake, India (Gopinath et al., 2020), and Atoyac River (Shruti et al., 2019). According to Shruti et al. (2019), the presence of metals

such as Al, Ca, Fe, K, Na, Mg, and Si are derived from geological sources. Future research on the interactions of microplastics with biofilms and hazardous pollutants, in addition to the release of contaminants attached to microplastics into the environment, is required.

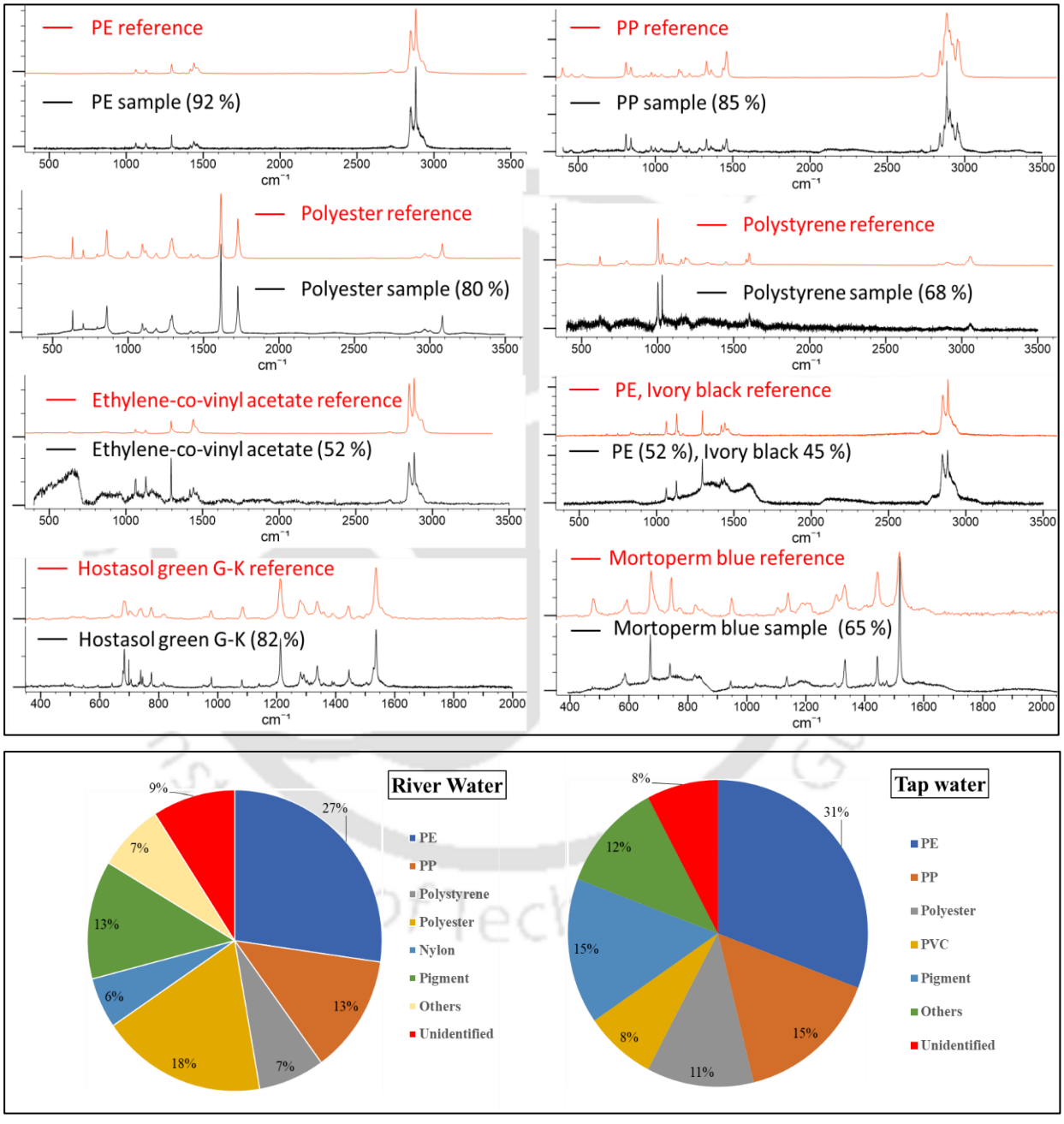


Figure 13. Raman spectra of different microplastics and their average composition.

Table 10. Chemical composition (wt.%) of polymer and minerals on microplastic particles before and after digestion using EDX analysis.

MPs samples	**Sample 1**		**Sample 2**		**Sample 3**	
	Before digestion	After digestion	Before digestion	After digestion	Before digestion	After digestion
C	70.2	85.1	67.8	90.1	70.3	85
O	20.6	11.5	22.5	7.6	20.4	11
Si	2.2	0.8	4.4	0.6	3.2	1.1
Fe	2	0.6	1.1	0.4	1.4	0.1
Al	1.5	0.6	1.6	0.4	1.8	0.4
Ca	1.2	0.7	0.4	-	1.3	1.4
K	0.7	-	0.5	0.1	0.5	0.1
Cu	0.5	0.2	0.2	-	-	-
Na	0.4	0.1	0.3	-	0.2	-
Mg	0.3	0.1	0.3	0.1	0.3	0.3
Mn	0.2	-	0.2	-	-	-
Zn	0.2	-	-	-	-	0.1
Ti	-	-	-	-	0.2	0.3
P	-	0.2	0.1	0.2	0.1	0.2
Cr	-	-	-	-	-	-
Co	-	-	0.6	0.3	-	-
Ni	-	-	-	0.1	-	-
As	-	0.1	-	0.1	0.3	-
Total	100	100	100	100	100	100

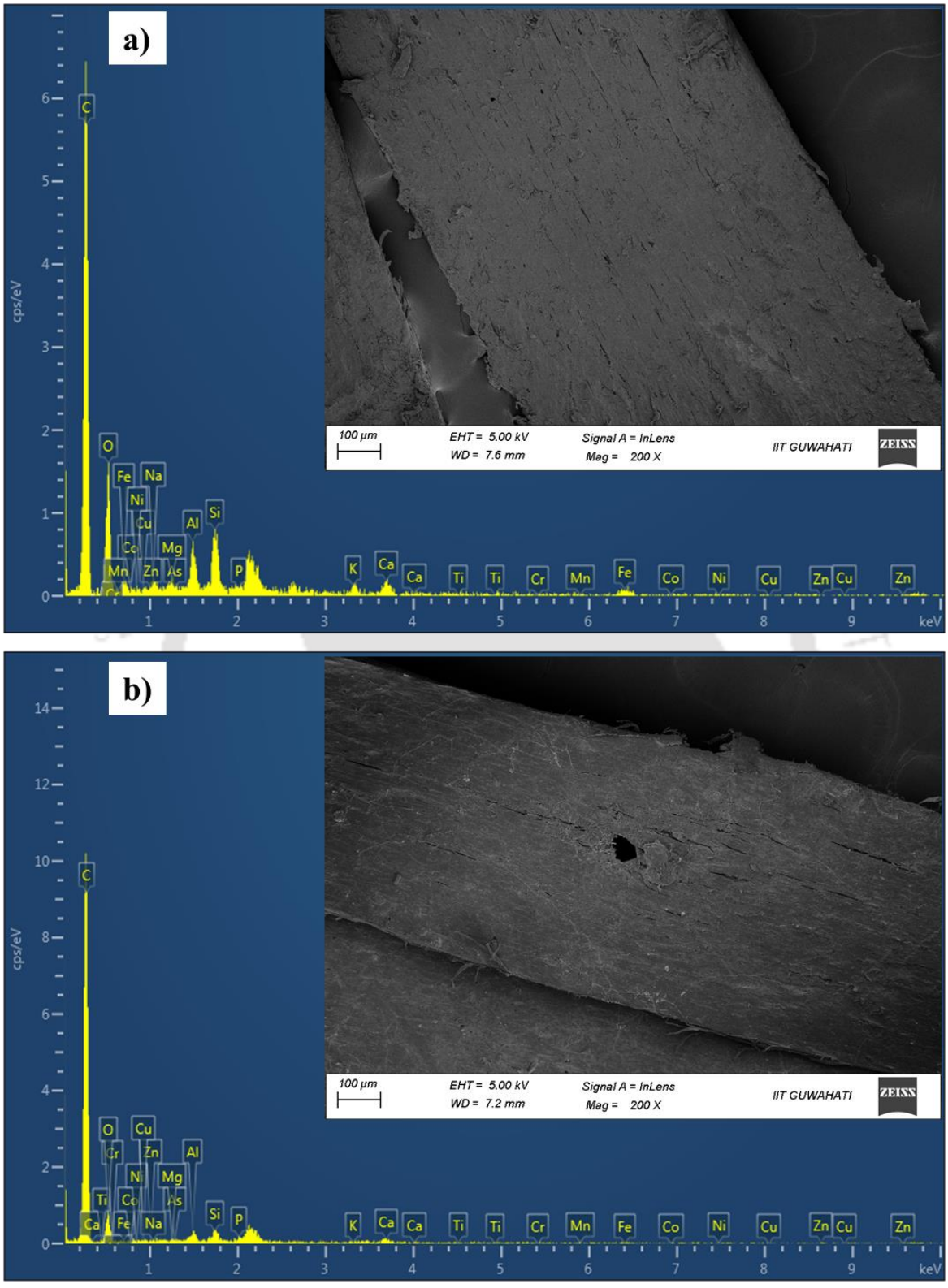


Figure 14. FESEM-EDX images of a) aged microplastics with absorbed particles before the digestion b) after microplastics digestion.

3.2.4. Removal of microplastics from tap water

microplastics removal methods should be simple, cost-effective, and time-efficient. An attempt was made to remove microplastic particles from tap water using HF-UF membrane and it was found that 100 % effective at eliminating microplastics (≥ 10 μm).

3.2.5. Comparative study of the detected concentrations of MPs in aquatic environments

The average abundance of microplastics in the Brahmaputra River water was 0.30 ± 0.08 to 2.56 ± 0.13 particles L (the sampling year 2022). This value is higher than levels detected in other rivers in India, such as the Adyar River in Tamil Nadu (2019, 0.33 particles/L), the Kosasthalaiyar River in Tamil Nadu (2019, 0.67 particles/L), and Ganga River (2019, average 0.038 ± 0.004 particles/L). The **Table 6** shows that microplastic abundance is very low compared to rivers from other countries, such as the West River in China (2019, 2.99 - 9.87 particles/L), the Llobregat river basin in Catalonia, Spain (2019, 3.60 particles/L), and The Chao Phraya and Maeklong River in Thailand (2021, 0.4 - 2.4 particles/L). Present findings show that microplastic pollution levels in the tap water were relatively low compared to others literature. Furthermore, the comparison of microplastic concentrations detected suggests that the current study could serve as reference data in the field of microplastic abundance in fresh water.

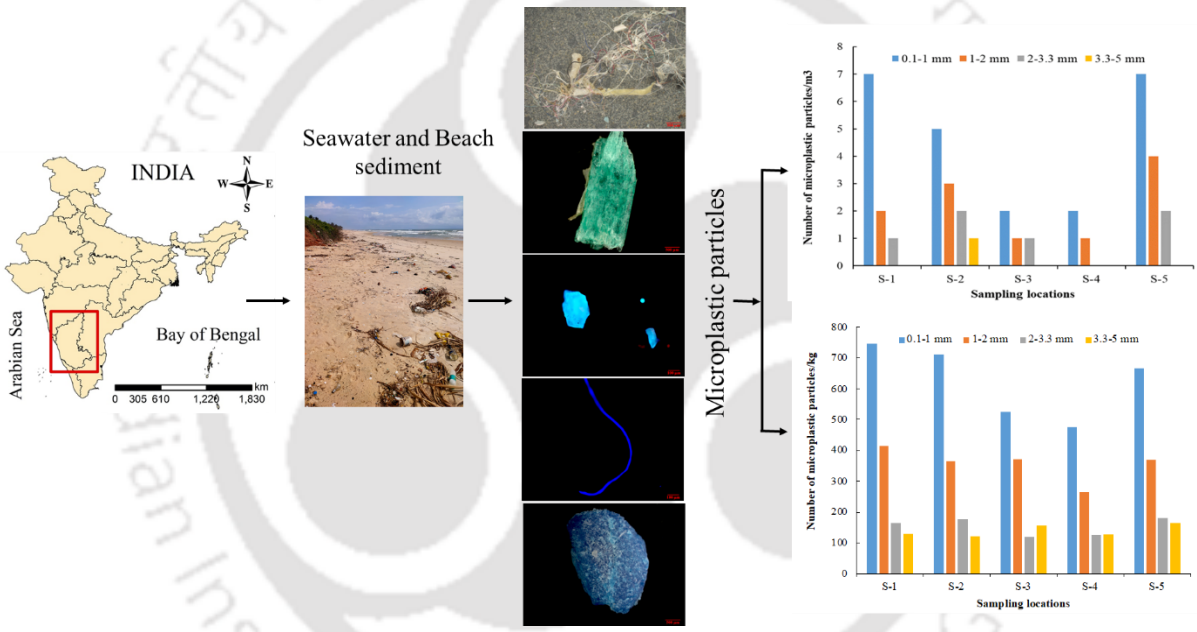
3.3. Summary

Rivers are generally the main source of water to use for human consumption. The current study focused in investigating microplastic contamination in both Brahmaputra River water and tap water collected from various locations. The microplastic particles was observed in both river and tap water. The study also revealed that microplastic fibres were dominant, and a majority of them were in the 0.1 - 1 mm size range. Most microplastic properties, including size, shape,

and composition, detected in tap water also matched those in river water. The presence of various elements on the surface of microplastics, as well as adhered particles onto their surface, were found. Using commercially available membranes, the removal of microplastics from tap water was assessed. Microplastics were significantly reduced after passing through the HF-UF membranes.

Chapter 4

4. Distribution and characterization of microplastics in Indian (Karnataka) coastal environments



Preface: *In this chapter, identified the occurrence and distribution of microplastics in the marine water and beach sediments collected from Karnataka state.*

4.1. Materials and methodology

4.1.1. Study area

Dakshina Kannada (DK) and Udupi are the districts in the state of Karnataka in India. The total population of the region is 2,089,649 and 1,177,361 (census2011.co.in), respectively. The major anthropogenic activities that prevail along the coast are urban centers, industries, tourism, fishing harbors, and landing centers. Both these cities attract a huge number of tourists because of their location between the Arabian Sea and the Western Ghats mountain range. Five beaches have been selected for sediment sampling; Tannirubavi (N12º.8912, W74º.8138) (S1), Panambur (N12 º.9390, W74º.8035) (S2), Surathkal (N12º.9913, W74 º.7929) (S3) beaches in the Mangalore region of DK, Kapu (N13º.2221, W74º.7379) (S4), and Malpe (N13º.3607, W74 º.6977) (S5) beaches in Udupi (**Fig.15)**.

Figure 15. Beach sample collection.

4.1.2. Sample collection

Sampling was carried out at the high tideline in August 2020 (**Fig. 16**). Samples were collected from each beach in three different regions. A gap of about 100 m was kept between the two

sampling points. Samples were scooped out after placing a 1 m^2 quadrant (Patchaiyappan et al., 2021, 2020). Approximately three kilograms of sand were collected from each quadrant location and stored in glass jar to prevent additional plastic contamination.

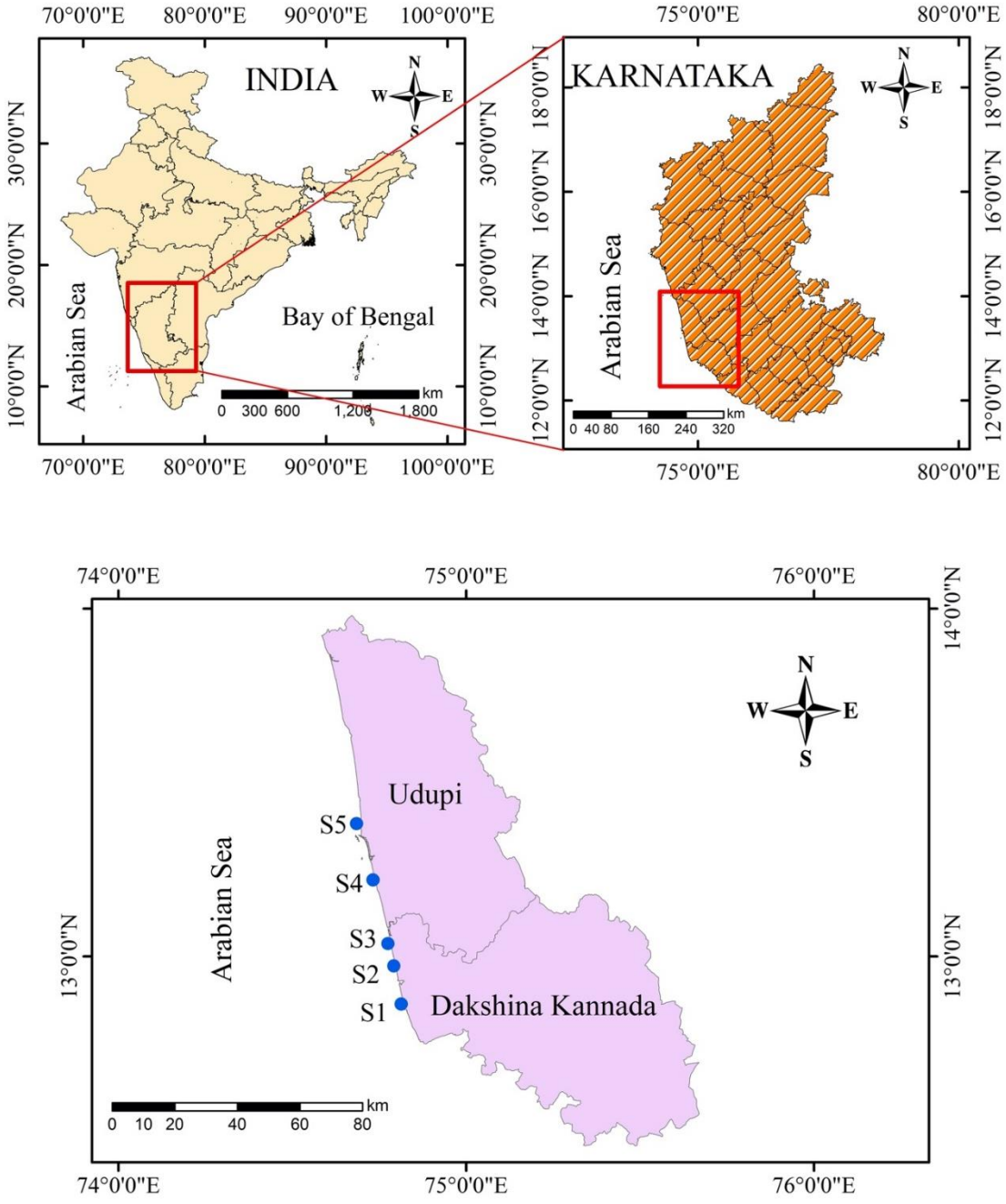


Figure 16. Map representing sampling locations across Indian coastal environment.

4.1.3. Microplastics extraction from seawater and beach sediments

The bulk sediment samples were dried in the hot air oven at 65 °C for 24 h. For the sample analysis, 1 kg of dry beach sediment was sieved with 5, 4, 3.3, 2, and 1 mm sieves. The particles retained on the 4, 3.3, 2, 1 mm sieves, and 100 g of a sieved sand sample from 1 mm were collected and then transferred to a conical flask. For organic matter removal, samples were digested with hydrogen peroxide (H_2O_2, 30%) (Merck, USA) (Patchaiyappan et al., 2020). 20 mL of filtered H_2O_2 and 100 mL distilled water was then added to each conical flask and covered with aluminium foil. The samples were kept in an incubator shaker (Jeio Tech, Korea) and agitated at 120 rpm for 24 h at 65 °C followed by 48 h at 30 °C. Microplastics were isolated by the density separation method as reported by Lots et al. (2017) and Patchaiyappan et al. (2020). The same method with slight modification was adopted in this work to enhance the yield of microplastics. 360 g NaCl (HiMedia Laboratories Pvt. Ltd, Mumbai, India) was dissolved in 1 L distilled water to prepare a supersaturated NaCl solution (density 1.2 g/cm^3). Salt solution were filtered through nylon filter (0.2 µm pore size, 47 mm dia) to remove impurities. 300 mL of filtered saturated salt solution was added to each conical flask after the digestion process. The sample was then transferred to the separating funnel and kept for 24 h. Further, the supernatant solution was filtered using Whatman Grade 1 filter paper with the help of a vacuum filtration setup. To increase the recovery of microplastics, a three times isolation process was repeated. The filtered paper was dried for 24 h at 50 °C and then characterized. Similarly, seawater samples were filtered through a mesh of 100 µm and retained samples were collected. The samples were digested in the same manner as sediment samples. Flowchart for the sample collection and sample processing is shown in **Fig. 17**. Special care was taken during the experiments, and no plasticware was used. To prevent airborne microplastics, experiments were performed in laminar flow.

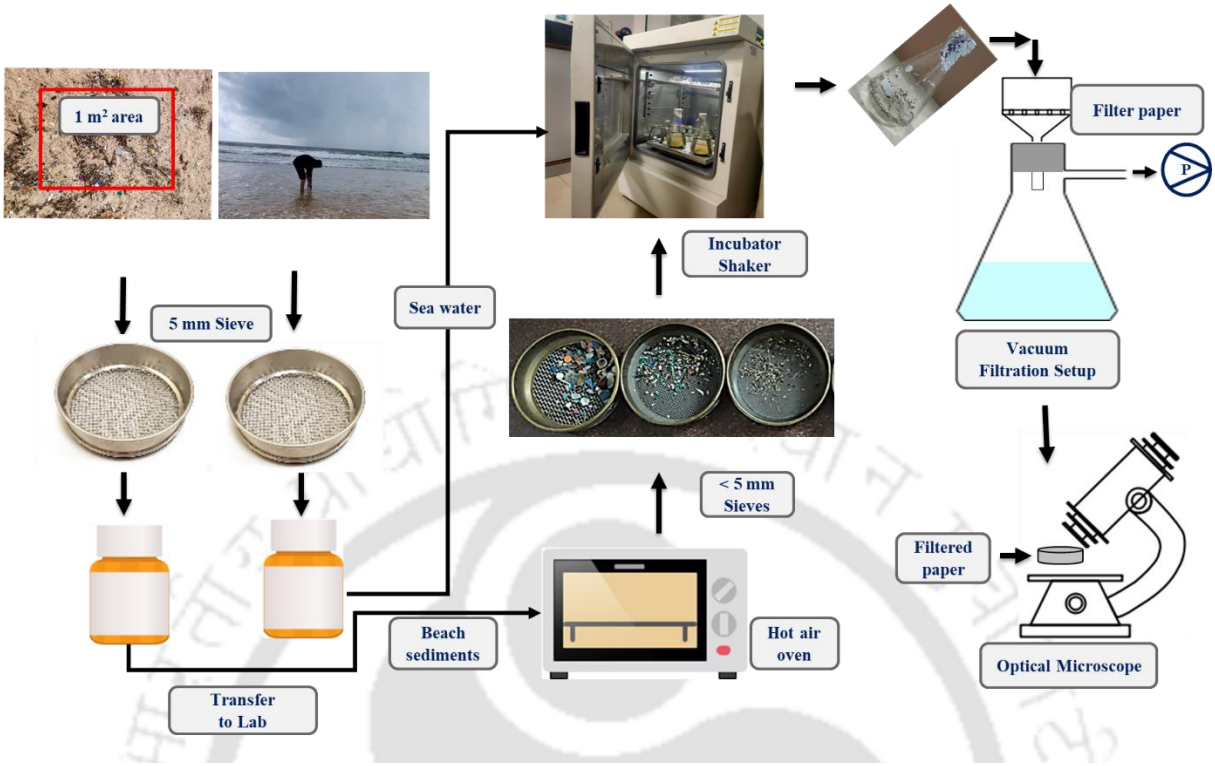


Figure 17. Protocol for the sediment and beach water sample collection and microplastic extraction.

4.2. Microplastics characterization

4.2.1. Optical microscopy

The optical microscope was used to identify and counting of microplastics (Axio Scope.A1, Carl Zeiss, Germany). Images with lenses of 2.5x, 10x, 20x, and 50x were obtained. Visual estimation of microplastics was performed to classify their shape, size, and color, according to their physical characteristics. It was hard to distinguish white and transparent microplastics in both bright and dark field imaging. The use of fluoresce microscopy mitigates this type of problem. The fluorescence staining method highlights the polymer particles present in the sample, providing a precise and facile approach for staining for which Nile Red dye was used.

The microplastics readily adsorbs the dye and fluoresce when irradiated with either red, green, blue, or UV light. Nile Red dye stock solution, and working solution, were prepared.

4.2.2. Raman spectroscopy

Micro-Raman spectroscopy has been used for the compositional analysis of microplastics (Horiba Scientific, LabRam HR, LabSpec 6 software). A 20x, 50x, 100x objective lens (NA 0.55) and 488, 532, and 633 nm laser source (5 mW; grating 1800 lines/mm) were used for the Raman spectroscopy. For the 400 cm^{-1} to 3500 cm^{-1} range, spectra were scanned with an accumulation and acquisition time of 2 s and 10 s, respectively. The polymers spectrum has been identified and processed using Bio-Rad's KnowItAll software.

4.2.3. FESEM-EDX analysis

The surface structure and morphology of the microplastic particles were analyzed using FESEM by Carl Zeiss instrument, model Sigma 300. To avoid charging, samples were coated with gold at the vacuum level of 8 - 10 Pa. High-magnified images of microplastics were captured with an accelerated voltage of 3 – 5 kV and at the working distance of 6 to 8 mm. The EDX analysis was performed with an accelerated voltage of 20 kV using an Oxford Instruments system with Aztec V2.1 software. The surface elemental composition of particles is helpful in identifying plastics dominating carbon over other interfering materials (non plastics). Also, the adhering chemicals on microplastics can be acquired by EDX analysis.

4.2.4. Data analysis

ANOVA was performed in R software (version 4.0.3) to understand the variability of microplastics abundance in five beach sediments. The statistical significance with p-value (< 0.05) was used for all statistical analysis. All data are shown as mean ± standard deviation. The

graphical representation of the microplastics abundance and physical characteristics data were produced with RStudio using ggplot2 and Microsoft Excel (2019).

4.3. Results and Discussion

4.3.1. The abundance of microplastics

Microplastics have been found in all seawater and beach sediment samples. 0.1 to 5 mm sized microplastic concentrations are reported here. Microplastic particles were observed under optical and fluorescence microscope, images have been shown in **Fig. 18 and Fig. 19**. The order of total microplastic abundance in seawater was Malpe (13 ± 1 particles/m^3) > Panambur (11 ± 0.5 particles/m^3) >Tannirubavi (10 ± 0.8 particles/m^3) > Surathkal (4 ± 2 particles/m^3) > Kapu (3 ± 1 particles/m^3). Similarly, in case of beach sediment, microplastics abundance was Malpe (1002 ± 174 particles kg^{-1}) > Panambur (931 ± 156 particles kg^{-1}) >Tannirubavi (781 ± 121 particles kg^{-1}) > Surathkal (350 ± 108 # particles kg^{-1}) > Kapu (264 ± 62 particles kg^{-1}). The highest abundance of microplastics were observed in Malpe beach (S-5), and the lowest was found in Kapu beach (S-4). ANOVA study projects a statistical difference between the five sampling locations in the concentration of microplastics in beach sediment. The F value determined was 19.5, and the corresponding p-value was 0.000103 ($\alpha = 0.05$), which suggests that the beaches are distinct from each other. Microplastic concentrations at the various sampling sites in the Indian coastal region are shown in **Fig. 20**. The variation of microplastics abundance in beach sediment depends on the degree of urbanization (waste generated on land entering the ocean), tourism, and fishing (Sathish et al., 2019). Another major pathway of microplastics that can get reflected in beach sediment is road and city stormwater runoff (Tiwari et al., 2019; Veerasingam et al., 2016). Kapu and surathkal beaches are having very less tourist activity and far from the industrial sector; hence the concentration of microplastics

was less compared to other beaches. The microplastics found on the beaches of Mumbai (In the Indian state of Maharastra) are less compared with the present study (Tiwari et al., 2019). Studies in China, Netherland, Italy, Turkey, and Tunisia reported maximum microplastics concentrations (Abidli et al., 2017; Lots et al., 2017; Vianello et al., 2013; Yabanlı et al., 2019). However, the presence of microplastics in Tamil Nadu and Andaman beaches (of India) showed nearly similar results to present study (Dowarah and Devipriya, 2019; Patchaiyappan et al., 2020; Sathish et al., 2019).

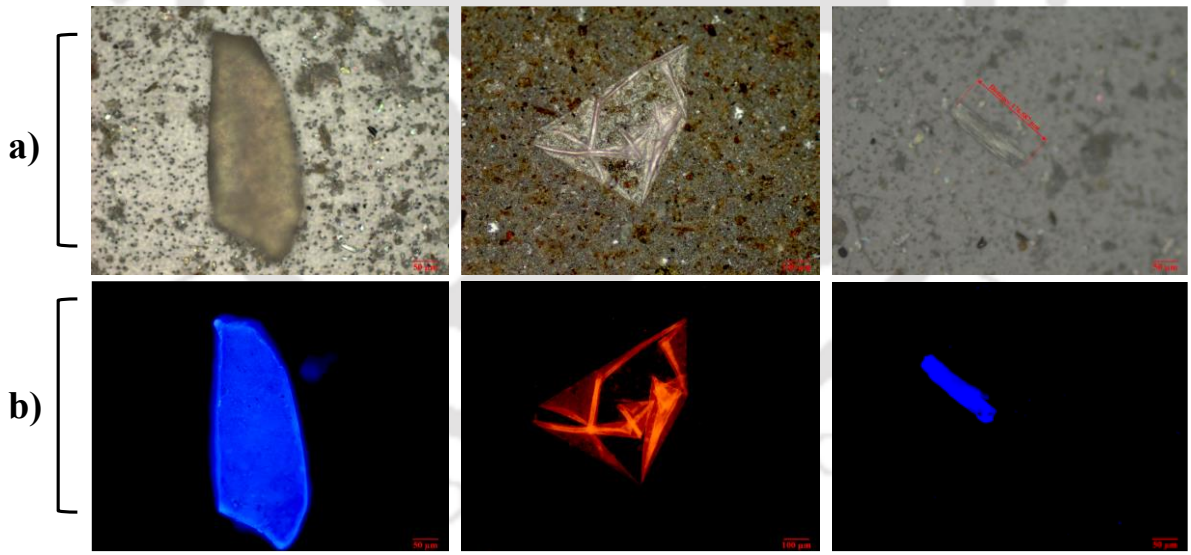


Figure 19. Microplastic particles observed under a) optical microscope and b) fluorescence microscope using Nile Red dye.

Fig sediments.

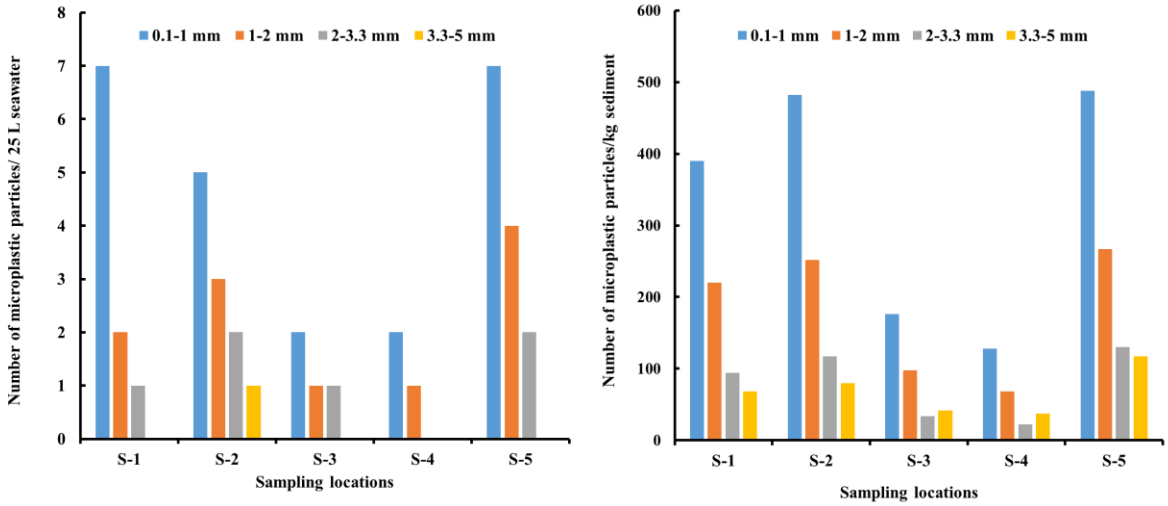


Figure 20. Microplastic concentrations at the various sampling sites in the Indian coastal region a) seawater and b) beach sediments.

4.3.2. Morphological characteristics of microplastics in beach sediment

Microplastic particles of different sizes, shapes, and colours were observed and analysed in all five beach samples (**Fig. 21**). Microplastics were classified according to their size into four subcategories to understand the size distribution: 0.1–1; 1–2; 2–3.3, and 3.3–5 mm. More microplastics were observed in the size range of 0.1 to 1 mm. The microplastics' size distribution is right-skewed, which is consistent with recent reports (Lots et al., 2017; Vianello et al., 2013). In addition, aquatic life faces greater risk from these tiny plastics due to their large surface area and a high adsorption capacity towards contaminants (Al-Thawadi, 2020; Sana et al., 2020). The samples collected were sorted according to their morphology into different types, including fragments, fibers, films/sheets, and pellets. The microplastics in beach

seawater were mostly fibers-type, which were abundant, with an average of 55 %, fragment 44%, and films/sheets 5%. The colour distribution was in the order of white (32%), blue (22%), transparent (16%), black (12%), green (9%), red (7%), brown (2%). The microplastics in beach sediments were mostly fragment-type, which were abundant, with an average of 46%, fibers 34%, films/sheets 12%, and pellet 8%. The colour distribution of microplastic was in the order of white (26%), transparent (18%), blue (13%), red (12%), black (8%), green (8%), purple (4%), brown (4%), pink (3%), orange (2%), and yellow (2%). The variation of the colour may be due to their origin from different sources. The distribution of the shape and colour of the microplastic in beach is given in Fig. 6 a) seawater and b) beach sediment, respectively.

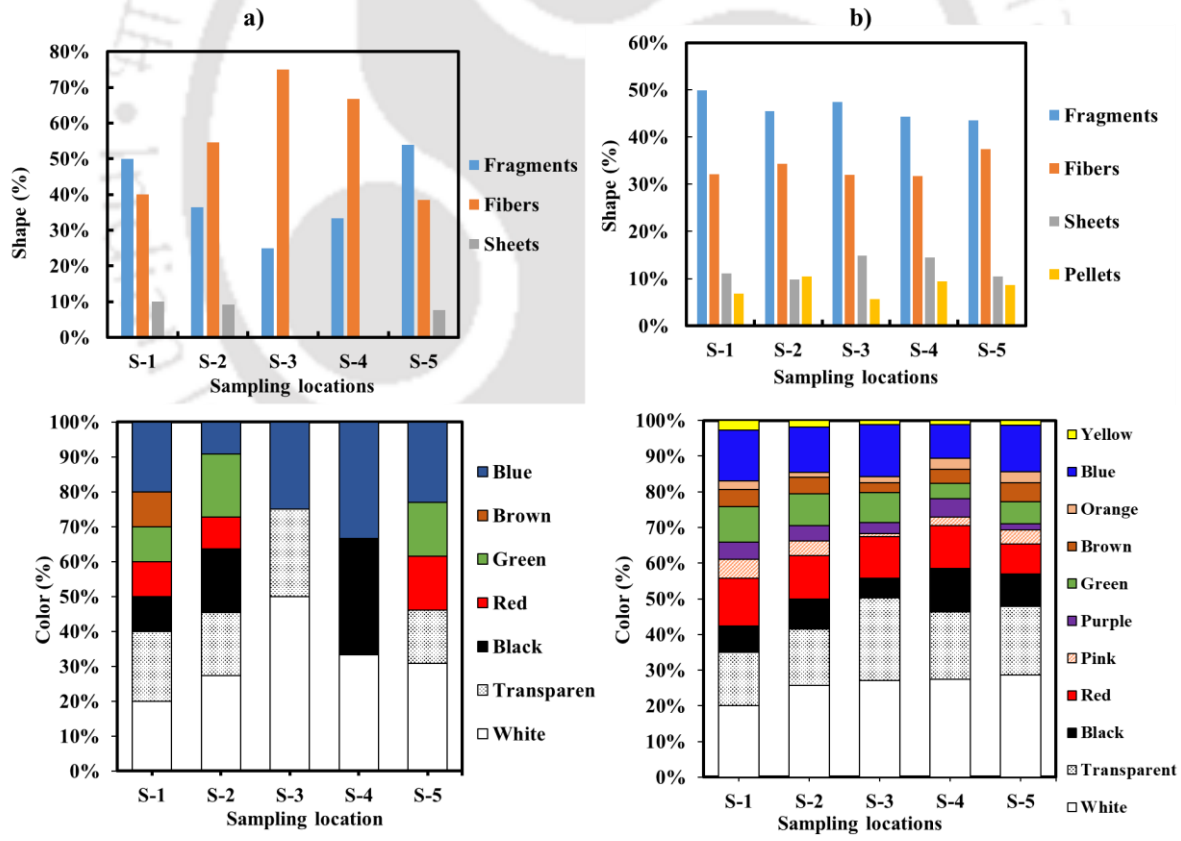


Figure 21. Distribution of shape and colour of microplastics in a) seawater b) beach sediment.

4.3.3. Raman spectroscopy analysis

It is essential to study the microplastic composition as it offers valuable information on specific particles, which in most cases can be traced back to their origin. For the micro-Raman spectroscopy study, 100 and 250 numbers individual particles were collected from all the seawater and beach sediment samples respectively. In seawater samples PE (31 %), PP (22%), nylon (10 %), polyethylene terephthalate (PET) (6 %), PVC (6 %), PS (3 %), and other polymers (19 %) were observed. Among the 250 particles, of beach sediment, 215 particles were able to fulfil the criteria to be classified as microplastics. The predominant presence of PE (37%) is followed by PP (26%), PS (17 %), nylon (PA) (9 %), PET (2 %), PVC (2 %) and other polymers (7 %). The results of Raman spectroscopy of the various microplastics are shown in **Fig. 22**.

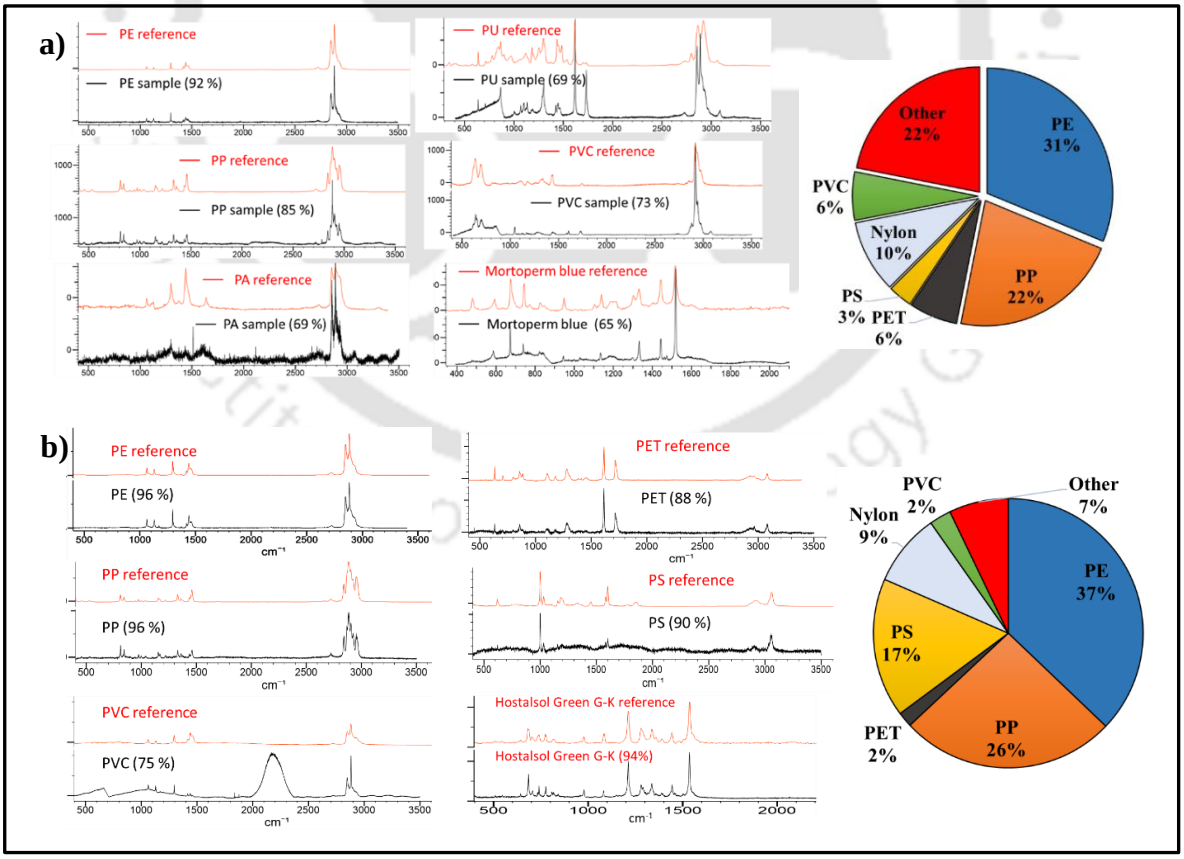


Figure 22. Raman spectra of different microplastic particles a) seawater, and b) beach sediment.

Analysis of Raman spectroscopy shows considerable differences among samples collected from sediment in polymer types (Robin et al., 2020; Tiwari et al., 2019). Amongst all the sediment samples, PE was dominant. Most of the sheets were PE category plastics. This is because PE is commonly used in containers, bottles, and materials for packaging (Yu et al., 2016). The next abundant type of plastic that was found in beach sediment was PP and PS. Possible sources of these PP and PS products may be from domestic waste around the vicinity and industrial-scale fishing activities (Yu et al., 2016). There are more fishing industries in Dakshina Kannada and Udupi districts, so PS plastic materials used as packaging are predominant in marine sediments. Widespread degradation of nylon from fishing nets is evident from the presence of polyamide (PA) in the beach sediments. Pigmented particles were also identified along with polymers. The interference of pigment spectra with polymer spectra impeded microplastics' identification (Patchaiyappan et al., 2020; Seth and Shriwastav, 2018). The present findings have reported the presence of microplastics in beach sediments and are in line with the previous studies from different parts of the world, as shown in **Table 5** of section 2.2.

4.3.4. Surface morphology and elemental analysis

FESEM images (**Fig.23, a)**) of PE and PP microplastics showed cracks, roughness (indicates its aging), and accumulation of fine dirt particles on the surface due to environmental exposure (Pathak and Navneet, 2017; Zbyszewski et al., 2014). A similar pattern of cracks, pits, and surface erosion has been seen in Tamil Nadu beach sediment samples (Sathish et al., 2019). The EDX analysis (**Fig. 23, b)**) showed the microplastics having high carbon content, which suggests the presence of polymeric materials. In the present study, 18 trace elements such as Al, Ba, Bi, Br, Ca, Cd, Cr, Cu, Fe, Hg, Mg, Mn, Ni, Pb, Sb, Sn, Ti, V, and Zn were observed

in the beach microplastics. The presence of heavy metals in microplastics (Hg, Cd, Pb, Cr, etc.) was probably due to their regular use as stabilizers, flame-retardants, antioxidants, colorants catalysts (Azeh Engwa et al., 2019; Robin et al., 2020). Sathish et al. (2019) reported that $CaSiO_3$ and $CaCO_3$ are broadly used as additives in plastic. Since gold was used for surface coating, the X-ray peak of gold (Au) is dominant and visible in all spectra. In addition, other metals carried by microplastics were accumulated from the surrounding environment (Qiu et al., 2015; Wang et al., 2017). The extent of metals present in the microplastics was determined in terms of observed elements to carbon ratio (OECRs) (Velaga et al., 2021). The OECRs mentioned almost similar (7 ± 1) in all the collected samples. This study highlighted the need for a future understanding of the potential mechanism for the release of heavy metals from these particles under various environmental conditions. This data would be helpful in identifying the source and characteristics of plastics that are likely to cause possible hazards to the marine and coastal environment.

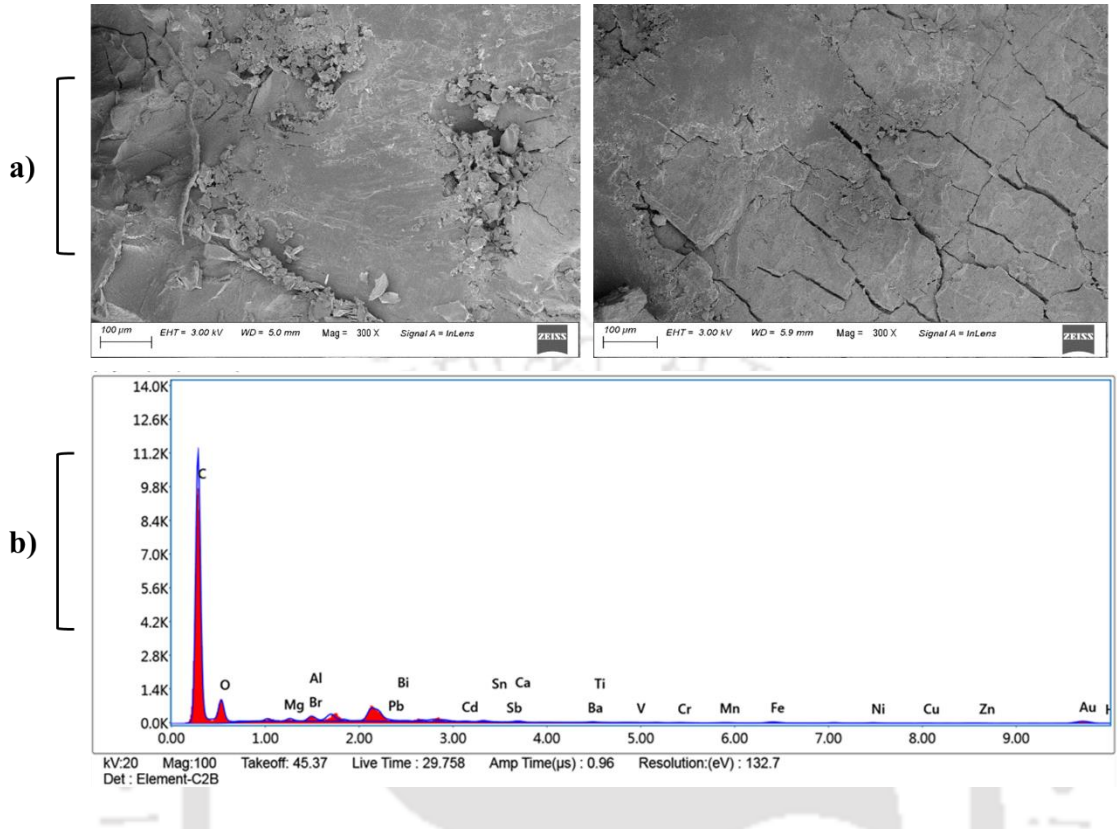


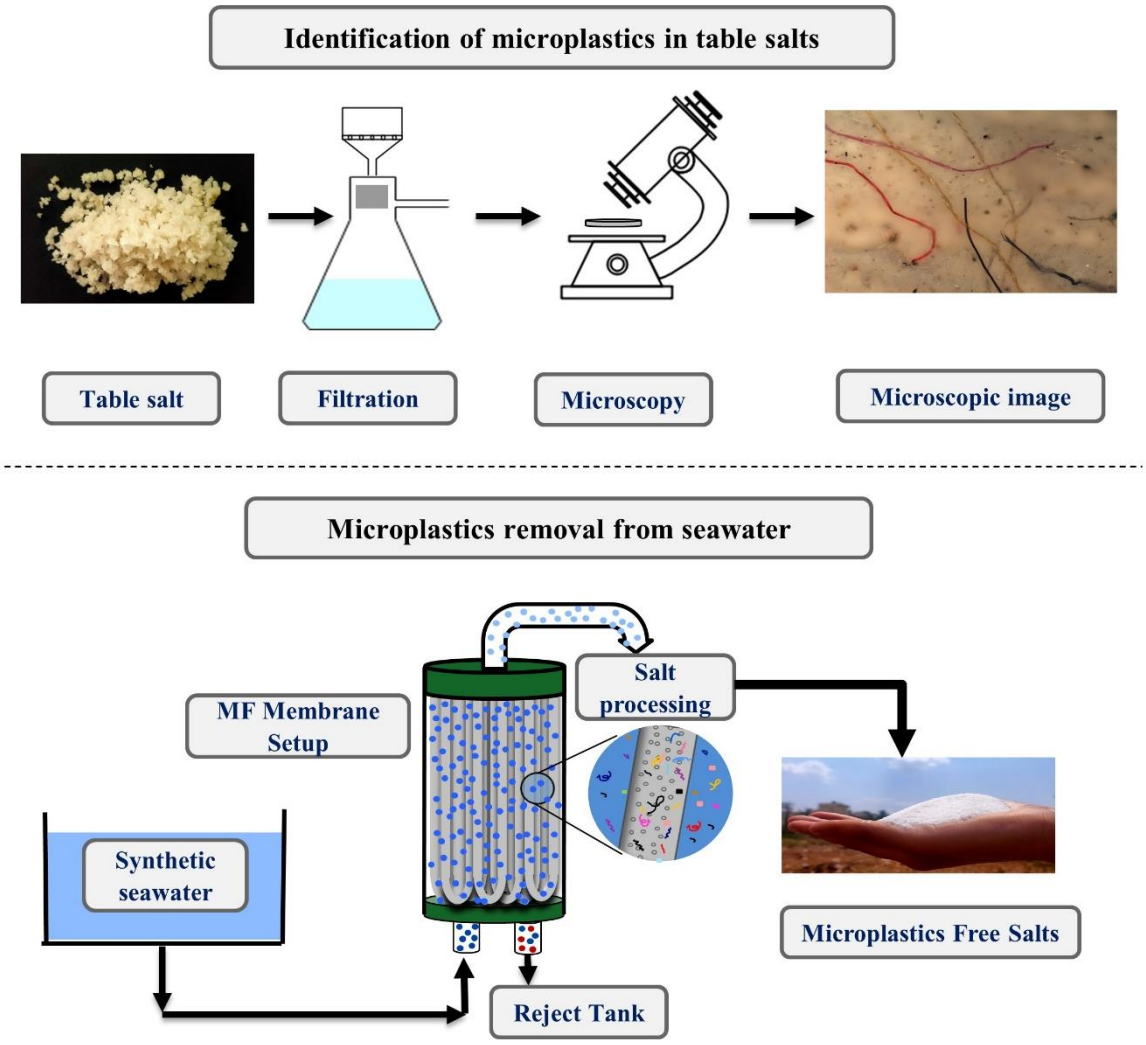
Figure 23. Morphological analysis of microplastics a) FESEM images and b) EDX spectra.

4.4. Summary

The results revealed that marine water and sediments from Indian beaches has been polluted with microplastics. The total mean microplastic abundance from all considered locations is 8.2 ± 3 particles /m^3 (marine surface water) and 664 ± 114 particles /kg (beach sediment). Malpe beach had the highest concentration of microplastics, whereas Kapu beach had the lowest, which is connected with a population, tourists, and industry around the shores. Based on morphology, the occurrence of fragment-shaped microplastics was dominant, followed by fibres. PE, PP, and PS microplastics were abundantly observed in all selected locations.

Chapter 5

5. Identification, extraction of microplastics from edible salts and removal of microplastics from contaminated seawater



Preface: *In this chapter, a cost-effective and straight forward protocol was adopted to identify microplastics in the Indian edible salts (rock and sea salt). Further, microplastics were removed using hollow fibre microfiltration membranes for the production of microplastics free salt. Additionally, membranes performance was checked and to avoid fouling and scaling on the membrane surface, the membrane were backwashed.*

5.1. Materials and Methodology

5.1.1. Materials

For the present study, twelve different commercial salts (rock and sea salts) sold in Indian markets were chosen. Details of the salts such as source, type, and origin are given in Table 1. Hydrogen peroxide (H_2O_2, 30%), sodium iodide (NaI), Ion chromatography cation multi-element standard VII and anion multi-element standard were purchased from Merck Life Science Pvt. Ltd, Mumbai, India. Nile red dye was procured from HI-Media Laboratories Pvt. Ltd, Mumbai, India. Nylon filter paper (pore size 0.2 µm, dia 25 mm) was purchased from Axiva Sichem Pvt. Ltd and PCTE filter paper (pore size 0.2 µm, dia 25 mm) was purchased from Sterlitech Corporation, USA. Microplastics removal was carried out by polypropylene based hollow fiber- microfiltration membranes (HF_MF). The HF membrane contains 2200 fibers, with inner diameter 0.35 mm, outer diameter 0.5 mm and length 0.38 m. The total membrane surface area was 2.64 m^2. The HF membrane was procured from HI-TECH Sweet Water Technologies Pvt. Ltd., Bangalore, India.

5.1.2. Extraction of microplastics from edible salts

Initially, 200 g of salt was taken in a 500 mL conical flask. 100 mL of filtered H_2O_2 was then added to the beaker for the digestion of organic compounds. For each brand of salt, three such replicates were made, and these bottles were covered and kept in an incubator shaker (Jeio Tech, Model No.ISS-3075R, Korea). The samples were agitated at 100 rpm for 24 h at 65 °C and followed by 48 h at 30 °C. After digestion, deionized water (800 mL) was added to each sample, and the samples were stirred and left for 24 h to dissolve salts. Further, the solution was filtered using vacuum filtration with PCTE/nylon filter paper. During filtration, If there is no cake layer on the surface of the filter paper, microplastics characterization can be done directly. In case of the formation of a cake layer that hinders the analysis of MP (presence of

impurities such as organic materials, sand, and mineral particles in the salt), the following step can be incorporated. The solution was transferred to a separating funnel and allowed the particles to settle for 24 h. It was seen that due to density difference, the low-density microplastics moved to the top side and high-density microplastics, minerals, and undissolved impurities stay at the bottom of the separating funnel. The supernatant salt solution was then transferred and filtered through filter paper, and the remaining solution left in the bottom was centrifuged at 5000 rpm. The centrifuged bottom solution was mixed with 4.4 M NaI solution (approximate density is 1.8 g/mL) to separate the high-density microplastics (> 95% recovery observed) (Herbort and Schuhen, 2017; Rocha-Santos and Duarte, 2015). The supernatant was then collected and filtered. The filtered membrane was dried at 40 °C for 12 h and then characterized. Flowchart for the microplastics extraction from salts is shown in **Fig. 24**. Experiments were conducted in laminar flow to avoid any airborne. Throughout the experiment, no plasticware was used, and special care was taken to avoid contamination during the whole analysis as much as possible.

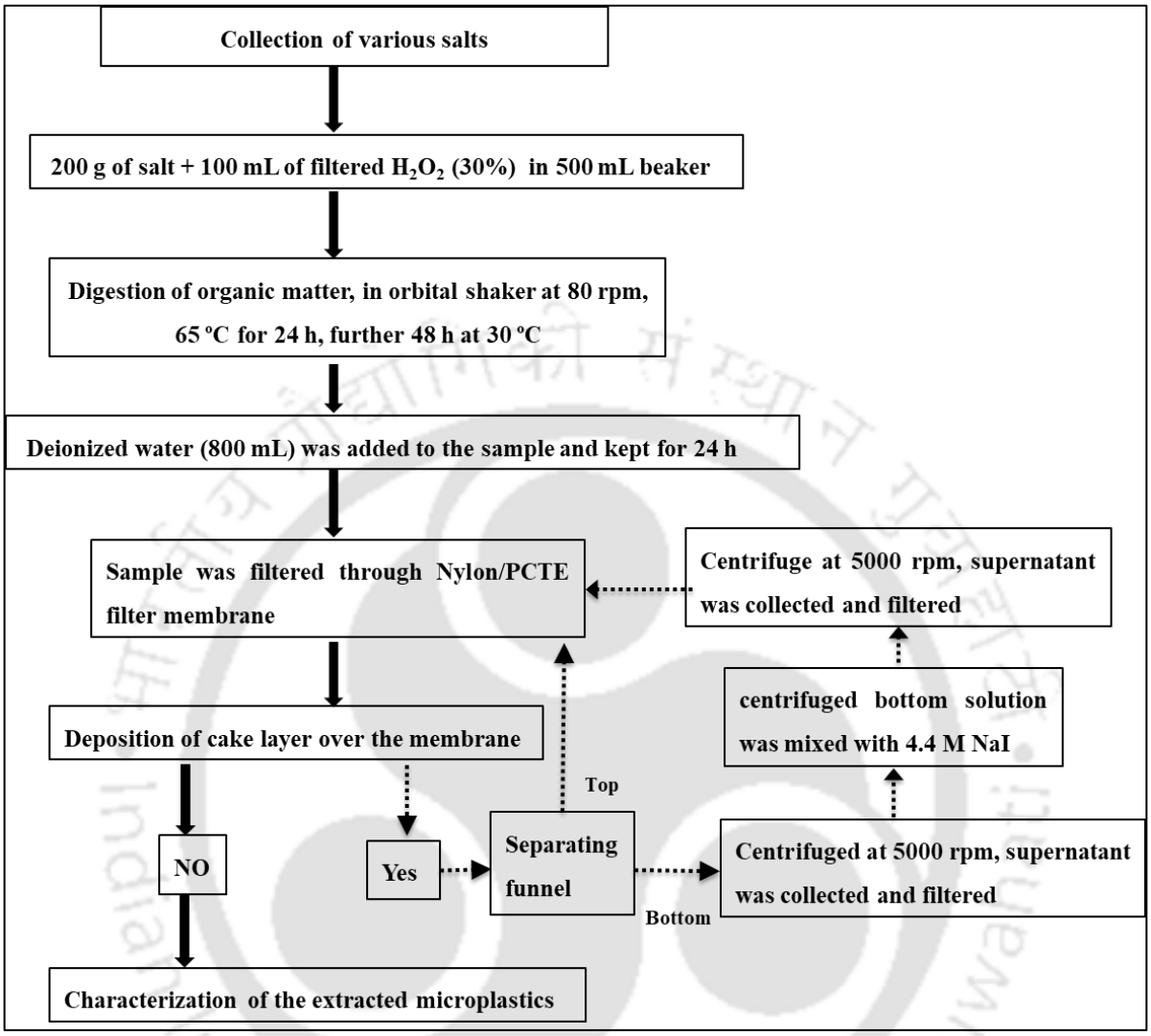


Figure 24. The protocol for microplastics extraction from edible salt.

5.1.3. Separation of MPs by microfiltration membrane

Removal of microplastics was carried out using the PP-HF-MF membrane. The flowsheet of the MF membrane set-up is shown in **Fig. 25**. The feed solution (synthetic seawater) was prepared by dissolving edible salt (highest microplastics contained salt) in 50 L deionized water (feed concentration maintained at ~ 35 g/L). The process was performed at room temperature, and the feed solution was first passed to the tank. A pump was used to maintain the feed pressure at 3 psi, which was connected to the HF-MF membrane through a control valve (CV_1)

in cross-flow mode operation. The pump speed controller and control valve (CV_2) was used to adjust the feed flow rate and pressure, respectively. The Permeate from the MF setup was collected in the permeate tank, and the flow rate was measured using a flow meter (Ascorp, intelligent flow totalizer). After 50 L of permeate collection, membranes were backwashed with deionized water for 2 min to prevent fouling, and the backwashed water was collected from the reject side. Experiments were repeated to check the membrane performance (flux and fouling) and microplastics removal. The collected permeate solution was filtered through PCTE filter paper using a vacuum filtration setup. The membranes and filter paper were further characterized using a microscope, FESEM and micro-Raman spectroscopy.

5.1.4. Light and fluorescence microscope

Identification and counting of microplastics were done with optical (light) and fluorescence microscope (Make: Carl Zeiss Model: Axio Scope.A1, Germany). Images were obtained with 10x, 20x, 50x and 100x lenses. According to their physical features, visual estimation of microplastics were carried out to identify their shape, size, and color. Most of the microplastics were white and transparent, which was difficult to identify them visually (Shim et al., 2016). Nile red (NR) dye readily adsorbs onto the microplastics and makes them fluorescent when irradiated under UV/Blue/Red/Greenlight (Erni-Cassola et al., 2017; Maes et al., 2017; Shim et al., 2016). The NR stock solution was prepared by using acetone (50 mg/L) and store at 4°C in an amber bottle. The working solution (0.5 mg/L) was prepared by diluting the stock NR with n-hexane. By using 200 µL of working solution of NR, the extracted microplastics were stained, and excess dye was washed with 200 µL of n-hexane and observed under a fluorescent microscope after air-dried.

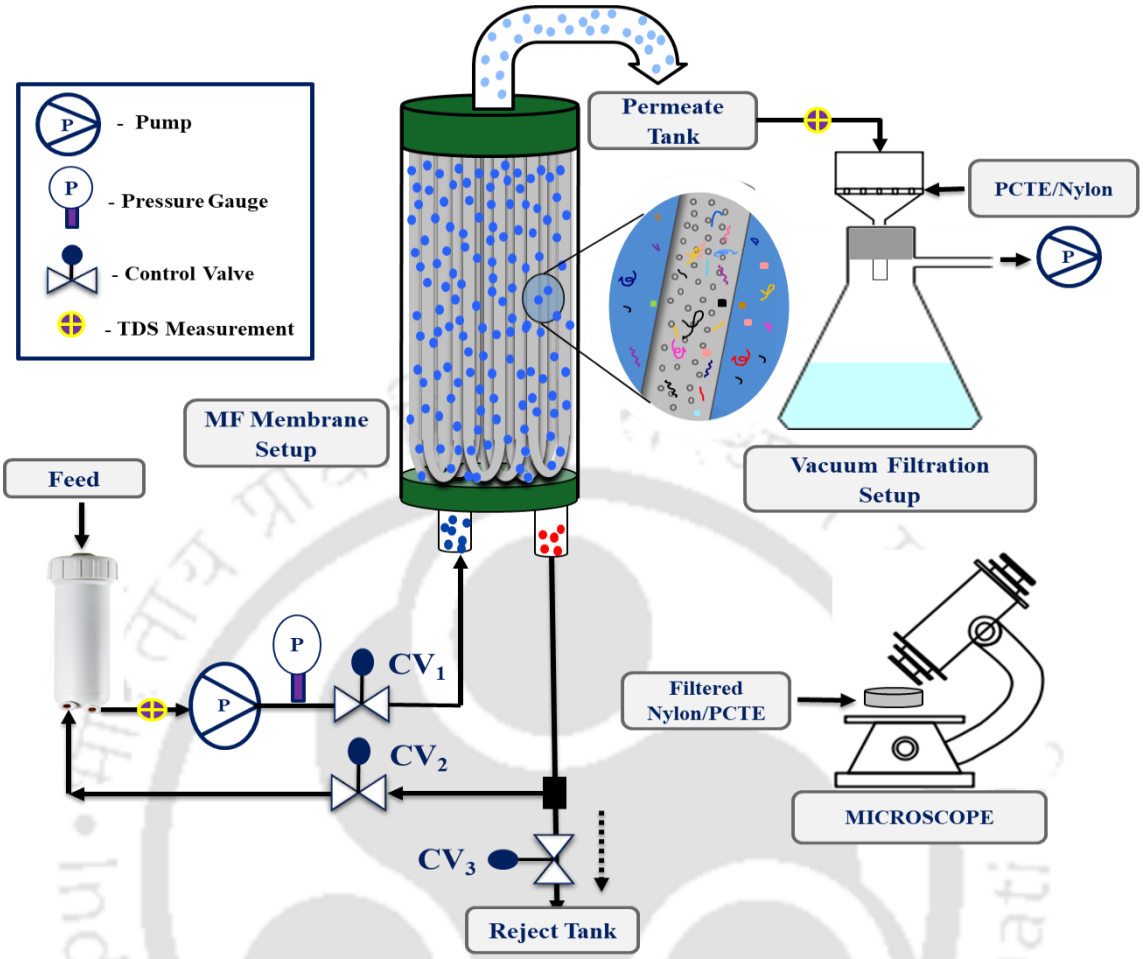


Figure 25. Flow diagram for removal of microplastic from synthetic seawater.

5.1.5. Raman spectroscopy

The compositional study of microplastics was done with micro-Raman spectroscopy (Make Horiba). micro-Raman spectroscope was attached with a 20x and 100x objective lens (NA=0.55), and Raman laser of 532 nm and 633 nm (5 mW; grating of 1800 lines/mm) with adjustable laser power. Spectra were scanned for the range of 400 cm^{-1} – 3500 cm^{-1} with an acquisition and accumulation time of 10 s and 2 s, respectively. The polymers were identified from the raw Raman spectra and processed using Bio-Rad KnowItAll software.

5.1.6. Field emission scanning electron microscope (FESEM)

The membrane morphology and composition (before the experiment and after the experiment) were characterized by FESEM (Sigma 300, Zeiss) attached with an energy dispersive x-ray spectrometer (EDS). The MF membrane samples were vacuum-dried at 50 °C for 24 h and sputter-coated with a thin gold layer for conduction. Images were obtained in Inlens mode at different magnification with an acceleration voltage of 3/5 kV.

5.1.7. Ion chromatography

The salt solution composition (anion and cation concentrations) was determined using ion chromatography (925 Eco IC, Metrohm) with a conductivity detector attached with Metrohm A Supp 5-250/4.0 and Metrosep C 6-150/4.0 analytical columns.

5.2. Results and Discussion

5.2.1. Identification of MPs in salt samples

All twelve Indian edible salt brands contaminated with microplastics. A variety of microplastics with varying shape, color, size and polymer types were found in all salt brands, which are shown in **Fig. 26**. Microplastic size ranging from ≥ 20 µm to ≤ 5 mm were found. White and transparent microplastics visual identification was carried out with the fluorescence microscope using NR dye (Maes et al., 2017; Shim et al., 2016) as shown in Fig. 3b and 3c). **Table 11** shows the origin, source and number of microplastics present in the salt samples. A high count of microplastics were observed in IN-8 (1832 ± 40 particles/kg) and fewer counts in IN-11 (275 ± 25 particles/kg) salt samples. Unrefined (coarse) sea salts (IN-8 and IN-9) contained a large number of microplastics with bigger particle size when compared to refined salts. IN-10, IN-11, and IN-12 are refined rock salts which showed more of flat sheet microplastics.

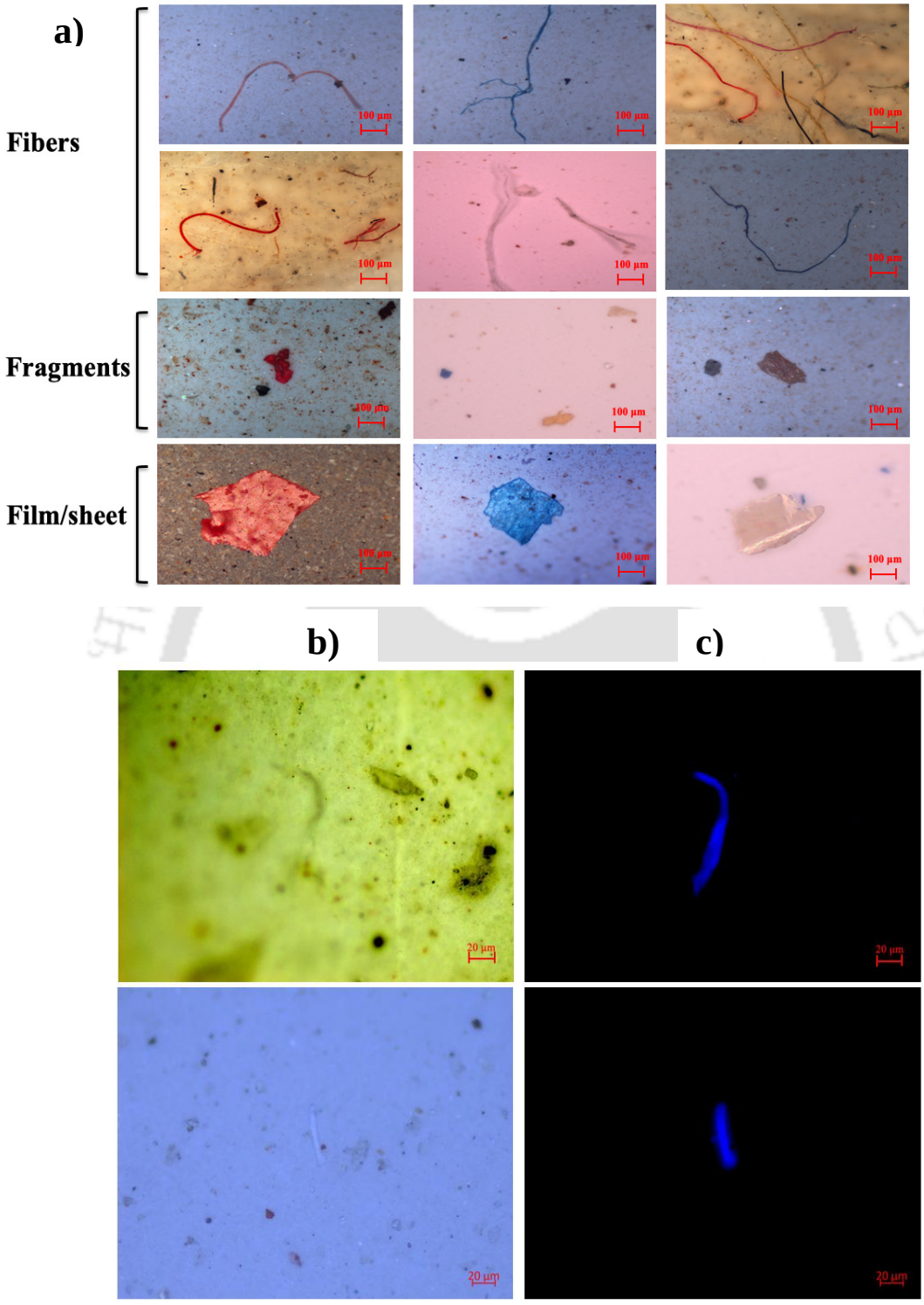


Figure 26. Microscopic images of some of the extracted MPs from various salt brands: a) and b) optical microscopic images and c) florescence microscopic images.

Table 11. Microplastics in edible salts.

Sample ID	Source	Type of salt	Produced/ Procured	Cake forms on the membrane filter	Particles /kg: Optical microscope	Particles/kg: Florescence microscope
IN-1	Sea salt	Refined	Gujarat, India	No	1326 ± 30	1525±25
IN 2	Sea salt	Refined	Gujarat, India	No	1291 ± 35	1464±30
IN 3	Sea salt	Refined	Gujarat, India	Yes	1428 ± 30	1634±20
IN 4	Sea salt	Refined	Gujarat, India	No	1549 ± 25	1755±18
IN 5	Sea salt	Refined	Gujarat, India	Yes	1604 ± 20	1878±40
IN 6	Sea salt	Refined	Gujarat, India	Yes	1544 ± 40	1736±25
IN 7	Sea salt	Refined	Gujarat, India	Yes	1540 ± 35	1865±22
IN 8	Sea salt	Unrefined	Tamil Nadu, India	Yes	1832 ± 40	2248±36
IN 9	Sea salt	Unrefined	Tamil Nadu, India	Yes	1760 ± 35	1910±15
IN 10	Rock salt	Refined	Mumbai, India	Yes	282 ± 30	347 ± 20
IN 11	Rock salt	Refined	Haryana, India	Yes	275 ± 25	313 ± 26
IN-12	Rock salt	Refined	Gurgaon, Delhi	Yes	308 ± 24	387 ± 30

5.2.2. Classification of microplastics

In terms of shapes, fragment type microplastics were abundant in all brands of salts, with an average of 44%, followed by fibers 39%, film/sheet 13%, and pellet 4%. The distribution of microplastic particles in terms of colors was in the order of white (20%), transparent (18%), blue (11%), red (9%), green (8%), brown (8%), black (7%), purple (6%), pink (5%), orange (4%), yellow (2%) and grey (2%). The percentage of the microplastics in the size ranges of 10 - 100 µm, 0.1-1 mm, 1-2 mm, 2-3 mm, 3-4 mm, and 4-5 mm are 38%, 32%, 17%, 9%, 3%, and 1% respectively as shown in **Fig. 27**. It was observed that in all salt samples, white and transparent colour microplastics were abundant, and more than half of the microplastics were in the range of 10 µm to 1 mm size.

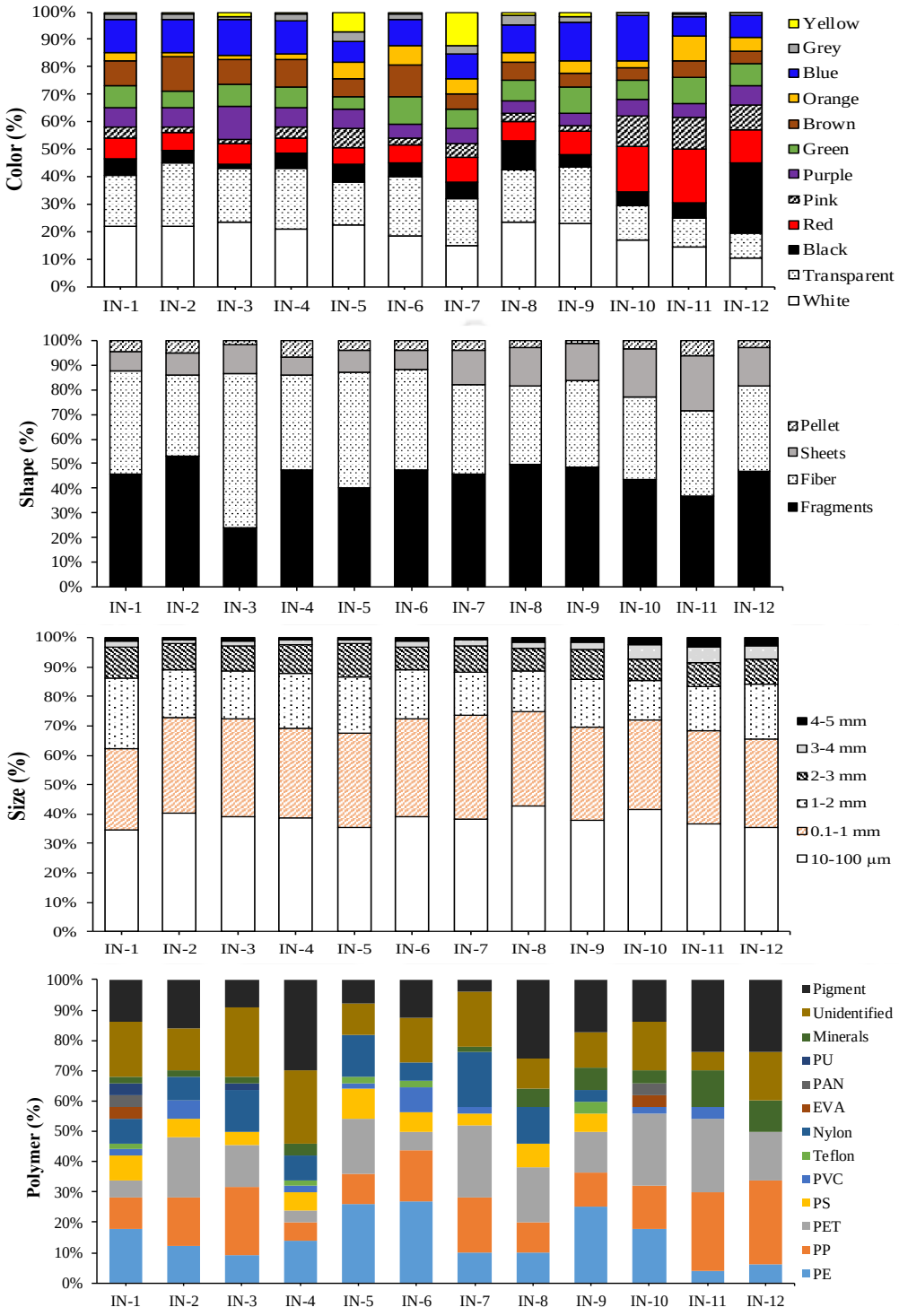


Figure 27. Microplastics detected in edible salts are classified based on a) colour, b) shape, c) size, and d) composition profile by polymer type.

5.2.3. Raman spectroscopy analysis

A total of 36 samples, including replicates, were verified through µ-Raman spectroscopy. 50 microplastics of each salt samples were taken and analyzed. The µ-Raman spectroscopy results of the microplastics are shown in Fig. 5. Overall, 60% of particles were identified as synthetic polymers, 14% particles were unidentified because of their unclear spectra, 19% particles matched with color pigments which were used in the plastic production, and about 7% matched with silicate mineral particles. Major polymers such as PE, PP, PET, Nylon, PS and PVC were found. The average composition of the microplastics in sea salts was PE (26%) > PP (20%) > PET (20%) >PS (10%) > Nylon (15%), and > PVC (4%). Whereas, in rock salts it was PP > (41%) PET > (37%) PE > (15%) and PVC > (3%). The other polymers were rarely found and had an average contribution of about < 10%. Additionally, the obtained spectra were also compared with the earlier report (Dowarah and Devipriya, 2019; Gündoğdu, 2018). A comparison of average profiles of microplastics in Indian edible salts based on color, shape, size, and polymers are analyzed (**Fig. 28**).

Along with polymer, pigmented particles were also identified (**Fig. 29** and **30**). Pigments spectra interference with polymer spectra hindered the identification of microplastics (Karami et al., 2017) (Seth and Shriwastav, 2018). Furthermore, raw Raman spectra was hard to analyze, as polymers underwent a different type of degradation and biofilm formation (Dowarah and Devipriya, 2019). Compositional characteristics of the microplastics have been done with µ-Raman spectroscopy, which can identify < 20 µm microplastics compared to FTIR (Schymanski et al., 2018). PE, PP, and PET were the predominant microplastics in the salt samples, which was reliable with reports on their widespread distribution in the marine environment (Karami et al., 2017). Almost 20% of the isolated particles were identified as

color pigments (Irgalite blue, indigo, burnt umber, and mortoperm blue) (Dowarah and Devipriya, 2019; Horton et al., 2017a).

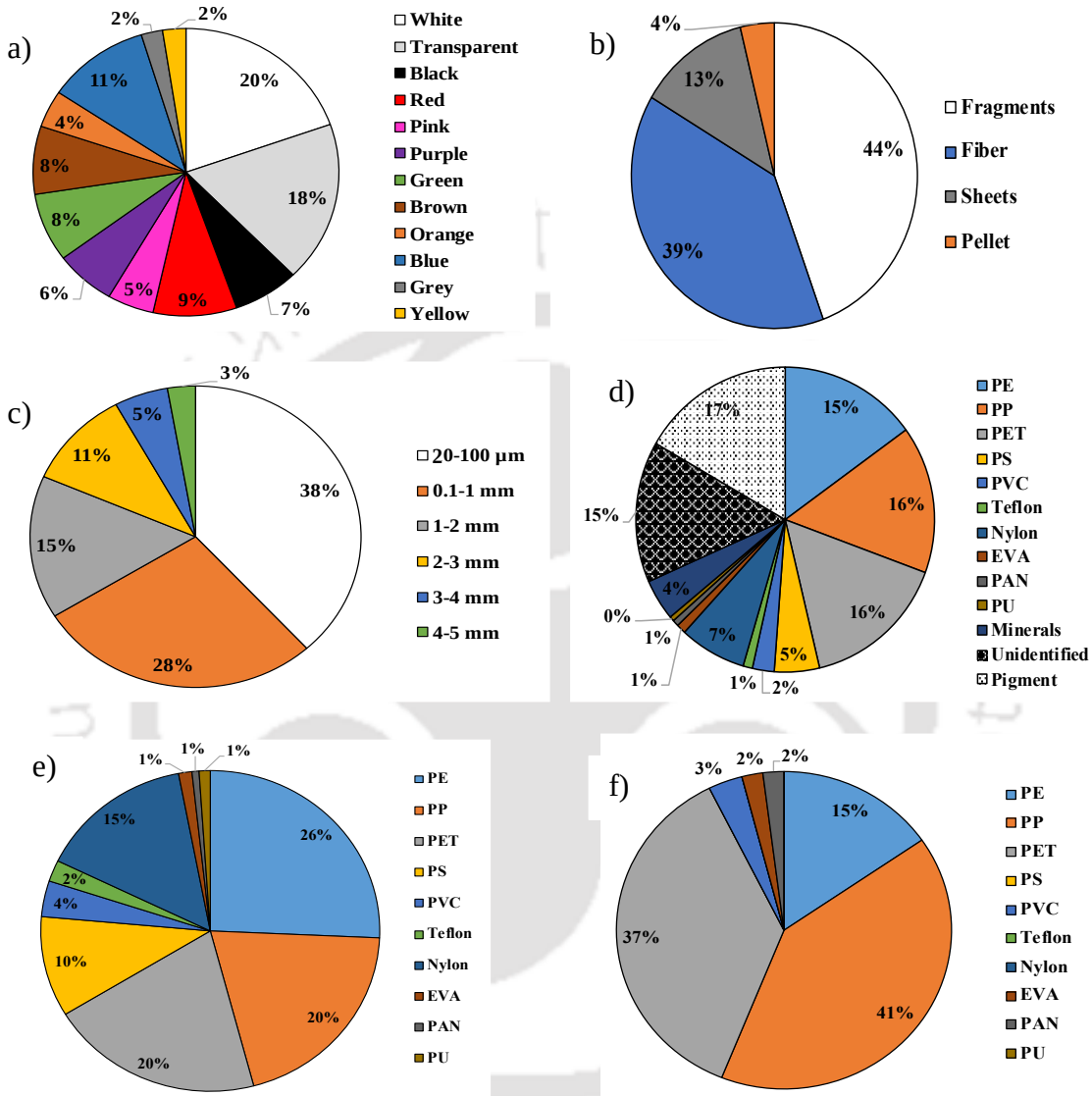


Figure 28. Comparison of average profiles of MPs in Indian edible salts: a) colour, b) shape, c) size, and d) polymer, e) synthetic polymer in sea salts, f) synthetic polymer in rock salts.

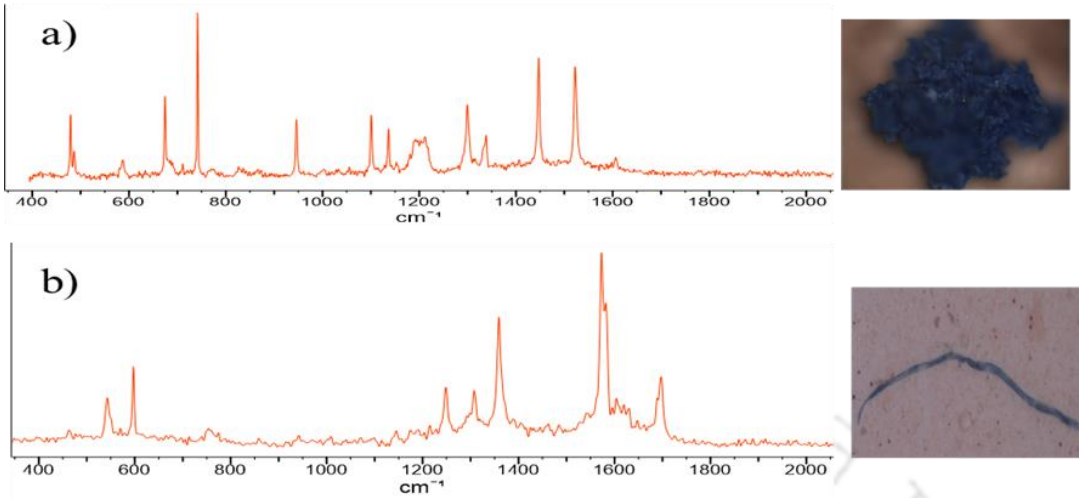


Figure 29. Raman spectra of the extracted polymer particles and corresponding images. a) Irgalite blue, b) Indigo.

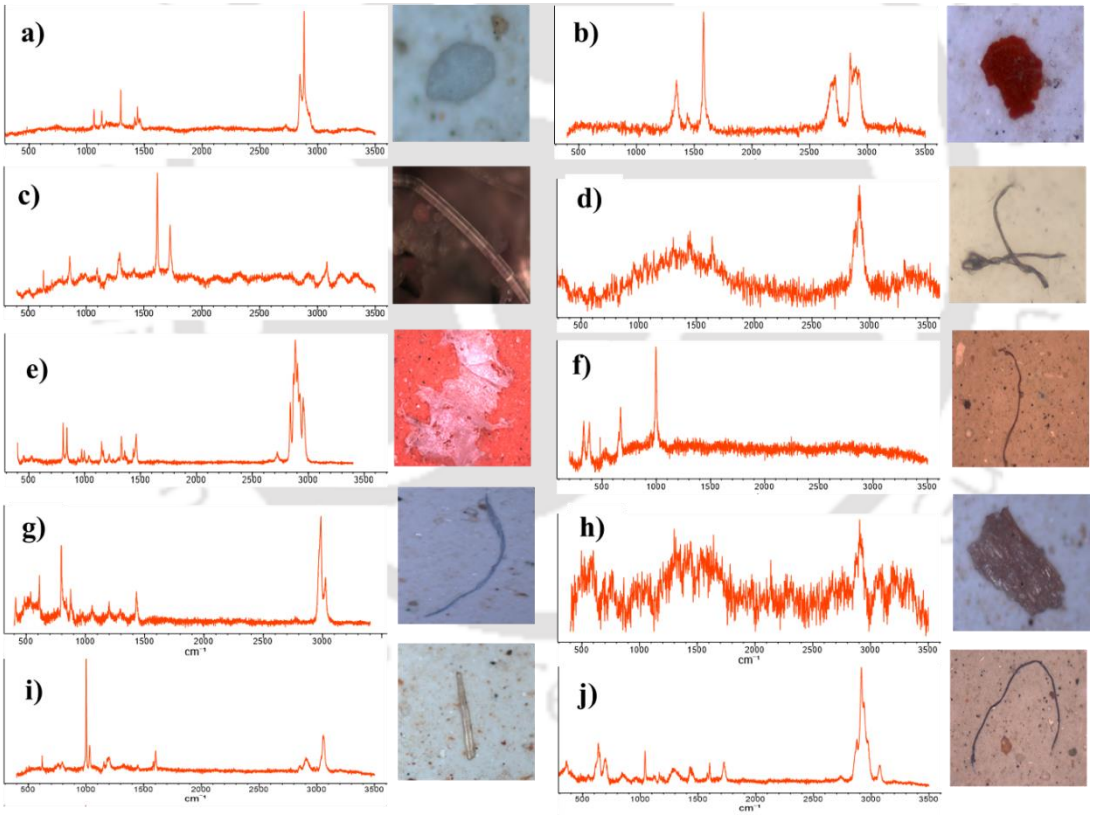


Figure 30. Raman spectra of the extracted polymer particles and corresponding images. a) PE, b) Poly (ethylene-co-vinyl acetate, c) PET, d) Nylon, e) PP, f) poly 4-vinylpyradine, g) PVDF, h) PU, i) PS, j) PVC.

5.2.4. Separation of microplastics using HF-MF membrane

A simple micro-filtration technique was used for the removal of microplastics from synthetic seawater. For the experimental purpose, unrefined IN8 (1.75 kg) salt was chosen, which dissolved in 50 L distilled water to produce synthetic seawater. The prepared feed solution (5 L) was filtered through nylon filter paper and observed under the microscope, and the images are shown in **Fig. 31 a)**, which contains approximately 403 microplastics per 5 L (~ 2300 particles/kg). The synthetic seawater was passed through the HF-MF setup maintained at 3 psi pressure. Permeate collected was passed through a nylon filter and observed in the microscope. The result is shown in **Fig. 31 b)**. Varying from 0-3 numbers and size less than 10 μm fibres were observed, which is maybe due to airborne contamination. Pure water permeability after the experiment was slightly reduced from 66. 5 to 65.3 LPH at 3 psi pressure due to the deposition of the foreign particles over the membrane, and the permeate flow rate was also continuously decreased with respect to time (Verma and Subbiah, 2019). Every 50 L permeate collection, membrane were backwashed for two minutes to prevent fouling. Permeate flow rate and membrane life increases with membrane backwashing (**Fig. 32**).

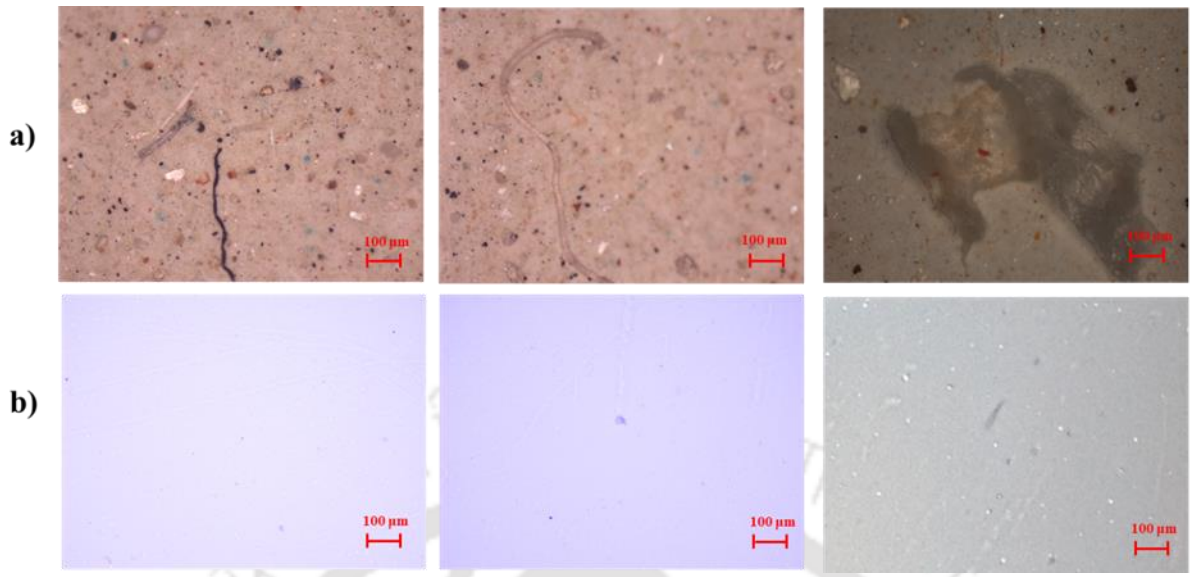


Figure 31. Microscopic images of some extracted MPs in a) synthetic feed solution and b) permeate side MPs.

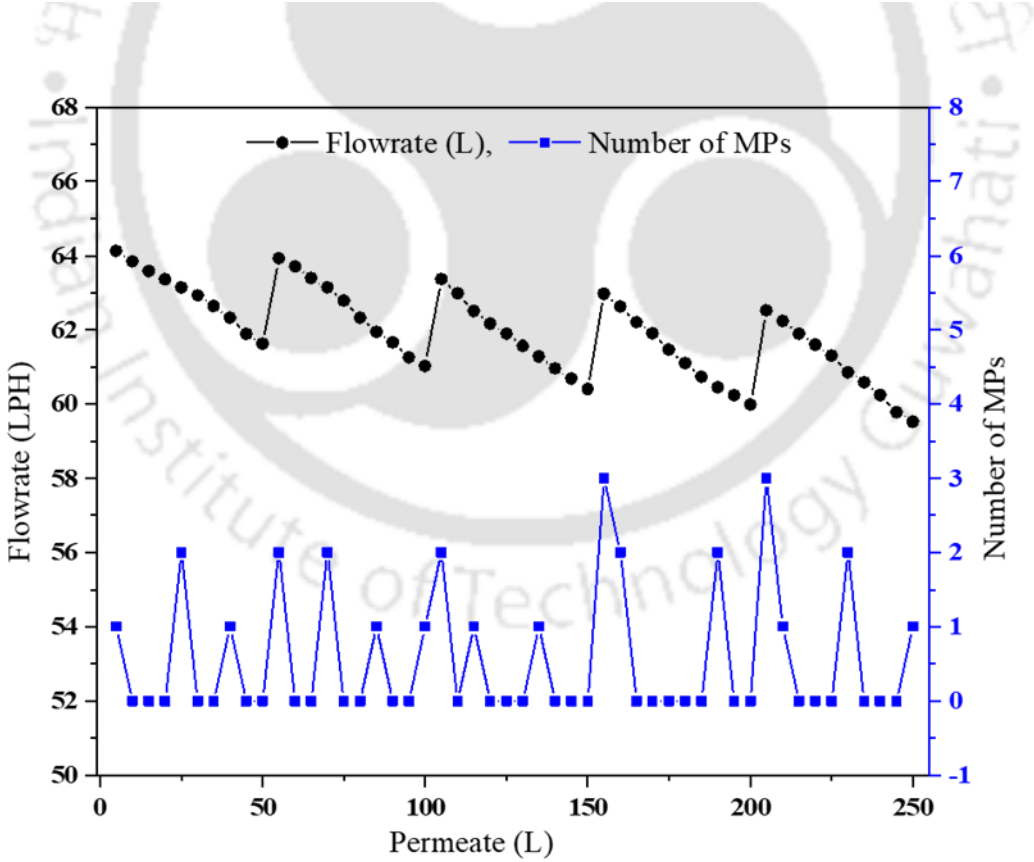


Figure 32. Effect of backwash on microfiltration configuration and number of MPs in permeate.

5.2.5. Ion Chromatography (IC)

When synthetic seawater was passed through the HF-MF membrane, the permeate collected was analysed using Ion Chromatography. Sample solutions were prefiltered through a 0.2 μm membrane filter and diluted for the analysis (200x and 500x). It is widely accepted that apart from the prevailing concentration of NaCl, saltwater also contains trace amounts of calcium, potassium, and magnesium salts. These salts are either chlorides or sulphates and constitutes about 5%-10%. The standard solutions of both anions and cations were used for making the calibration curve, and the linearity of the calibration graph was in the concentration range of 0.1 to 10 mg/L. Prepared synthetic feed solution contained Na^+ and Cl^- concentration of 11.22 g/L and 20.88 g/L, respectively. When the feed is passed through the HF-MF membrane, the feed concentration at the permeate side decreased initially, however, after 15 minutes, the feed concentration becomes equal to the permeate concentration (**Fig. 33**).

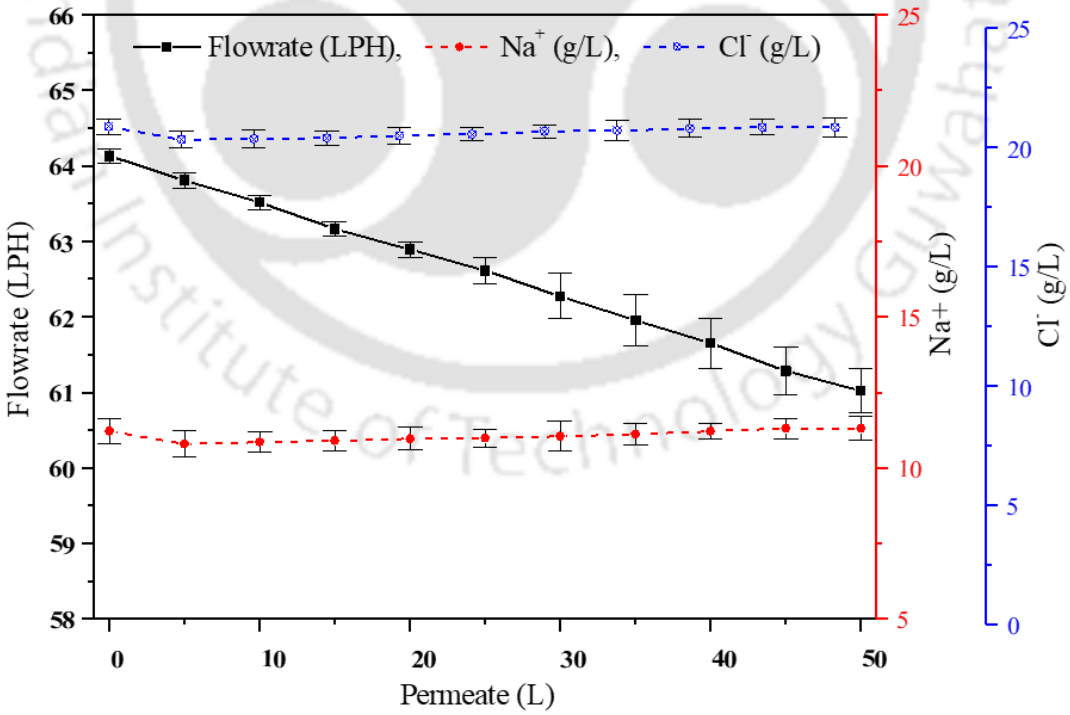


Figure 33. Salt solution concentration determined with respect to permeate (time).

5.2.6. FESEM and EDX

The surface morphology of the virgin and experimented membranes were evaluated by FESEM. FESEM images clearly show that the membrane is porous at the inner and outer surface. The membrane was in excellent condition even after backwashing (**Fig. 34**). Furthermore, the Energy-dispersive spectra show that there was no deposition of salts over the membrane.

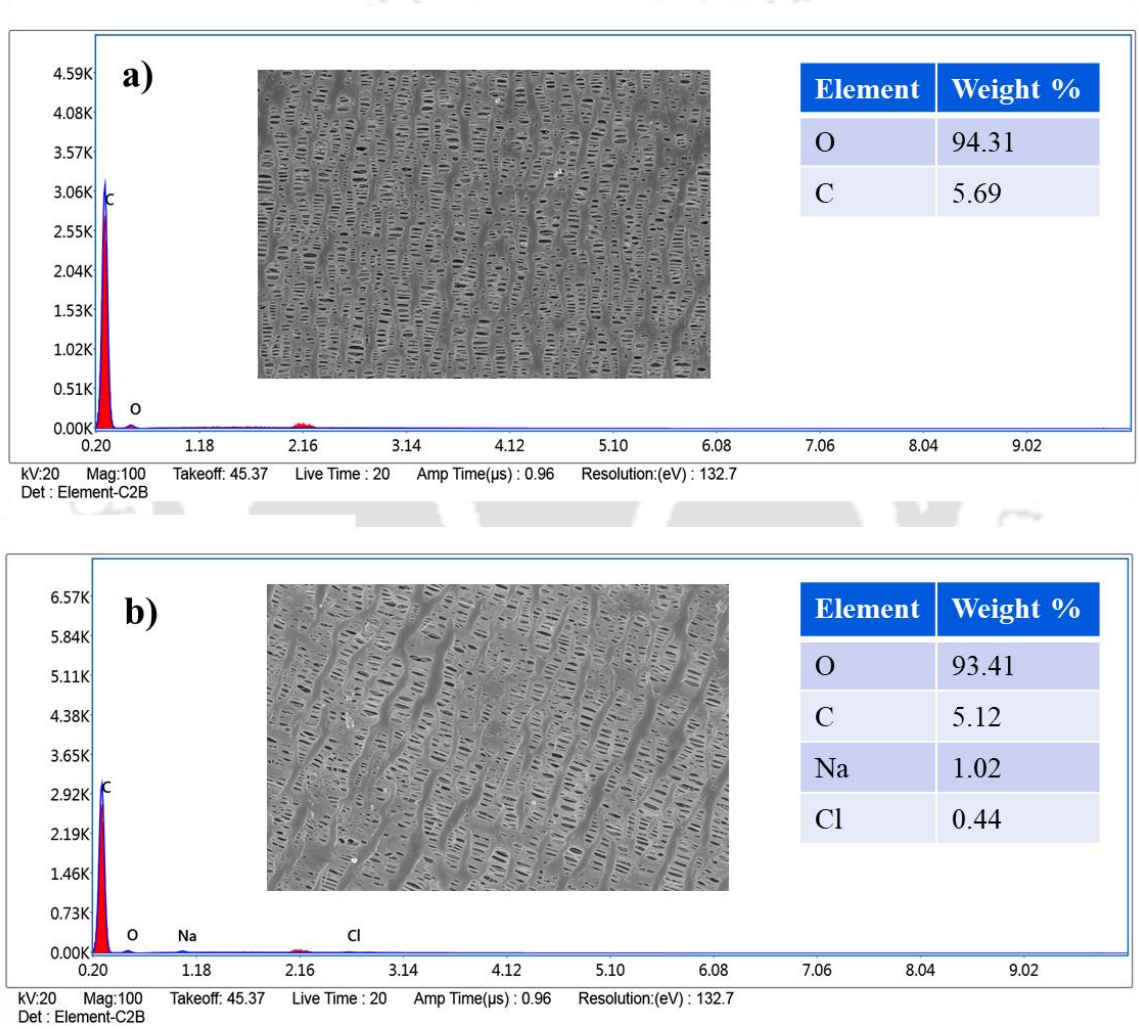


Figure 34. FESEM-EDX analysis of a) virgin PP-HF membrane and b) PP-HF membrane after filtering 250 L of synthetic seawater.

5.3. Discussion

Various studies reported different methods to extract microplastics from edible salts. However, the efficiency and cost were not up to the mark (Karami et al., 2017). Mostly the loss of microplastics was observed in the centrifugation step (Iñiguez et al., 2017). In the present study, extracted more than 95% of microplastics from the solution by density separation with the addition of NaI followed by centrifugation. The number of microplastics in sea salt and rock salt was 1291 ± 35 - 1832 ± 40 particles/kg, and 275 ± 25 - 308 ± 24 particles/kg respectively. The number of microplastics in sea salts is higher (approximately five times) than the rock salts due to its extensive distribution of anthropogenic microplastics in the marine environment (Courtene-Jones et al., 2017). The majority of the particles were less than 1000 μm in size as similarly observed by Yang et al. (2015). We also estimated that the consumption of microplastics through Indian sea salts is about 2700 - 4200 particles per year and the same for rock salts was nearly 520 - 760 particles per year. Further, the health effects by the ingestion of microplastics on humans were reported extensively (Galloway, 2015; Oßmann et al., 2018b; Rist et al., 2018; Wright and Kelly, 2017). Health issues may arise due to the following ways: i) ingest of plastic particles, ii) leaching of plastic additives to the bloodstream, and iii) desorption of the absorbed persistent organic pollutants (POPs) and biological pathogens on plastic particles (Andrady, 2017; Revel et al., 2018; Robin et al., 2020; Zhang et al., 2017).

The present results confirmed the occurrence of microplastics in edible salts and are in line with the earlier reports from various parts of the world as compared in Table 2. Edible salt is obtained in 3 different ways: solar evaporation, mining of rock salts, and well brines. Almost everywhere, the harvesting of salt from seawater is carried out with solar evaporation, which gives 99% NaCl (Shreve Norris, 1977). Processing of salt can be divided into two approaches i) coarse salt and ii) fine salt. (i) saltwater pumped to the artificially created ponds and

evaporated up to its saturation point in open basins. Impurities present in the brine are drained off. The concentrated brine is crystalized, which is then gathered by mechanical harvesting machines (Akridge, 2008; Ossandón et al., 2010). Coarse salts can be further packed for distributions; (ii) to increase the quality of salt, coarse/crude salt is washed by a highly saturated brine solution to remove suspended solids and again dissolved in water to remove impurities which are trapped inside the salt crystals (Mg and Ca compounds are removed by the chemical-physical process to get 99.6 % purity) (Rathnayaka D., 2014). Mixed with a known quantity of iodine, it is then crystalized and fine particle salts are packed. microplastics can be entirely removed by adopting a microfiltration method either in the first approach, before pumping the saltwater to the ponds or in the second approach when salt dissolves in water. This method is capable of eliminating microplastics having a size greater than 10 μm from the saltwater. The study reveals that PP-MF membranes can be used to remove microplastics based on its low cost, high packing density, pre-existing configuration, and manufacturers.

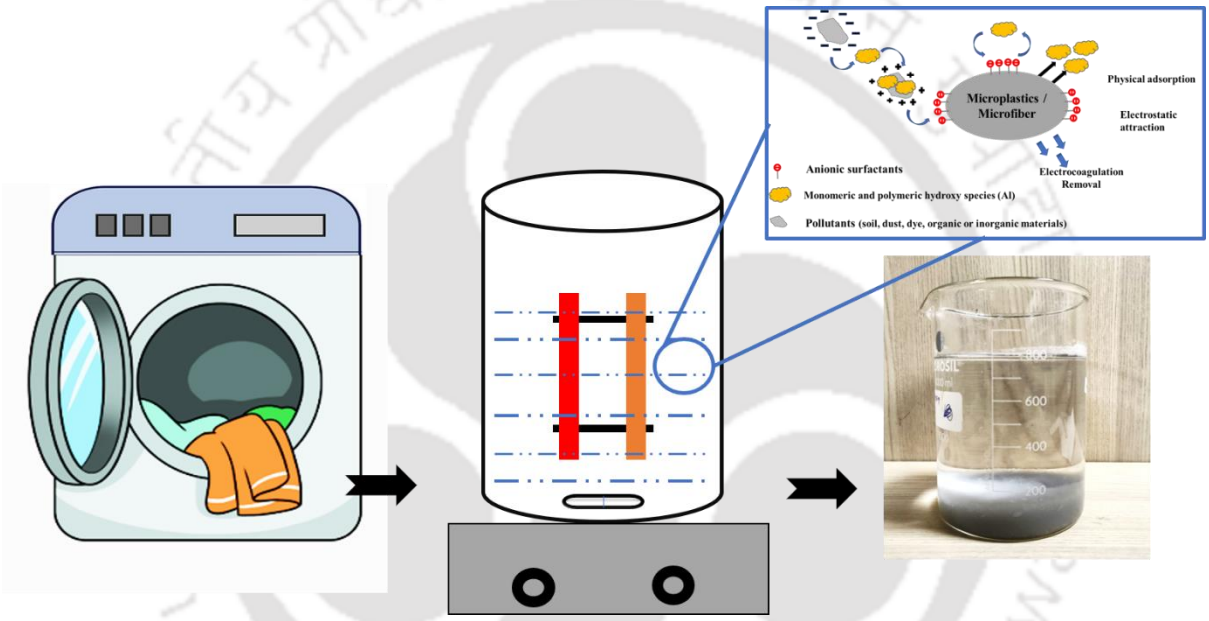
5.4. Summary

The visual assessment was performed to identify the shape, size, number, and colour of microplastic particles using light and fluorescence microscopy. Indian edible salts (derived from sea or rock/Himalayan salts) are contaminated with microplastics. The Nile red dye makes it simple to count white and transparent microplastics. A relatively high number of microplastics were found in sea salts rather than rock salts. Sheet-type microplastics with size 1 ~ 4 μm were observed in rock salts. The most common microplastics were PE, PP, PET, nylon, and PS. A total of 99.3 % of microplastics were effectively removed from synthetic seawater using a hollow fiber membrane, and the Ion Chromatography result showed that membranes have no impact on salt concentration. As a result, MF membranes can be used to

remove anthropogenic pollutants from seawater. Microplastics, that contaminate water sources, can negatively impact the quality of salt. Membrane technology offers a promising solution for addressing the issue of microplastics in salt production. By incorporating membrane technology into salt production processes, manufacturers can enhance the purity of their final product, meeting quality standards. This application of membrane technology aligns with environmental and quality concerns, offering a sustainable approach to mitigate the impact of microplastics on salt production.

Chapter 6

6. Removal of microplastics from the laundry effluent by electrocoagulation method



Preface: *Microplastics and surfactants are generally recognized as emerging contaminants with complicated ecotoxicological impacts. Laundry wastewater is one of the primary routes for microplastics and surfactant to enter the aquatic environment. In this chapter, quantified presence of microfibers and surfactant concentration in laundry wastewater. Further, the removal of microplastics and surfactants from laundry wastewater by electrocoagulation will be discussed.*

6.1. Materials and methodology

6.1.1. Materials

All the chemicals used in the experimental study were Analytical or laboratory reagent grade. Chloroform ($CHCl_3$), Ethanol (C_2H_6O), Hydrogen peroxide (30 % H_2O_2), Hydrochloric acid (HCL), Methylene blue dye ($C_{16}H_{18}ClN_3S$), Phenolphthalein indicator, Sodium Hydroxide (NaOH), Sodium Chloride (NaCl), Sodium dodecylbenzene sulfonate (SDBS), Sodium Lauryl Sulfate (SLS), Sodium carbonate ($NaCO_3$), Sodium bicarbonate ($NaHCO_3$), Sulfuric acid (98 % H_2SO_4) and Distilled water. The electrocoagulation technique was carried out using aluminum electrodes. In order to maintain the effectiveness of the electrodes, diluted HCl (0.01 M) was used to clean the electrodes.

6.1.2. Washing machine discharge collection

Polyester lining fabrics were brought from different stores in India. Fabrics were utilized for household work then fabrics were (1.5 m × 1.5 m, three clots materials, 2 Kg) washed. A top-load convection washing machine and liquid detergent were used. The quick program option in the washing machine was used to conserve energy and water (15 min, 45 L of ambient temperature water). Regularly, the discharge from washing machines was collected from residential areas, and it was determined that the sample's average residual turbidity was 145 NTU (Lutron, TU-2016). The washing machine effluent was turbid and muddy in colour. Before beginning the experiment, the washing machine went through several blank washes to clean it.

6.1.3. Sample preparation

The fabrics were measured in terms of length and weight and classified based on materials before starting the experiments. After washing fabrics, machine outlet samples were collected and stored in glass containers at 4 ºC. Further stored samples were characterized and utilized for electrocoagulation tests. To calculate the number of MFs present in washing machine outlet samples, 1 L washing machine outlet samples were taken and digested with hydrogen peroxide to remove organic and dirt materials. After digestion, samples were filtered with polycarbonate track etching/nylon filter paper. To prevent contamination, the filters were always stored in glass Petri dishes. Subsequently, the filtered membrane was dried at 50 °C for 24 h and then characterized. Complete sample filtration was conducted in laminar flow to avoid any airborne microplastics. Throughout the experiments, no plasticware was used, and special precautions were taken to avoid contamination.

6.1.4. Electrocoagulation experiment

The electrocoagulation operation was carried out using an electrochemical setup (semi-batch process) built of acrylic material with a 1.2 L volumetric capacity. Both of the electrodes were made with aluminium sheets having a surface area of 6.42×10^{-3} m^2 and dimensions of 0.088 m × 0.073 m. In order to deliver consistent current, anode and cathode electrodes were connected to a direct current (DC) power source (0-30 V/10 A, DC Crown regulated power supply). Induced polarization takes place when a voltage is applied to the electrode ends, leading to the monopolization of the whole assembly. The distance between electrodes was maintained at 5 mm. A detailed schematic diagram of the electrocoagulation system is shown in **Fig. 35**. Several current densities, ranging from 100 to 400 A/m^2, were applied. And observed that the removal effectiveness of the process approaches saturation when trials are

carried out at current densities of more than 300 A/m^2. These investigations demonstrated a comparable decrease % in terms of pollutant concentration. However, current densities of less than 200 A/m^2 were unable to completely eliminate all the contaminants that were over the acceptable limit.

As a result, the experiment's ideal current density of 300 A/m^2 was chosen. Similarly, an

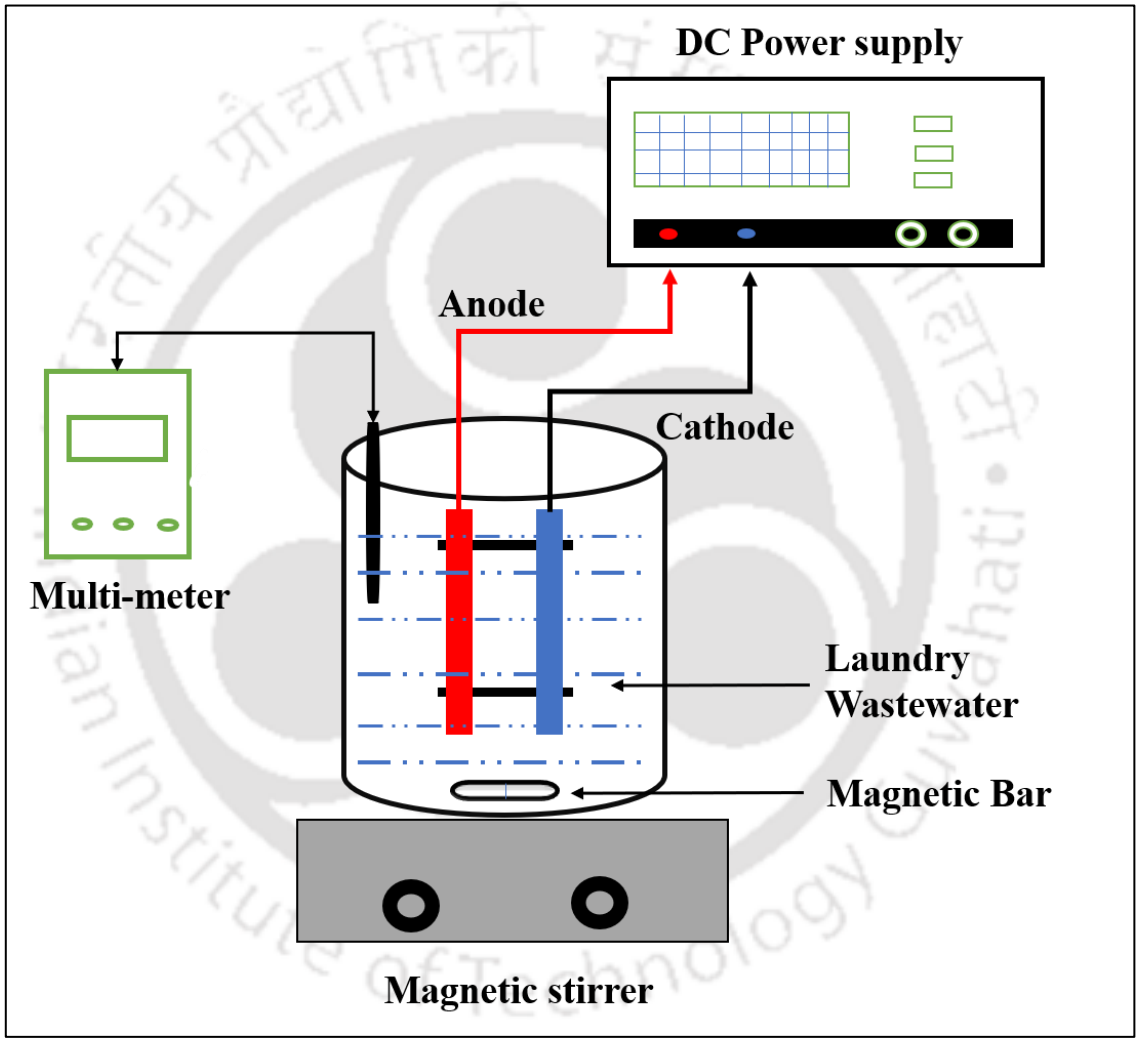


Figure 35. Schematic diagram of the electrocoagulation set up.

electrocoagulation duration of 25 minutes was chosen as the best working time since continuing the trials beyond this point results in negligible pollutants removal. As a result, the optimum conditions for all following experiments were 300 A/m^2 (current density) and 25 min

(treatment duration) considered. To properly spread the coagulant matter generated by anodic oxidation, a magnetic stirrer with a continuous stirring speed of 180 rpm was used. The experiments were carried out using aluminium electrodes. The entire experimental and analysis process took place at room temperature. The after-electrocoagulation process samples were allowed to settle overnight, and then the supernatant solution was decanted for further MFs and surfactant analysis. The reactions that take place during electrocoagulation (at the anode, cathode, and bulk medium) are detailed below (Das et al., 2021a).

At anode

$Al(s) \rightarrow Al^{3+}(aq) + 3e^{-}$ (1)

$2H_2O \rightarrow O_2(g) + 4H^{+}(aq) + 3e^{-}$ (2)

At cathode

$2H_2O + 2\,e^{-} \rightarrow H_2(g) + 2OH^{-}(aq)$ (3)

In solution

$Al^{3+}(aq) + 3OH^{-} \rightarrow Al(OH)_3(s)$ (4)

After settling the sludge, supernatant solution was taken gently to further analyse MFs, anionic surfactants and chemical oxygen demand (COD) concentrations. The sludge was separated and then dried for 24 hrs at 45 °C in a vacuum drying oven. Each experiment was repeated three times. To study the influence of initial pH, the pH values were adjusted by using diluted H_2SO_4 and NaOH solutions.

6.1.5. Visual inspection, quantification, chemical composition, and morphological characterization

After the sample preparation, filtered membranes were subjected to visual sorting under an optical microscope, which is the most generally used approach for identifying MFs. Microfiber particles were categorized based on their forms and sizes. To determine the functional groups contained in the obtained samples, attenuated total reflectance - Fourier transform infrared spectroscopy (ATR-FTIR) analysis of the MFs sample was performed using PerkinElmer Spectrum II. This type of spectroscopic investigation enables the accurate identification of the smallest synthetic plastic particles. After each electrocoagulation experiment, the sludge was recovered and dried at 45 ºC for 48 hours and further examined with a field emission scanning electron microscope (FESEM; model: Gemini 300; manufacturer: Carl Zeiss) to study the morphological characterization of the sludge and MFs.

6.1.6. Anionic surfactant

Anionic surfactants were assessed via spectrophotometric techniques using methylene blue as the active substance, and this standard method was utilized to determine surfactant concentration in tap water (IS 3025: Part 78: 2021, which is identical to ISO 16265: 2009). The technique works by forming an ionic pair between anionic surfactants and methylene blue. Anionic surface-active agents form salts in reaction with methylene blue in an alkaline medium. These salts are extracted with chloroform and acid treatment of the chloroform solution. Later, the absorbance of the isolated organic phase at the maximum absorption wavelength of 650 nm was measured by a double-beam UV spectrophotometer (Shimadzu, UV-2600) (Barz et al., 1996; Jurado et al., 2006; Kruszelnicka et al., 2019). Identification annexure of surfactant concentration using the UV spectrophotometer method was a tedious process which has been simplified in the schematic diagram and shown in **Fig. 36**.

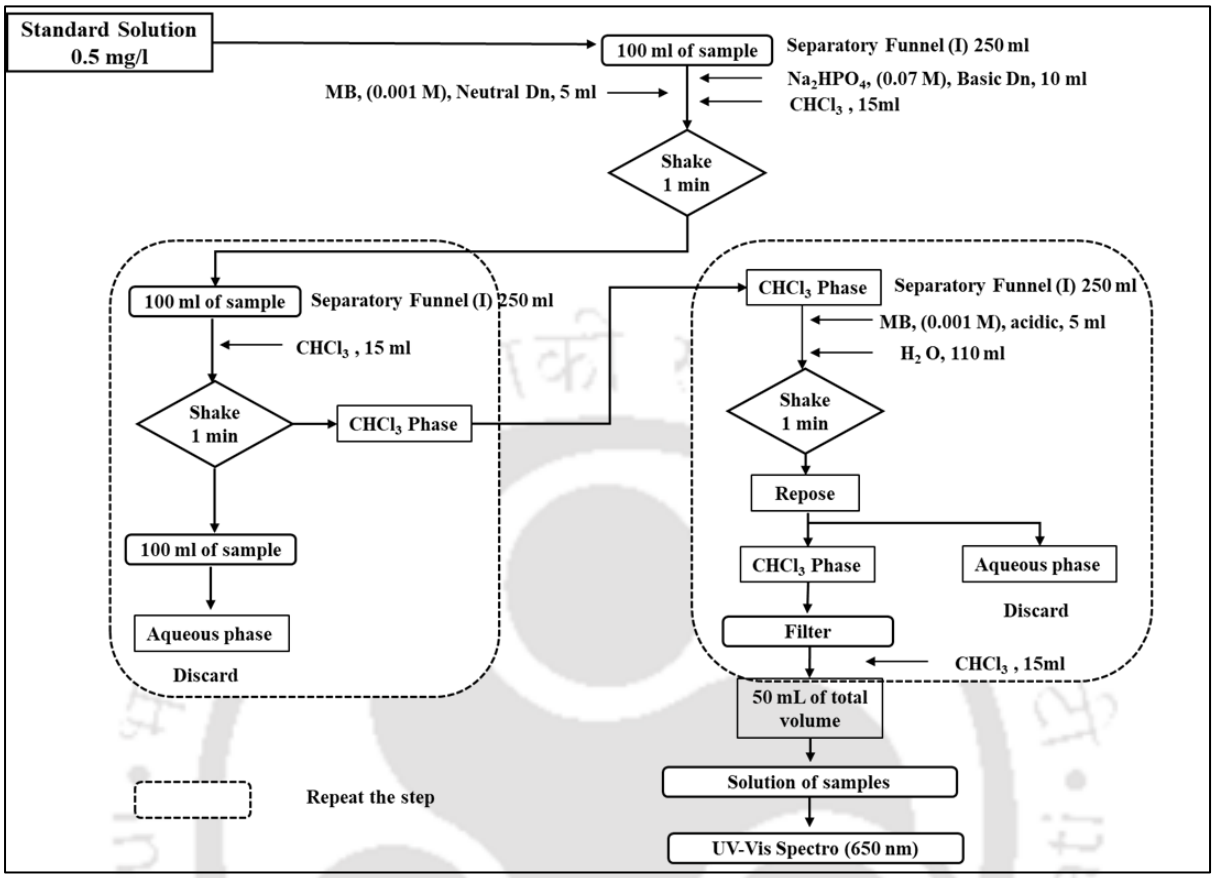


Figure 36. Schematic representation of the normalized analytical method for identifying anionic surfactants.

6.2. Results and discussion

The characteristics of laundry wastewater effluent parameters are shown in **Table 1**. Effluent waste from laundry was collected from domestic on October 2022 at IIT Guwahati, Assam, India. In this study, all of the results shown in **Table 12** are the mean values of three test samples. The experiment was conducted at room temperature. Sample pH, turbidity, COD, anionic surfactant, and concentration of MFs were analysed.

Table 12. Characteristics of laundry wastewater before and after electrocoagulation treatment.

SI. No	**Parameters**	**Initial effluent characteristics**	**Electrocoagulation operating parameters and cost estimation**	**After electrocoagulation treatment**
1	pH	9.1 ± 0.5	T= 25 min, pH= 7, CD 300 A m^{-2}, 0.53 US$ m^{-3} (44.12 INR/ m^3), Sterring speed 180.	8.9 ± 1
2	Turbidity	145 ± 10 NTU		1.2 ± 0.7 NTU
3	COD	830 ± 25 (mg/L)		112 ± 5 (mg/L) (>85 %)
4	Anionic surfactant	48 ± 3 (mg/L)		4.3 ± 1 (mg/L) (> 90 %)
	Microfibers	2300 ± 240 MFs/L		50 ± 18/L (> 95 %)

6.2.1. Effect of pH

The pH of laundry wastewater is a very significant factor in the performance of the electrocoagulation process. To investigate the influence of pH, the laundry effluent pH was adjusted with dilute aqueous NaOH and H_2SO_4. **Fig. 37** depicts the removal efficiency of MFs, surfactant concentration, and COD at different pH after 25 min of electrocoagulation operation. From the figure, it can be seen that the removal efficiency of MFs in all the samples was above 90 %, with pH levels ranging from 4 to 10. However, the removal efficiency of MFs at pH = 4 and 10 is slightly lower when compared with pH = 6 - 8. Further, the removal efficiency of surfactant and COD was 91 % and 85 %, respectively, at pH 7. At pH = 7, the maximum removal efficiency of all contaminants was determined to be optimal. The results show that a more neutral pH is likely to provide greater removal due to the favourable formation of coagulants at neutral pH, which is in line with the results of Perren et al. (2018) and Dimoglo et al. (2019). The difference in removal efficiency with pH is due to the hydrolysis and polymerization of Al^{3+} in the pH range of 6 - 8, which leads to the formation of particles $Al(OH)^{2+}$, $[Al_2(OH)_2]^{4+}$, $Al(OH)_3$, and highly charged polymeric hydroxy complexes $[Al_{13}(OH)_{32}]^{7+}$, which are effective for coagulation. As the pH rises over 10, the main hydrolysis product is $Al(OH)_4$, which inhibits the synthesis of anodized aluminium species as well as the adsorption of dispersed particles. The adsorption effect is negligible at low pH 4 because only aluminium ions are present. (Dimoglo et al., 2019; Perren et al., 2018; Tripathi et al., 2013).

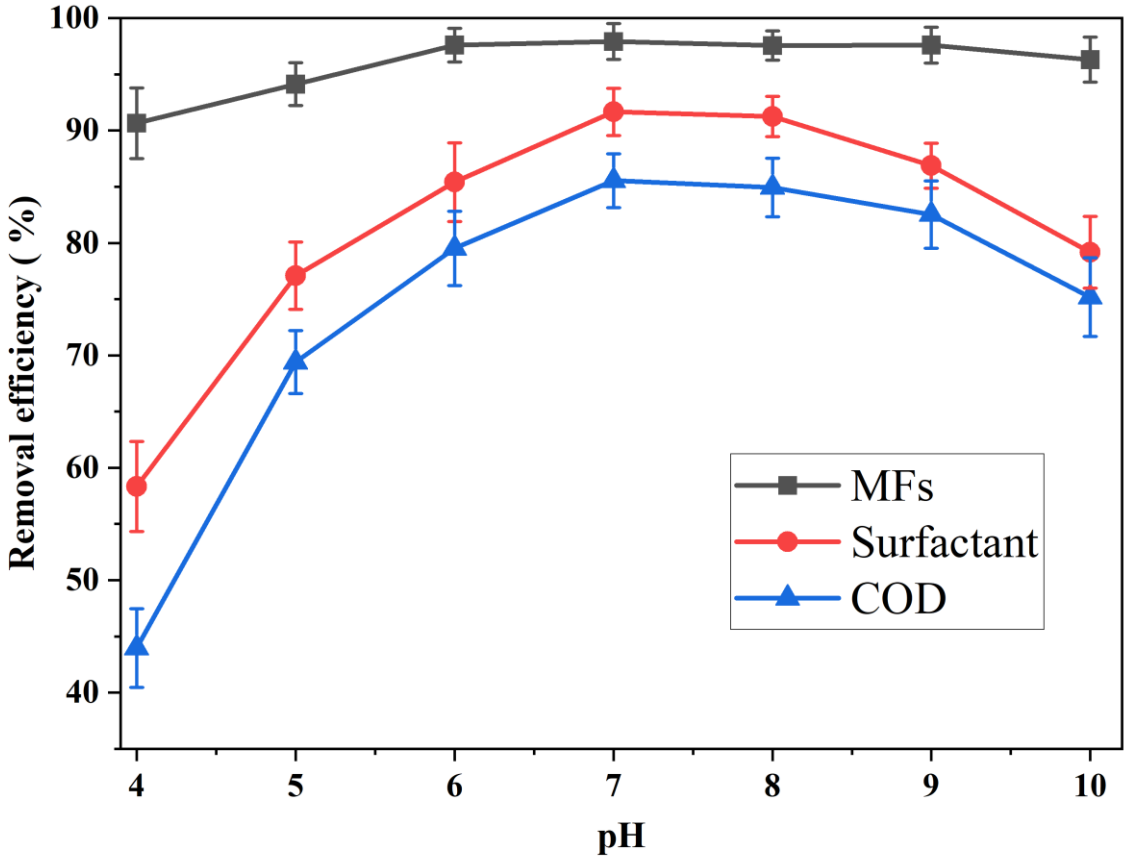


Figure 37. Efficiency of removing MFs, surfactants, and COD after 25 minutes of electrocoagulation at various starting pH levels. Electrode spacing is 0.5 cm, stirring speed is 180 rpm, and current density is 300 A/m^2.

6.2.2. Effect of current density

Current density is an essential parameter in electrocoagulation operations since it is an operating factor that may be directly regulated using the DC power source. **Fig. 38** depicts the average removal rates for the various current densities from 100 to 400 A/m^2. It can be observed from the figure that the change in current density has a considerable impact on the removal efficiency of MFs, surfactants, and COD concentrations. The removal efficiency increased with an increase in current densities. This is in line with Faraday's law of electrolysis. When the current density of the cell is increased, metal ions released from the electrodes are also

increased, and flocculants are likely to be present at high current density. Yet, there was no apparent change in removal efficiency between 300 and 350 A/m^2; taking operational cost into consideration, a current density of 300 A/m^2 was found to be optimum, with a removal efficiency of 97 %, 90.6 %, and 86 % for MFs, surfactants, and COD, respectively. Hence 300 A/m^2 has been considered for further experiments.

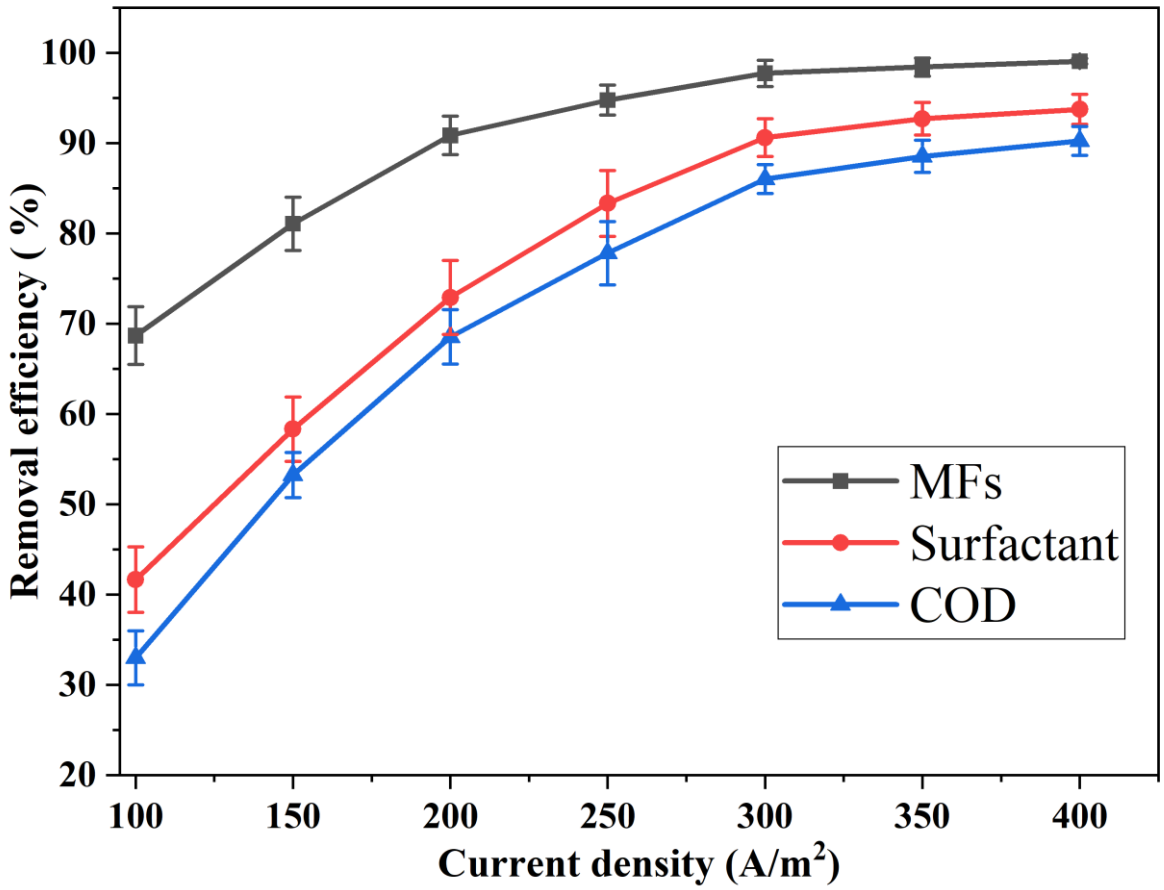


Figure 38. The effect of current density on microfibers, surfactant, and COD removal efficiency Reaction time 25 mins time, pH 7, electrode spacing 0.5 cm, and stirring speed of 180 rpm.

6.2.3. Effect of processing time

The results of MFs, surfactant, and COD removal from laundry wastewater by electrocoagulation as a function of processing time is depicted in **Fig. 39.** Contaminant removal efficiency starts increasing when the electrolysis duration is raised from 5 to 35 min, as seen in the figure.

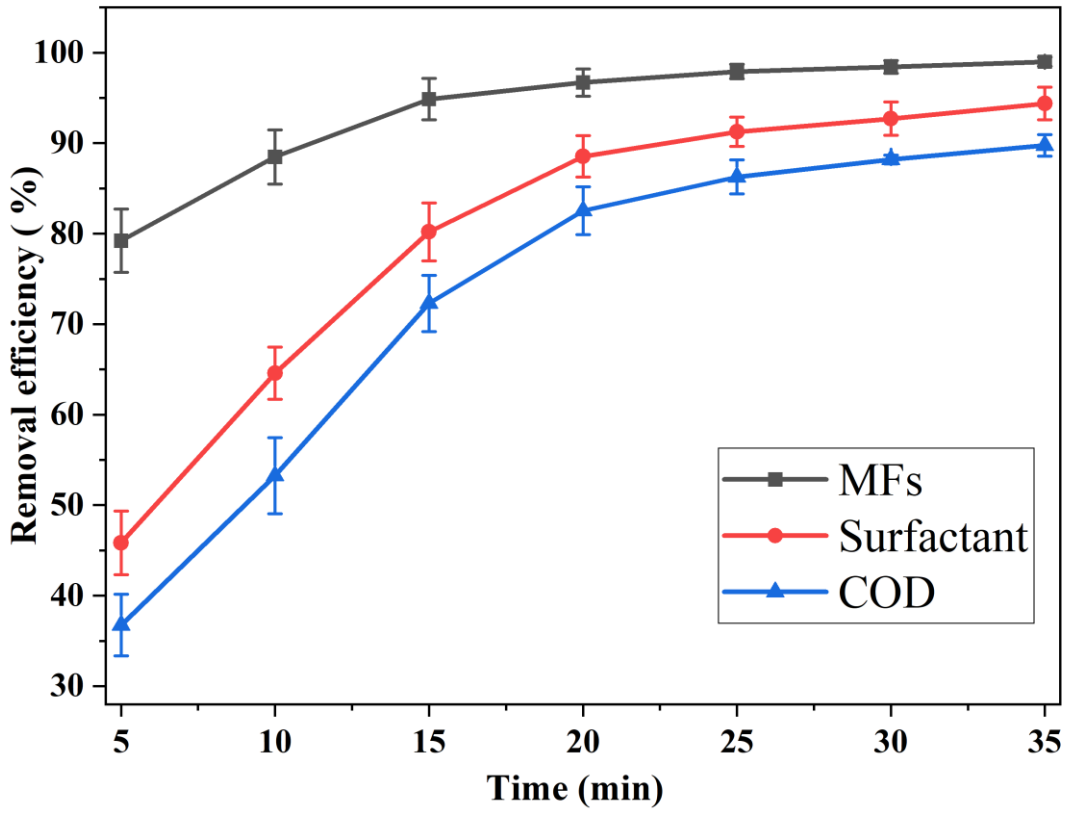


Figure 39. Effect of electrolysis time on the removal efficiency of microfibers, surfactants and COD. Reactor CD 300 is A/m2, pH 7, electrode spacing 0.5 cm, and stirring speed 180 rpm.

The results show that reaction time has a favourable influence on electrochemical treatment efficiency. Anodic electro-dissolution causes the release of coagulating species during electrocoagulation. Pollutant removal efficiency is directly related to metal ion dissolution concentration. The concentration of metal ions and associated hydroxide flocs in the water solution increases as the electrolysis duration increases. An increase in the treatment time

results in a significantly greater removal. The time when coagulants are in excess and flocculation takes control appears to be between 20 and 25 mins since current density seems to have a greater impact on contaminates elimination during this time period. Running the reactor for more than 25 mins would result in excess coagulant with minimum effect on removal efficiency. Additionally, prolonged operation results in increased electrode and energy consumption. Therefore, the operating time of 25 mins was found to be optimum with a removal efficiency of 97.9 %, 91.2 %, and 86.3 % for MFs, surfactant, and COD, respectively, at pH 7 and 300 A/m^2. The initial turbidity decreased from 145 ± 10 NTU to 1.2 ± 0.7 NTU.

6.2.4. Characteristics of microfibers and flocs

6.2.4.1. Optical microscopic analysis:

Laundry wastewater was observed under an optical microscope. The results confirmed the existence of MFs in all of the laundry wastewater samples, as shown in **Fig. 40 a).** It was observed that laundry wastewater contained around 2300 ± 240 MFs/L or 46,350 to 57,150 MFs/kg fabrics. The length of the collected MFs varied between 20 to 5000 µm with a diameter between 10 to 20 µm. After electrocoagulation, the supernatant solution and sludge were observed under the optical microscope. Around 50 ± 18 MFs/L was observed in the supernatant solution. It was found that there was a huge deposition of organic and inorganic particles (soil/dust, surfactants, dye, heavy metals) on the surface of MFs, which makes the MFs precipitate easily, as shown in **Fig. 40 b).**

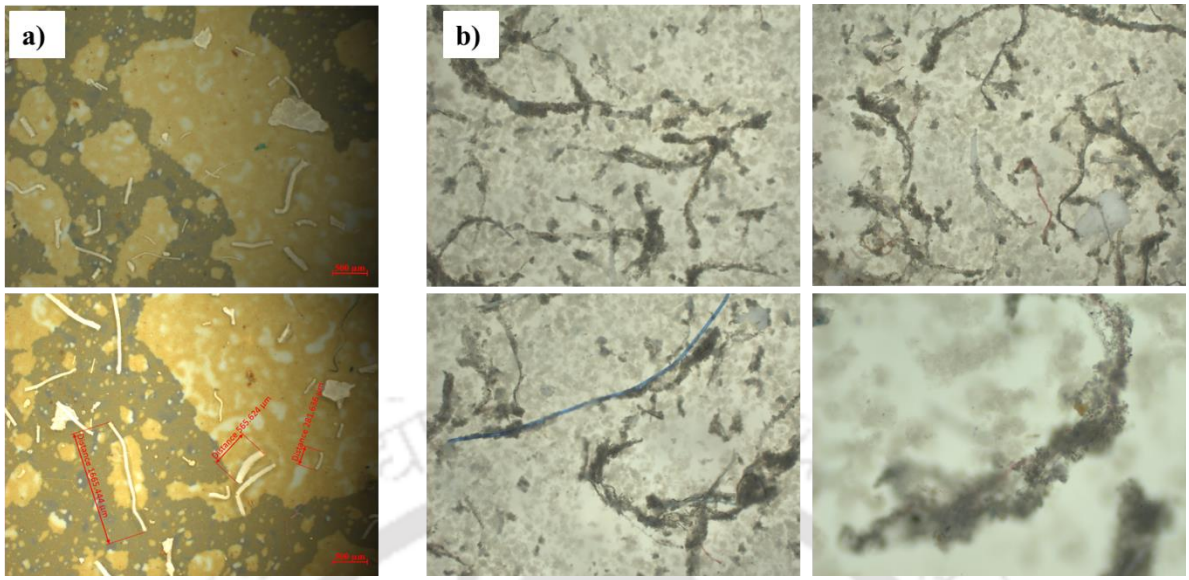


Figure 40. Microscopic images of a) MFs in laundry effluent b) sludge obtained after electrocoagulation.

6.2.4.2. FTIR analysis

A total of ten samples of laundry effluent were collected, including replicates. After the procedure, the validation of MFs polymer types was performed using ATR – FTIR technique. Spectra were scanned in the range of 4000 cm^{-1} - 400 cm^{-1}. The raw spectra were processed using Bio-Rad KnowItAll software and identified the composition of MFs polymers. The graph obtained from the ATR-FTIR of microfiber particles was examined and depicted in **Fig. 41**. It can be seen from the figure that laundry wastewater contained a polyester type of polymer. The results were compared with reference samples using Bio-Rad KnowItAll software.

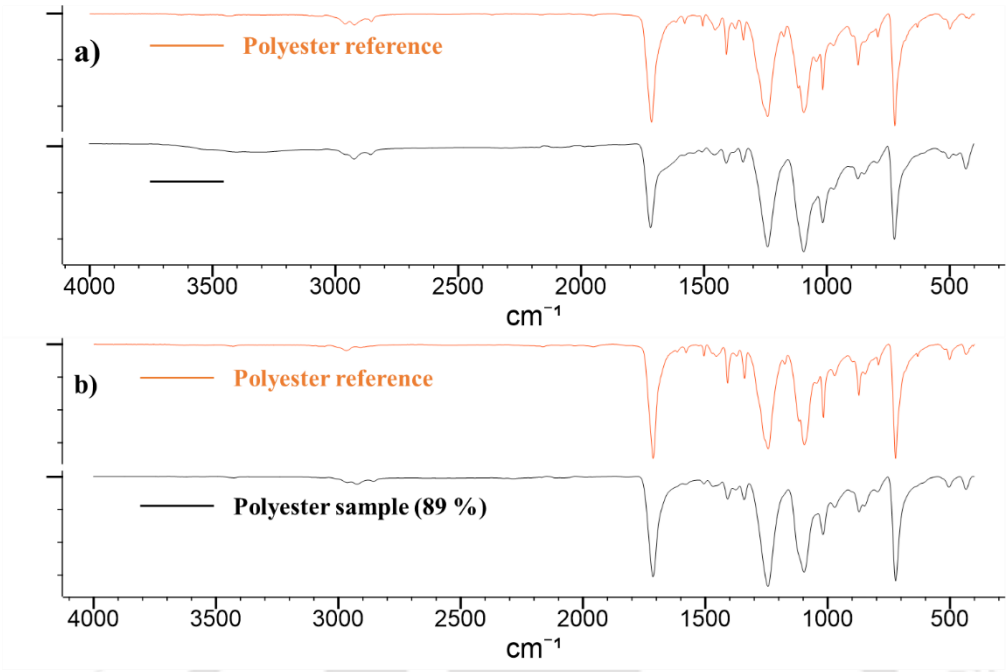


Figure 41. ATR-FTIR spectra of polyester microfibers in laundry wastewater.

6.2.4.3. FESEM analysis

After electrocoagulation, the flocs were analysed using scanning electron microscopy. The presence of MFs in the sludge can be clearly seen in FESEM images, as shown in **Fig 42**. The FESEM study is reliable, consistent, and accurate, making it possible to measure the size of fibres present in laundry wastewater and sludge. The MFs are long and thick and often occur in aggregates within flocs. The size of the flocs was> 50 µm which was confirmed using a scanning electron microscopic technique.

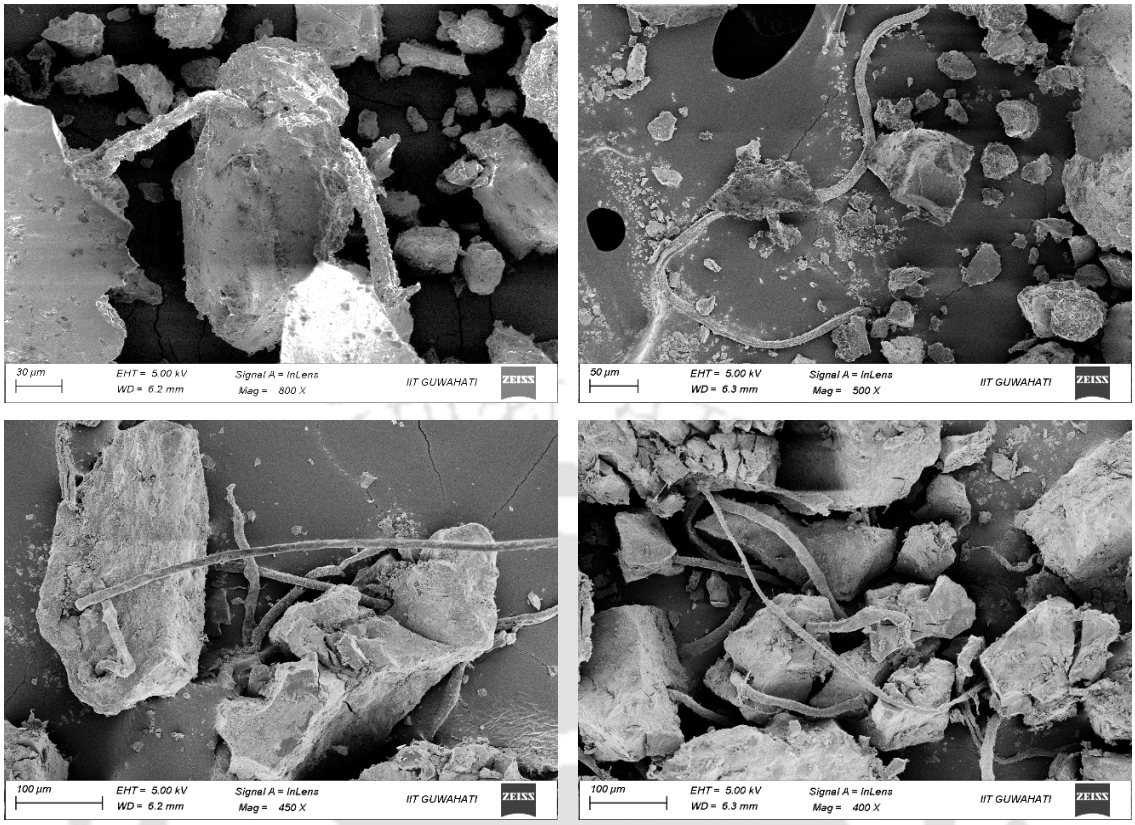


Figure 42. Scanning electron microscopic images of microfibers flocs after electrocoagulation.

6.2.5. A proposed possible mechanism

Electrocoagulation is a complicated process that uses a combination of procedures that work together to remove contaminants from wastewater. It allows for anodic oxidation and the formation of in situ active adsorbent (for example, aluminium hydroxides). Simultaneously, cathodic reactions take place, and H_2 gas is generated, allowing the absorbents to float (Perren et al., 2018; Shim et al., 2014). The produced Al^{3+} ions instantaneously hydrolyse to form equivalent hydroxides and poly hydroxides at a suitable pH. Generated aluminium hydroxides and aluminium poly hydroxides have a stronger attraction to collect the contaminants in the wastewater, producing more coagulation compared to the typical aluminium coagulants (Bayramoglu et al., 2004; Wang et al., 2009). Furthermore, the gas bubbles produced by water

electrolysis might promote the floating of contaminants and coagulated materials. A possible mechanism is proposed based on the results obtained (**Fig. 43**).

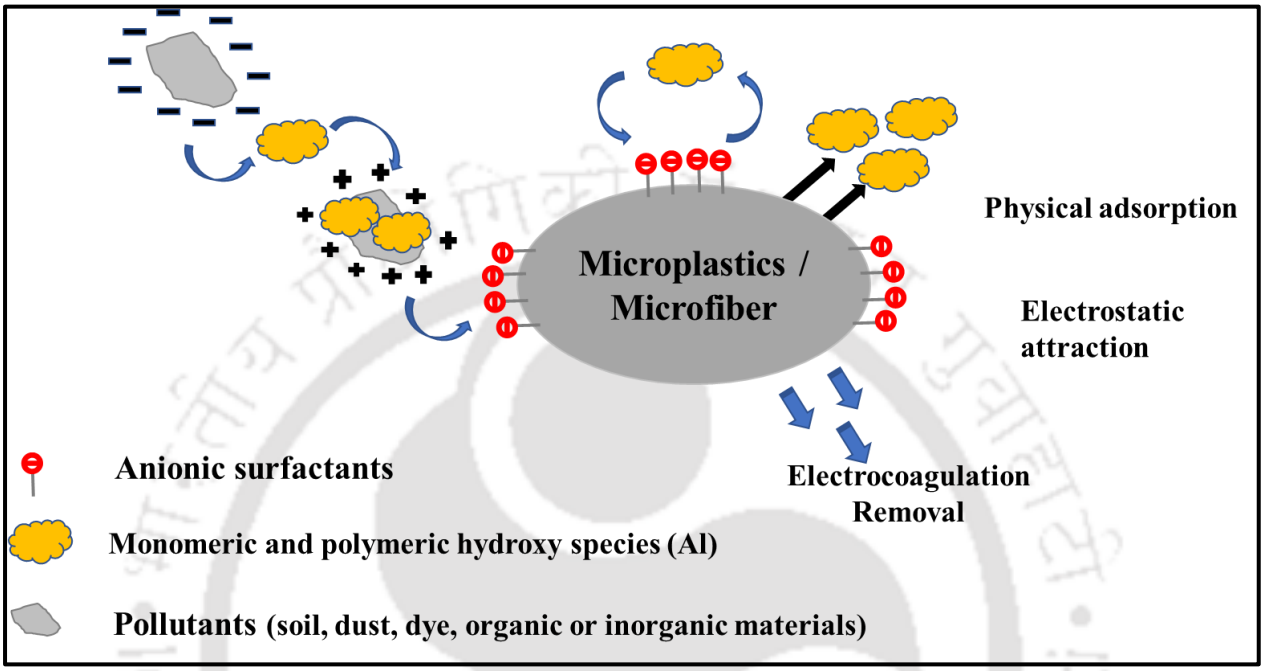


Figure 43. Schematic illustration of surfactant stealth effect of MPs/MFs during coagulation.

i) Natural clay particles, heavy metals, and dyes are usually negatively charged (Perren et al., 2018; Shen et al., 2021a; Xia et al., 2020). Monomeric species such as $Al(OH)^{2+}$, $Al(OH)_2^{+}$, $Al_2(OH)_2^{+}$, $Al(OH)_4$, and polymeric species such as $Al_6(OH)_{15}^{3+}$, $Al_7(OH)_{17}^{4+}$, $Al_8(OH)_{20}^{4+}$, $Al_{13}O_4(OH)_{24}^{7+}$, $Al_{13}(OH)_{34}^{5+}$, can neutralize the surface charges of heavy metals, dye, and clay particles (Bayramoglu et al., 2004; Shim et al., 2014) later it leads to surface deposition onto MFs during electrocoagulation.

ii) Amorphous flocs of aluminium hydroxides have a wide surface area, which is advantageous for faster MFs adsorption.

iii) Anionic surfactants are adsorbed on MFs, and the negative surface charges can be neutralized with an opposite charge or floc of aluminium hydroxides generated during

the process. Further, it leads to the formation of larger flocs, and these flocs can be removed from the aqueous medium by precipitation or hydrogen flotation.

6.2.5. Operating cost analysis of electrocoagulation

The operating cost (US$ m^{-3} of effluent) of the electrocoagulation process mainly includes electricity and electrode cost. The operational cost was calculated using Eq. 5 (Das et al., 2021b).

$$\boldsymbol{Operating\ cost}_{\boldsymbol{(electrocoagulation)}} = \boldsymbol{a} \times \boldsymbol{Q_{electrode}} + \boldsymbol{b} \times \boldsymbol{Q_{energy}} \quad (5)$$

Where, $Q_{electrode}$ and Q_{energy} denote electrode material and electrical energy usage, respectively. ‘‘a” indicates the cost of the electrode (2.82 US$ kg^{-1} of aluminium), and ‘‘b” means the cost of electricity use (0.0924 US$ kWh^{-1}). Faraday's law was used to determine the consumption of electrode material (Das et al., 2021b):

$$\boldsymbol{Q_{electrode}} = \frac{\boldsymbol{I \times V \times t}}{\boldsymbol{V_L}} \quad (6)$$

$$\boldsymbol{Q_{electrode}} = \frac{\boldsymbol{I \times t \times M.W}}{\boldsymbol{F \times z \times V_L}} \quad (7)$$

where I designates the current (A), t indicates the time of electrocoagulation process (s), MW is the molar mass of aluminium (26.98 g mol^{-1}), V is the voltage (V), V_L is the effluent volume (m^3), F is the Faraday’s constant (96,487 C mol^{-1}), and z is the number of electrons transported (z = 3). The energy and electrode costs for the electrocoagulation process increase with increasing current density. This is due to increased energy consumption and greater anodic oxidation. At 25 mins of study, the total operation cost was 0.53 US$/$m^3$ or 44.12 INR/m^3 with a rising current density of 300 A/m^2.

6.2.6. Comparative study with various literature

The release of MFs from the laundry is influenced by various parameters, including textile properties (for example, fabric type and age), washing temperature, detergent properties and dosage, and abrasion during laundry (Le et al., 2022; Yang et al., 2019). New clothes shed more MFs during washing because of residues from the fabric production process (De Falco et al., 2018). Various literature estimates MFs detachment from fabrics. However, their results are difficult to compare since they employed utilized materials, different washing durations, and other detergents and are presented in different units. A summary of these previously published works is shown in **Table 13**. To make the comparison easier, all the finding are given in the same unit. In comparison to previous investigations, present study shows less number of microfibers. This may be due to less washing time, lower water temperature (25 to 30 ºC), and use of liquid detergent, which resulted in less release of micro MFs fibres in laundry outlets. Similar interference was also reported by Yang et al. (2019) and Le et al. (2022).

The studies on the removal of MFs and surfactant in various wastewater using the electrocoagulation process is shown in **Table 14**. Significantly, the available research on the removal of MFs and surfactants from laundry wastewater by the electrocoagulation process is very limited. The comparison of targeted parameters and data based on pollutant removal obtained in this work suggests that aluminium electrodes are best suited when compared to other electrodes. This is also comparable with the studies that have used aluminium electrodes for pollutant removal from various wastewater, as shown in the table. Further, the research conducted in this work could serve as benchmark data in the field of treatment of MFs and surfactants from the laundry industry.

Table 13. Comparison of various works of literature with the present work.

Type of fabric	New clothes	Synthetic MFs	MFs length	Surfactant concentration	Reference
Commercial garments (PES, PP)	Yes	12 × 10^5 to 35.40 × 10^5 MFs/kg	20 to 2,000 µm	n.d	(De Falco et al., 2018)
Commercial garments (polyester)	Yes	6.40 × 10^5 to 15 × 10^5 MFs/kg	Avg 360 to 660 µm	n.d	(De Falco et al., 2019)
Textile garments (PES, polyester-elastane and polyamide-elastane)	Yes	30,000 to 4,65, 000 MFs/m^2	20 to 5,000 µm (Avg 0.2 to 0.4 mm)	n.d	(Belzagui et al., 2019)
Household clothes and linens (Cotton, PES, PA, viscose, elastane, acrylic)	No	30 × 10^5 MFs/kg	0.17 mm (50 to > 500 µm)	n.d	(Galvão et al., 2020)

Type of fabric	New clothes	Synthetic MFs	MFs length	Surfactant concentration	Reference
PES, PA, and poly acetate fabrics	Yes	74,816 ± 10,656 MFs/m^2	5 to 4000 µm (avg PES: 499.49 ± 505.65 µm, PA: 1056.53 ± 761.42 µm, acetate: 1128.00 ± 750.72 µm	n.d	(Yang et al., 2019)
Household clothes	No	4,400 to 10,800 MFs/L	6 to 4000 µm	8 ± 2 to 800 ± 50 (mg/L)	(Luo et al., 2022)
Household lining fabric	No	2300 ± 240 MFs/L, 7,725 to 9,525 MFs/m^2, or 46, 350 to 57, 150 MFs/kg	> 50 to 5000 µm	48 ± 3 (mg/L)	Present work

Avg., average length; n.d, not determined

Table 14. Removal of microfibers and surfactant in various wastewater using the electrocoagulation process.

Type of wastewater	Pollutants	Treatment method	Electrode	Removal efficiency	References
Domestic wastewater (PE, PVC)	Microplastics	Electrocoagulation-electroflotation and membrane filtration	Aluminum and iron	Microplastics 100 % (Electrocoagulation and membrane)	(Akarsu et al., 2021)
Effluent from the local WWTPs	Polyester microplastics (25 mg/L) were added	Electrocoagulation	Aluminum	Microplastics 96.5 %	(Elkhatib et al., 2021)
Synthetic wastewater	Microplastics (PE, PMMA, CA, PP)	Electrocoagulation	Aluminum and iron	For Al: PE 93.2 %, PMMA 91.7 %, CA 98.2 %, PP 98.4 %. For Fe: PE 71.6 %, PMMA 58.6 %, CA 85.4 %, PP 82.7 %.	(Shen et al., 2022)

Type of wastewater	Pollutants	Treatment method	Electrode	Removal efficiency	References
Secondary effluent of the sewage treatment plant	PE microplastics (25 mg/L) were added	Electrocoagulation	Aluminum	Surfactants 97.5 %	(Xu et al., 2022)
Laundry wastewater	Surfactants	Electrocoagulation	Aluminum	Surfactants 80 %	(Ramcharan and Bissessur, 2017)
Laundry wastewater	Surfactants	Electrocoagulation/ electroflotation	Aluminum	Surfactants 90 %	(Dimoglo et al., 2019)
Laundry wastewater	Surfactants	Electrocoagulation	Aluminum and iron (Al-Al, Fe-Fe, Al-Fe, and Fe-Al	72.89 % in Al-Al, 54.33 % in Fe-Fe, 62.70 % in Al-Fe, and 49.01 % in Fe-Al	(Oktiawan et al., 2021)
Laundry wastewater	Microfibers and surfactants	Electrocoagulation	Aluminum	Microfibers 97.9 %, Surfactants 91.2 %,	Present work

6.3. Summary

Synthetic MFs accumulation and deposition in the aquatic ecosystem are presently undeniable. The work emphasized on the presence of MFs in laundry wastewater and the effective removal of pollutants by the electrocoagulation process. According to the results, a reference load of 2 kg of synthetic materials releases 92,700 to 1,14,300 synthetic MFs. From the study, it was envisaged that initial pH, operating time, and current density have a substantial impact on the removal of contaminants from laundry wastewater. The removal efficiency of MFs, surfactants, and COD is higher at neutral pH. The Electrocoagulation system was effective in reducing more than 86 % of the amount of pollutant concentration in laundry wastewater. The total operation cost of laundry wastewater treatment by electrocoagulation was 0.53 US$/$m^3$ or 44.12 INR/m^3.

Chapter 7

7. Conclusion and future scope of research

Preface: *This chapter summarizes the inferences drawn from the present work and recommendations for future work.*

7.1. Conclusion

Brahmaputra River water and tap water were both contaminated with microplastics. The average microplastic concentration was between 0.30 ± 0.08 to 2.56 ± 0.13 and 0.20 ± 0.03 to 0.52 ± 0.09 particles/L in the river and tap water, respectively. The study also revealed that microplastic fibres were dominant, and a majority of them were in the 0.1 - 1 mm size range. Most microplastic properties, including size, shape, and composition, detected in tap water also matched those in river water. Household water storage tanks and water supply pipes are releasing the microplastics particles which has been clearly observed and identified from microscopy and Raman spectroscopy. Most of the microplastics found in the river water were fibers (58 %), colorless (49 %), polyethylene (30 %), polyester (20 %), and polypropylene (13 %). The tap water had 56 % fibers, 53 % colorless, and 31 % polyethylene, followed by 15 % polypropylene and 15 % pigmented polymer. The removal of microplastics from tap water was evaluated using commercially available membranes and 99 to 100% reduction was observed after passing through the HF-UF membrane.

The sea water and beach sediments from Indian beaches have been polluted with microplastics. The total mean microplastic abundance in sea water and beach sediment from all considered locations is 8.2 ± 3 particles/m^3 and 664 ± 114 particles/kg respectively. The order of total microplastic abundance in beach sediment was Malpe (1002 ± 174 particles/kg) > Panambur (931 ± 156 particles/kg) > Tannirubavi (781 ± 121 particles/kg) > Surathkal (350 ± 108 # particles/kg) > Kapu (264 ± 62 particles/kg) which can be connected with a population, tourists, and industry around the shores. The highest abundance of microplastics was observed in Malpe beach, and the lowest was found in Kapu beach. Around 49% of microplastics were observed in the 0.1 to 1 mm size range. PE (37%), PP (26%), and PS (1 7%) microplastics were abundantly observed in all selected locations. The effects of long-term exposure of

microplastics in the environment were visualized from FESEM images which showed the cracking, roughness of the surface, mechanical and oxidative weathering.

In the current study, successfully developed a facile and cost-effective methodology to extract the microplastics from the salt samples. More than 95% of microplastics were isolated from the salt samples. White and transparent microplastics visual identification was made easy with the NR dye. A wide range of microplastics were found: 1400 ~ 1900 particles/kg in refined sea salt, 1900 ~ 2300 particles/kg in unrefined sea salts, and 200 ~ 400 particles/kg in rock salts. A relatively high number of microplastics were found in sea salts rather than rock salts. Sheet type microplastics with size 1 ~ 4 μm was observed in rock salts. The most common microplastics were polyethylene, polypropylene, polyethylene terephthalate, nylon, and polystyrene. A total of 99.3% of microplastics were effectively removed from synthetic seawater using a hollow fiber membrane, and the Ion Chromatography result showed that membranes have no impact on salt concentration. As a result, HF-MF membranes can be used to remove anthropogenic pollutants from seawater.

The presence of MFs in laundry wastewater and the effective removal of pollutants by the electrocoagulation process was effectively evaluated. From the study, it was envisaged that initial pH, operating time, and current density have a substantial impact on the removal of contaminants from laundry wastewater. According to the results, a reference load of 2 kg of synthetic materials releases 92,700 to 1,14,300 synthetic MFs. The removal efficiency of MFs, surfactants, and COD is higher at neutral pH. The percentage removal efficiency of MFs, surfactants, and COD was 97.9%, 91.2%, and 86.3%, respectively, at an operating time of 25 min, a current density of 300 A/m^2 with optimum consumption of electrodes. The Electrocoagulation system was effective in reducing more than 86% of the amount of pollutant

concentration in laundry wastewater. The total operation cost of laundry wastewater treatment by electrocoagulation was 0.53 US$/$m^3$ or 44.12 INR/m^3.

7.2. Future scope

The current research tried to address the various challenges faced during the identification and removal of microplastics from various sources. However, the above studies were limited to a lab-scale. Therefore, the research can be extended in the following mentioned areas to augment the work further.

- For a better understanding of the sources, fate, and movement of microplastics in river water, it is strongly advised to extend this monitoring effort into a long-term study.
- The performance of the drinking water treatment plant to remove microplastic particles was less effective. Further studies are required on the factors affecting the efficiency of microplastic removal in drinking water treatment plants.
- A regular and permanent waste management system should be implemented for beach protection that are connected with a population, tourists, and industry around the shores.
- The viability for production of microplastics free sea salt by using MF/UF treated brine of the desalination plant can be explored and that provide dual benefit such as effective brine management and production of low-cost microplastics free sea salt.
- Electrocoagulation system was effective in reducing more than 86% of the amount of pollutant concentration in laundry wastewater. Further research should look at the impact of salt percentage and current density on the efficiency and cost operations.
- A two-stage, continuous electrocoagulation reactor with membrane or adsorption or sand-based separation appears to be the most practical solution for a large-scale industrial

electrocoagulation process. Additional studies may be conducted to investigate different reactor designs and combinations to improve the process.

- There is a significant global interest in Pyrolysis-GC/MS as a technique to identify and quantify the microplastics in various matrices. This method reduces background chemical interference and aids in the detection of lower microplastic quantities in complicated environmental samples. This method can be used in further research to analyse microplastics in complex samples.
- Detecting nanoplastics is becoming an increasing topic of interest among scientists in this field; future research should consider incorporating nanoplastics analysis into pyrolysis-GC/MS studies, which could open up opportunities for understanding the presence, distribution, and potential environmental impacts of these tiny plastic particles.

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List of publications and conferences presentation

Research paper:

1. **Naveenkumar Ashok Yaranal**, Senthilmurugan Subbiah, and Kaustubha Mohanty. "Identification, extraction of microplastics from edible salts and its removal from contaminated seawater." *"**Environmental Technology & Innovation**"* 21 (2021): 101253.
2. **Naveenkumar Ashok Yaranal**, Senthilmurugan Subbiah, and Kaustubha Mohanty. "Distribution and characterization of microplastics in beach sediments from Karnataka (India) coastal environments." *"**Marine Pollution Bulletin**"* 169 (2021): 112550.
3. **Naveenkumar Ashok Yaranal**, Senthilmurugan Subbiah, and Kaustubha Mohanty. "Microplastics from river to tap water: Occurrence and removal." (to be submitted).
4. **Naveenkumar Ashok Yaranal**, Senthilmurugan Subbiah, and Kaustubha Mohanty. "Microplastics and other organic materials removal from laundry wastewater using electrocoagulation method." "***Water Emerging Contaminants & Nanoplastics***" 2 (2023): 23.

Review paper:

1. **Naveenkumar Ashok Yaranal**, Senthilmurugan Subbiah, and Kaustubha Mohanty. "Review on Humans Exposure to Microplastics." *"Water, Air, & Soil Pollution"* (submitted).

Book Chapter:

1. **Naveenkumar Ashok Yaranal**, Senthilmurugan Subbiah, and Kaustubha Mohanty. **Chapter 4** "Microplastics in the Environment: Sources, Pathways and Abundance". In book titled Assessing the Effects of Emerging Plastics on the Environment and Public Health. *"**IGI global publisher of timely knowledge**"* 2022.

Conferences:

1. **Naveenkumar A Y,** Senthilmurugan Subbiah, and K. Mohanty, Microplastics free salts: removal of microplastics from contaminated seawater, International Conference on Advances in Chemical, Biological and Environmental Engineering, Malaviya National Institute of Technology, Jaipur, India, 23-24 April, 2021.

2. **Naveenkumar A Y,** Senthilmurugan Subbiah, and K. Mohanty 'Identification, extraction of microplastics from edible salts and its removal from contaminated seawater', Recent Innovations in Chemical Engineering" (RICE - 2021), Maulana Azad National Institute of Technology Bhopal, India, 08th – 09th February, 2021.