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Assessment of Microplastic in Raw and Drinking Water of Al-Hilla City

A Thesis

Submitted to the College of Engineering, University of Babylon in Partial Fulfillment of the Requirements for the Degree of Master in Engineering/ Environmental Engineering

By

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بسم الله الرحمن الرحيم

﴿وَإِن مِّن شَىْءٍ إِلَّا عِندَنَا خَزَآئِنُهُۥ وَمَا
نُنَزِّلُهُۥٓ إِلَّا بِقَدَرٍ مَّعْلُومٍ﴾

صدق الله العظيم

سورة الحجر

الآية (21)

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Dedication

To my family, “The safe base from which we take off and land when we decide to fly to anything in life, whether work or study, and nothing is preceded or based on its ruins.

It is for it that we build our successes, as it is the goal and the end, from which we begin and to which we return.

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Sura Mohammed

2023

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Abstract

Studying of microplastic distribution in the water bodies is important due to the potential of its chemical particle decomposition. Microplastic abundance is constituted by population activities which are not accompanied by adequate waste management systems. Since industrial and human activities have been developed in different ways in Iraq, water quality has been declining along surface and ground water. This study was conducted from August to December of 2022 using the garbage trap method, with monthly sampling. Five samples were taken with 30 days intervals between each. The sampling sites consisted of five stations for raw water and five stations for drinking water.

The collected water samples were tested using different techniques, including Fourier Infrared Spectrometer (FTIR), Scanning Electron Microscope (SEM) and Micro Raman Spectrometer (MRS).

The analyzing and testing results for raw water indicated that the most polluted locations by microplastic particle were in stations Al-Dora Bridge and Sadat Al- Hindiya with total plastic mass around 2.811 gm and 2.771 gm, respectively. FITR results of raw water samples revealed that it mainly consist of metal oxides, Poly Vinyl Chloride (PVC), polyamides polymer like Nylon6,6 and skeletal of cellulose. SEM results of raw water samples showed the presence of microplastic particles with that size smaller than 5 mm. The range of diameter of the plastic particles in the five locations were between (28.77 - 192.79) nm.

The five sampling stations for drinking water also show that the main components of microplastic pollutants are metal oxides, Poly Vinyl Chloride (PVC), polyamides polymer like Nylon6,6 and skeletal of cellulose. SEM tests for drinking water showed the presence of microplastic particles with that size ranged between (0.5 - 4.8) nm. In addition, Micro-Raman spectroscopy (MRS) results indicated a variation

in polyethylene (PE) particles, and it was between (1450 – 4220) cm^{-1} for raw water samples, the band originating from variations of the crystalline group $-CH_2$ has a larger intensity (1200–1500) cm^{-1}, than the band originating from variations of the group in the liquid phase, and between (121.404 – 4391.729) cm^{-1} for samples of drinking water, the intensity of the symmetric CH_2 stretching mode at 2600 cm^{-1} relative to the asymmetric CH_2 stretching mode at 3100 cm^{-1} appears to be higher for Low density polyethylene (LDPE) compared to High density polyethylene (HDPE) for samples of drinking water.

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List of Symbols

Symbol	*Description*	*Unit*
%	Percent	
mm	milemeter	mm
L	Liter	L
µm	micrometer	µm
cm	centimeter	cm
mg	Milligram	mg
P	Particle	No.
Kg	Kilogram	Kg
Km	Kilometer	Km
Fe(II)	Iron Binary	
NaCl	Sodium Chloride	
g	gram	g
^{o}C	Degrees Celsius	^{o}C
$FeSO_4$	Iron Sulfate	
Mol	Mole	Mol
mL	milliliter	ml
NTU	Nephelometric Turbidity Unit	NTU
ppm	Part Per Million	ppm
S	Siemens	
D	Diameter	mm, µm
nm	nanometer	nm

Table of Abbreviations

Abbreviation	*Description*
MP$_{S}$	**Microplastic Pollutions**
POPs	**Persistent Organic Pollutants**
PHA	**Polyhydroxy-alcanoates**
PLA	**Polyactic Acid**
bio-PBS	**Polybutylene Succinate**
bio-PE	**Bio-Polyethylene**
bio-PET	**Ethylene Bioterephtalate**
bio-PTT	**Trimethylene Biopolyterephthalate**
bio- PA	**Bio-Sources Polyamides**
PUR	**Polyurethanes**
PCL	**Polycaprolactone**
PBAT	**Polybutylene Adipate-co-Terephthalate**
PBS	**(Polybutylene Succinate) – Copolymers**
PE	**Polyethylene**
PET	**Ethylene Terephthalate**
PS	**Polystyrene**
PP	**Polypropylene**
PVC	**Polyvinyl Polychloride**
PA	**Polyamides**
PUR	**Polyurerthane**
PET	**Polyethylene Terephthalate**
PAA	**Polyactic Acid**
PCCP	**Personal Care and Cosmetic Products**
PC	**Polycarbonate**
PU	**Polyurethane**
BpA	**Bisphenol A**
WHO	**World Health Organization**
FTIR	**Fourier Infrared Spectrometer**
UV	**Ultraviolet Visible**
MRS	**Micro-Raman Spectrometer**
SEM	**Scanning Electron Microscope**
EDS	**Energy Dispersive X-ray Spectroscopy**
WWTP	**Wastewater Treatment Plant**
IVM	**Instituut Voor Milieuvraagstukken**
NOAA	**The National Oceanic and Atmospheric Administration**

Chapter One

Introduction

CHAPTER ONE

INTRODUCTION

1.1 General

Pollution is defined as the introduction into the environment of substances harmful to humans and other living organisms. Pollutants are harmful solids, liquids, or gases produced in higher than usual concentrations that reduce the quality of our environment (Manisalidis et al., 2020).

Plastics are a wide range of synthetic or semi-synthetic materials that use polymers as a main ingredient. Their plasticity makes it possible for plastics to be moulded, extruded or pressed into solid objects of various shapes. This adaptability, plus a wide range of other properties, such as being lightweight, durable, flexible, and inexpensive to produce, has led to its widespread use (*Life Cycle of a Plastic Product, 2011*).

The success and dominance of plastics starting in the early 20th century has caused widespread environmental problems, due to their slow decomposition rate in natural ecosystems. Toward the end of the 20th century, the plastics industry promoted recycling in order to ease environmental concerns while continuing to produce virgin plastic and to push the responsibility of plastic pollution onto the consumer. The main companies producing plastics doubted the economic viability of recycling at the time, and the economic viability has never improved. Plastic collection and recycling is largely ineffective because of failures of contemporary complexity required in cleaning and sorting post-consumer plastics for effective reuse. Most plastic produced has not been reused, either being captured in landfills or persisting in the environment as plastic pollution. Plastic pollution can be found in all the world's major water bodies, for example, creating garbage patches in all of the world's ocean and contaminating terrestrial ecosystems. Of all the plastic discarded so far, some 14% has been incinerated and less than 10% has been recycled (*Life Cycle of a Plastic Product, 2011*).

In developed economies, about a third of plastic is used in packaging and roughly the same in buildings in applications such as piping, plumbing or vinyl siding. Other uses include automobiles (up to 20% plastic), furniture, and toys. In the developing world, the applications of plastic may differ; 42% of India's consumption is used in packaging. In the medical field, polymer implants and other medical devices are derived at least *partially from plastic. Worldwide, about 50 kg of plastic is produced annually per person, with production doubling every ten years (*Life Cycle of a Plastic Product, 2011).

1.2 Definitions of Microplastics

The term ‘microplastics’ was coined in 2004 (Thompson, et. al., 2004) and research in this field has flourished since then, especially in the last 10 years (Horton and Barnes, 2020). The definition of microplastics is not completely straightforward. Microplastics represent a diverse range of material types, shapes, colors and sizes (Thompson, 2015). To account for these complexities, researchers have proposed definitions for plastic debris (Hartmann et al., 2019) and microplastics (Verschoor, 2015), for example, by describing them according to specific criteria. Because of the different methods used to collect and quantify microplastics, it was not possible to apply a uniform definition to the results of existing studies. Most definitions in the literature focus on composition and size. A widely used definition describes microplastics as plastic particles smaller than 5 mm in length (Californian state Water resources Control Board). However, this is a rather arbitrary definition and is of limited value in the context of drinking-water since particles at the upper end of the size range are unlikely to be found in treated drinking-water. Several researchers define a lower bound at about 1 μm. The lower bound is often simply a function of the sampling and analytical technique used in the study. A subset of microplastics smaller than 1 μm in length are often referred to as nanoplastics, but again with an inconsistent upper bound.

As for the composition of microplastics, there is again no standard definition. Many studies focus on particles made from synthetic polymers rather than using the International Organization for Standardization (ISO) definition, which excludes elastomeric materials (ISO, 2013). The German Federal Ministry of Education and Research defines plastics as a subgroup of polymers including elastomers and modified natural polymers (Braun et al., 2018). The European Chemicals Agency (ECHA, 2019) uses solid polymer-containing particles but excludes natural polymers that have not been modified.

Microplastic surveys have been carried out in locations globally, with microplastics discovered widely across marine, freshwater, terrestrial and atmospheric environments (Horton, et. al., 2017). Microplastics are now present even in remote areas including the deep sea, in remote mountainous areas (Allen, et. al., 2019), within Arctic sea ice (Obbard, et. al., 2014), and in Antarctic freshwaters (González-Pleiter, et. al., 2020).

1.3 Microplastic Pollutions (MPs)

The risks of microplastic pollutions to human health are still poorly known. Exposal may occur by inhalation of microplastic pollutions suspended in the air or by ingestion of microplastic pollution containing food and beverages. Outdoor air may contain 0.0003 to 0.0015 p/L, while indoor air may contain 0.001–0.006 p/L (Dris, 2017). Inhaling airborne particles can contaminate the airways, causing inflammation by dust overload, oxidative stress and may cause malignant damage (Prata, 2018). Ingested microplastic pollutions are bioresistant and not degradable in vivo. Potential damages can be expected by the mechanical properties of microplastic pollutions which are believed to cause local inflammatory responses in the intestine (Waring, 2018).

Common types of common microplastics include polyvinyl chloride, nylon, polyethylene, polypropylene, polyethylene terphaterene, and polyethyrene. Representing 95% of global plastic production, these microplastics have negative impacts on ecosystems by entering the food chain and acting as a vector for transporting pollutants, especially organic, which has an affinity for plastic surfaces (Lagard et al., 2016).

The large plastic waste referred to as “Macroplastic” is a source of concern because of its impact on water bodies in the long run, and causes aesthetic problems in addition to threatening navigation, fishing and aquaculture, in addition, it causes injury and death to marine organisms (Cole et al., 2011). It is known that many aquatic organisms are affected by plastic waste and the effects of microplastics, both micro and secondary, on producers (plants, algae), which are the basis of the food chain, which plays an important role in maintaining the balance of the ecosystem and is responsible for most of the oxygen through photosynthesis (Blhattacharya and Rao, 2010).

1.4 Statement of the Problem

Plastic pollution is most visible in developing countries like Iraq, where garbage collection systems with low recycling rates are often inefficient or nonexistent.

Once at surface, sunlight, wind, and wave action break down plastic waste into small particles, often less than 5 mm.

These so-called microplastics are spread throughout the water column and have been found in every corner of the globe, from the highest peak, to the deepest trough. Microplastics are breaking down further into smaller and smaller pieces. Plastic microfibers, meanwhile, have been found in municipal drinking water systems and drifting through the air (https://www.nationalgeographic.com/environment/article/plastic-pollution).

The short- and long-term environmental hazards of plastics' wide use remain unknown at this time. It is commonly acknowledged, though, that the large and constant inputs of plastic into the environment have a negative impact on ecosystems and public health. A precautionary approach requires a uniform characterization of the possible dangers associated with environmental plastic pollution as well as the location of these pollutants' sources. The introduction of river system plastics into the aquatic environment is another topic of concern. The water strategy framework directive in Iraq, need to require the measuring of these fluxes. Nongovernmental organizations have started to address the problem of plastic pollution by encouraging clean-up campaigns, such as citizen science initiatives that measure plastic pollution in rivers and streams.

It is important to conduct a study of microplastic contamination in the river because of deterioration of its chemical characteristics which significantly increases water pollution. Currently, the problem of plastic waste has become a national concern (Jambeck et al., 2015). In recent years, microplastics have been discovered abundant in the surface waters and along the shorelines of Iraqi rivers. Al-Hilla city center has been identified as one of the causes of pollution since the concentration and increase of this pollution exposes the river to direct threats and makes it unfit for life if the problem continue. Food scraps, plastic boxes, cartons, medical waste, restaurant waste, and household trash be readily ingested by fish and perhaps freshwater invertebrates, which are thus exposed to toxic contaminants leaching from their surfaces.

1.5 The aim of the Study

The aim of this study to assess the microplastic in raw and drinking water of Al-Hilla city.

1.6 The Objective of the Study

The Objective are :-

1-Analyze the abundance of MP in water samples by calculating the mass and density.

2-Identify the polymer composition op MP in water samples.

3-Visualize MP in water samples to calculate shape and size.

1.7 Thesis Layout

To meet the above-mentioned target, the present research is divided into the following tasks***:***

Chapter One: includes a brief introduction of microplastic, microplastic pollutions (MPs), statement of the problem and objective of the study.

Chapter Two: gives a basic concepts of the microplastic in the raw and drinking water and a literature review.

Chapter Three: this chapter outlines shows the experimental work.

Chapter Four: this chapter gives the results and discussion for raw and drinking water samples.

Chapter Five: the conclusion summarizes the most important outcomes, recommending the possible directions of further works to be continued.

Chapter Two

Basic Concepts and Literature Review

CHAPTER TWO

BASIC CONCEPTS AND LITERATURE REVIEW

2.1 Introduction

The UN calls plastic pollution a ‘planetary crisis’ (MacLeod, et. al., 2021). The marine plastic pollution problem has grown exponentially, plastic pollution is now ubiquitous and is projected to increase even if current corporate and government commitments are met (Borrelle, et. al., 2020). Production has rocketed in the last two decades, with as much plastic being produced between 2003 and 2016 as in all the preceding years combined.

The production process of the artificial and natural polymers along with generation of macroplastics and microplastics from synthetic polymers are illustrated in Fig. 2.1. In recent days, wide range of products are made up of plastics due to their ease of manufacture, inertness (chemical, temperature and light resistance as well), low cost, high strength/ weight ratio and resistance to water. These properties of plastic make them a suitable candidate for their use in a wide spectrum of biotechnological applications and more so in industrial organic synthesis. For this reason, the steep rise in plastic production has been seen during the last few decades and 288 million tons of plastics were produced worldwide in 2013 alone. The durability of plastic makes it highly resistant to degradation and therefore, disposing of plastic waste is a big challenge. Recycling is one of the solutions but unfortunately majority of the plastic debris ends up in landfill which takes a long duration for its breakdown and decomposition (Cole et al., 2011).

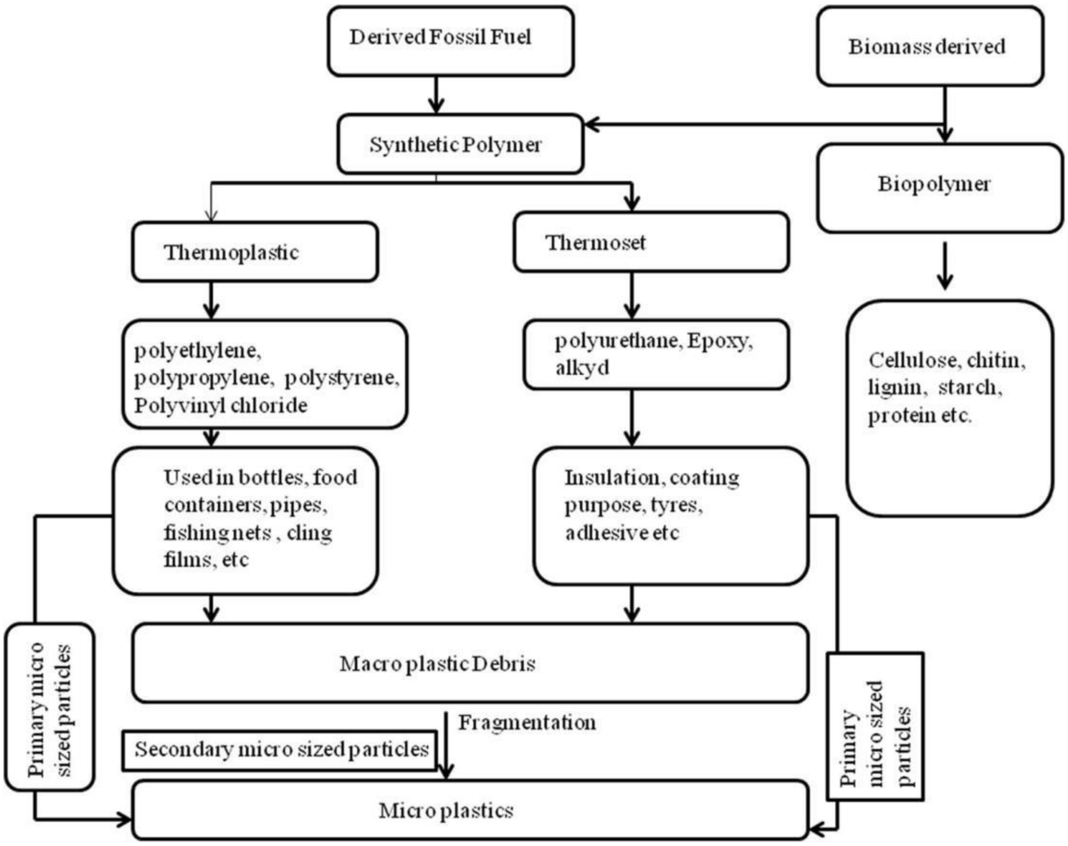


Fig. 2.1: Production process of the artificial and natural polymers and generation of macroplastics and microplastics from synthetic polymers (Sharma and Chatterjee, 2017).

Once enters in the environment, these plastic materials are degraded by various means and lost their structural rigidity (Fig. 2.2). The extensive degradation of plastics finally results into powdery fragments and microscopic-sized plastics, called microplastics (Barnes et al., 2009). These are microparticles having dimensions ranging between few micrometre to 500 µm (0.5 mm). Nowadays, pharmaceuticals and cosmetic industries are using microplastics in various daily used products and contaminating the environment via wastewater, ultimately transferred along food chain and impacting marine ecosystem after reaching into the sea(Sharma and Chatterjee, 2017).

The rise of single-use items is a major factor: By 2015, 60% of all plastic ever produced had already become waste, half of all plastic waste was from packaging alone (Geyer, et. al., 2017). While according to a 2018 estimate, single-use plastics account for 60-95% of global marine plastic pollution (Schnurr, et. al., 2018). Releases 307-925 million litter items into the ocean annually, of which 82% are plastic (González-Fernández, et. al., 2018). The air, too, is a vector for plastic pollution: wear of vehicle tyres and brakes are a major source of microplastic emissions, as is wind abrasion from plastic-coated surfaces, waste processing, roads and agriculture (Evangeliou, et. al., 2020).

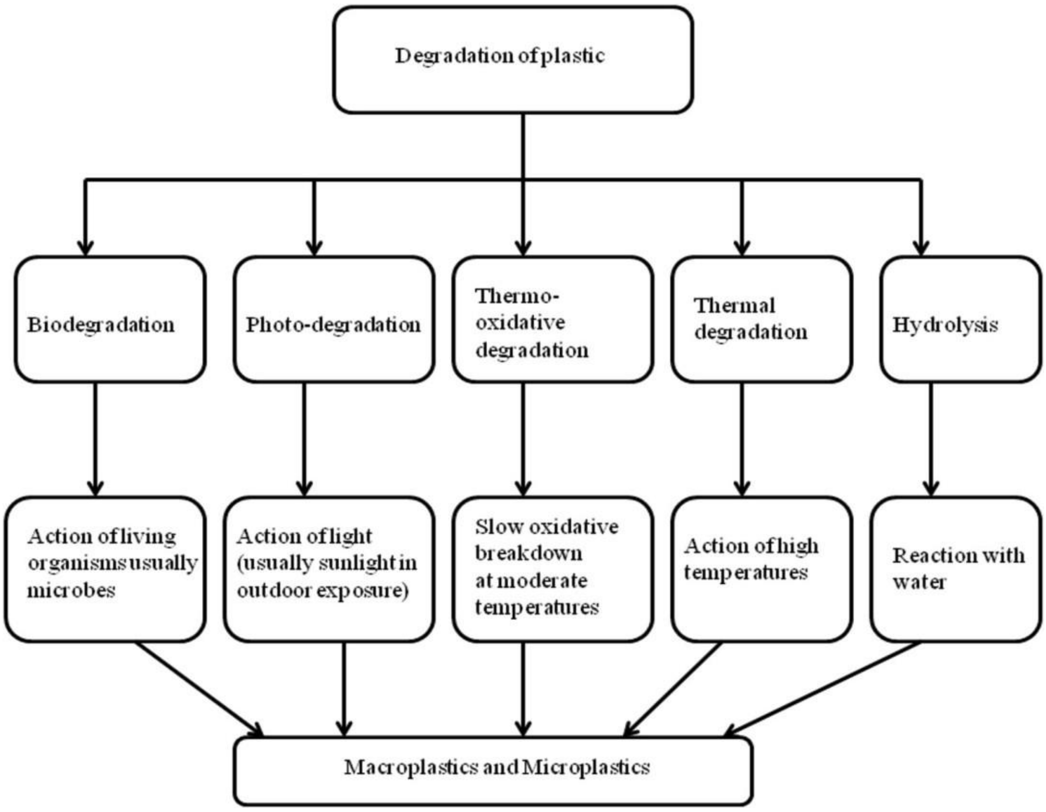


Fig. 2.2: Environmental degradation of plastics under different activity(Sharma and Chatterjee, 2017).

2.2 *Plastic Production and Types*

Plastics are formed by the reaction of small organic molecules called monomers resulting in long polymer chains. Some polymers, such as polyethylene (PE), are formed by addition polymerization. Others, such as nylon, are formed by condensation reactions in which small molecules, often water, are eliminated in the process of creating a longer chain.

The polymers used to make a plastic are almost always mixed with additives, including colorants, plasticizers, stabilizers, fillers, and reinforcements. These additives affect the chemical composition, chemical and mechanical properties and cost of the plastic.

There are two general types of plastics: thermoplastics and thermoset plastics.

Thermoplastics soften when heated, can be reformed and then harden when cooled. This process can be repeated numerous times, which means thermoplastics can be recycled. Thermoplastics include PE (used in toys, shampoo bottles, pipes, etc.), polypropylene (PP-used in food packaging, snack wrappers, auto parts, etc.), polyethylene terephthalate (PET-often used for water and other beverage bottles), polystyrene (PS-used in foam food containers, eyeglasses, building insulation, etc.), polyvinylchloride (PVC-used in window frames, pipes, cable insulation, etc.), and others including polycarbonates (PC) and polyamides (PA).

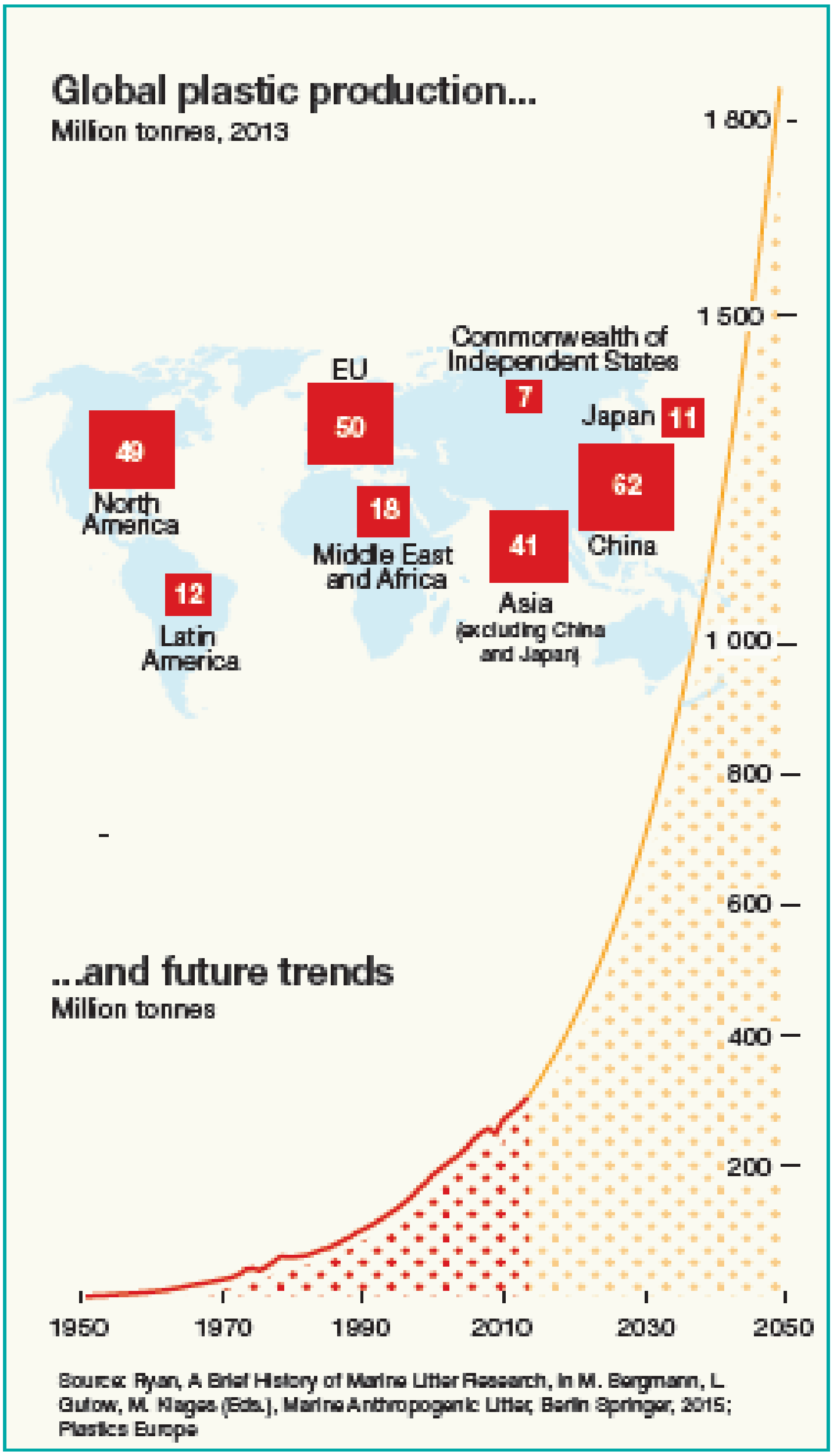
Thermoset plastics will not soften upon heating because permanent chemical bonds form between polymer chains (crosslinking). Thermoset plastics include polyurethane (PUR-used in building insulation, pillows and mattresses, insulating foams, etc.), epoxy resins, some acrylic resins and some polyesters. In the context of drinking-water, PE and PVC are used in water distribution mains and epoxy resins, PUR for relining existing mains, PP for various components and PA as coagulant aids in water treatment. Plastics are also used in membrane filters in water treatment systems. In the context of bottled water, PET is often used to make the bottles and PP and PE to make bottle caps. These materials are often regulated to ensure they do not leach substances (e.g. monomers, plasticizers or other additives) at concentrations of concern into drinking-water.

Biodegradable plastics are plastics that can be decomposed by hydrolysis or ultraviolet (UV) light degradation or the action of microorganisms, usually bacteria. They can be made from either renewable raw materials or from petrochemicals. However, some “biodegradable” plastics require prolonged exposure to temperatures above 50°C to degrade completely. Such conditions exist in industrial composting but are rarely, if ever, met in the environment. Some plastics contain pro-oxidants which promote fragmentation, but there is some controversy as to whether there is any actual biodegradation of these plastics in the environment because they have the potential to form microplastics (UNEP, 2015).

2.3 *Estimates of global quantities of plastics produced*

World plastic production has increased roughly exponentially since large-scale production first began in the 1950s (Fig. 2.3). Global plastic production, excluding fibres, increased from 322 million tonnes (Mt) in 2015 (Plastics Europe, 2017) to 348 Mt in 2017 (Plastics Europe, 2018). With fibres included, global production was estimated to be 381 Mt in 2015 and with additives included, 407 Mt (Geyer, Jambeck and Law, 2017). Considering the estimated worldwide population growth rate and current consumption and waste habits, plastic production is predicted to double by 2025 and more than triple by 2050 (FAO, 2017).

Of total non-fiber plastic production, 36% is PE, 21% is PP, 12% is PVC, and less than 10% each are PET, PUR and PS. The production of polyester PAs and acrylics fiber is the next largest group, much of which is PET. Together, these seven groups account for 92% of all plastics ever made (Geyer et al., 2017). Intentional microplastic production represents <0.1% of total plastic production based on European figures from Plastics Europe (2018) and ECHA (2019).



Source: Ryan, A Brief History of Marine Litter Research, in M. Bergmann, L. Gutow, M. Klages (Eds.), Marine Anthropogenic Litter, Berlin Springer, 2015; Plastics Europe

Fig. 2.3: Global plastic production, (Plastics Europe, 2018)

2.3.1 *Primary microplastic sources*

Primary microplastics are created in multiple forms for a variety of purposes. Of most recent and public concern are those being found in exfoliating facial cleansers, body washes, hand soaps, and even toothpastes. Certain brands of these products began adding polyethylene pieces, or “scrubbers,” to replace the natural exfoliates (such as pumice or apricot husks) once used by manufacturers. After the product’s use, these plastic pieces are washed down the drain with the product and end up in city wastewater systems, (Fendall & Sewell, 2009).

This is cause for concern as it represents an unnecessary contribution of micro-litter of which many consumers are unaware. As the issue has become more publicized by the media.

Other sources of primary microplastics are less well-known by the general public but have existed for quite some time. Microplastics produced for use in air-blasting technology help to remove dust and paint from machinery, boat hulls, and engines (Cole et al., 2011).

2.3.2 *Secondary microplastic sources*

Most of plastic debris found in the water environment comes fromland-based sources, with highly populated or industrialized areas servingas major contributors, due to littering and solid waste disposal (Li et al., 2016). Coastal recreational activities, raw manufacturing materials, wastewater effluent, and refuse site leachate are some of the known sources (Li et al., 2016). River systems, wastewater treatment works, and even extreme weather events transfer these plastics to the sea (Cole et al., 2011).

The remaining twenty percent of marine litter, according to Li et al., (2016) can be attributed to ocean-based sources, with the commercial fishing industry as the prime contributor. Good et al., (2009) estimates that approximately 705,479 tons of discarded fishing gear are lost into the ocean annually. In addition to becoming microplastics, monofilament lines and nylon netting, when discarded, are also known to float at a certain depth and entangle organisms in what's known as "ghost fishing" (Li et al., 2016). Secondary microplastic debris can be derived from various classes of plastics that come from land-based sources, primarily plastic packaging (including disposable single-use items), as well as fishing industry litter (Andrady, 2011). Several broad classes of plastics are used in packaging: Polyethylene (PE), Polypropylene (PP), Polystyrene (PS), Poly (ethylene terephthalate) (PET); and Poly (vinyl chloride) (PVC)". Other classes of plastics that are commonly found in the marine environment are Foamed Polystyrene (PS), Nylon, and Cellulose Acetate (CA) (Andrady, 2011).

2.4 Sources and fate of plastics in freshwater

2.4.1 Sources

Plastic particles less than 5 mm in diameter are referred to as microplastics (California state water resources control). These may be produced as such for industrial purposes (primary microplastics) or generated during degradation processes (secondary microplastics). Cosmetics, abrasive cleaning products and effluents from production sites are considered to be among the most relevant primary sources of microplastics, while plastic waste, laundry fibres, tyre wear, and paint are major sources of secondary microplastics (Hohenblum et al., 2016).

Macroplastic materials are the most visible form of plastic pollution. These larger plastic pieces become fragmented in the environment but are hardly mineralized. As the degradation times of the various polymer types range from a few decades to several hundred years, plastic particles accumulate in marine and freshwater environments. Plastics sources are usually discussed in the context of marine plastics pollution. Land-based sources (including beach litter) can account for about 80 % of the plastic debris (Andrady, 2011). It can be assumed that riverine inputs of plastics into the seas significantly contribute to the marine pollution.

In addition to release from industrial processes, private consumption, traffic and leisure activities, another source of microplastic emissions is agriculture. Low-density polyethylene films from agricultural crop coverings can fragment and leach into soil. In horticulture, synthetic polymer particles are used to improve soil quality (mulching) and as composting additives (Duis and Coors, 2016).

The relevance of tyre dust as an emission source of microplastics was also reported by other authors. According to (Sundt et al., 2014) wear and tear of car tyres is the most important land-based source of microplastics reaching the sea in Norway (2250 tonnes per year). Similar results were estimated for Denmark. According to (Lassen et al., 2015) the major source of secondary microplastics released to the aquatic environment in Denmark is car tyres (500 - 1700 tonnes per year).

Table 2.1 presents data from (Sherrington et al., 2016) on annual European emissions of microplastics into the marine environment from six different sources and potential relevance for freshwater environments.

Table 2.1: Estimated annual European emissions of microplastics into the marine environment from six different sources (Hohenblum et al., 2016).

Emission source	Year	Lower estimated value, (tonnes/year)	Upper estimated value, (tonnes/year)
Tyre dust	2012	25,122	58,424
Pellet spills	Unknown	24,054	48,450
Textiles	2010	7,510	52,396
Building paints	2002	12,300	28,600
Road paints	2006	7,770	18,069
Personal care and cosmetic products (PCCP)	2012	2,461	8,627

2.4.2 *Fate of microplastic in water*

The proportion of plastics present in surface waters and sediments varies depending on the biological (e.g., attachment of bacteria/algae), physicochemical (e.g., plastic density), and hydrodynamic conditions (e.g., mixing of the water column) (Alimi et al., 2018). Factors such as wind, surface water circulation, temperature and salinity influence the distribution of microplastics (Zbyszewski et al., 2014; Corcoran et al., 2015). In the aquatic environment, the rate of degradation of plastics is temperature-dependent, with degradation proceeding more slowly in cold water. Plastics found below the photic zone in the water column degrade very slowly, resulting in high persistence of plastics in the aphotic zone, particularly at the seafloor. In addition, biodegradation of plastics by microorganisms is negligible because of the slow kinetics of biodegradation at sea and the limited oxygen supply for these processes (Andrady, 2015). The main negative effects of plastics are: **Entanglement:** Items like ropes, nets, traps and monofilament lines from abandoned, lost or discarded fishing gear wrap themselves around marine animals causing strangulation, wounds, restricted movement and death. Birds also use marine debris for their nests, which can entrap parents and hatchlings. Fishing lines entangled 65% of coral colonies in Oahu, Hawaii, (Yoshikawa, and Asoh, 2004) and 80% of these colonies were entirely or partially dead. Even in the remote Arctic deep sea, up to 20% of sponge colonies have been entangled with plastic, and entanglements increased over time, (Parga, et. al., 2020).

Ingestion: Marine animals of all kinds from apex predators down to the plankton at the base of the food chain ingest plastic. This can cause serious harm to the animals, affecting food uptake by creating a false sense of satiation or blockages in digestive systems, as well as leading to internal injuries. Laboratory experiments have shown reduced growth in fish when their food is contaminated by high volumes of microplastics (Naidoo, and Glassom, 2019) while at the other extreme a single plastic drinking straw in its digestive system likely caused the death of a whale shark in Thailand (Haetrakul, et. al., 2009). Plastic ingestion in seabirds is global, pervasive, and increasing. It has been estimated that up to 90% of all seabird (Wilcox, et. al., 2015) and 52% of all sea turtle individuals nowadays ingest plastics (Schuyler, et. al., 2015). Some studies have shown altered or decreased food uptake, and negative impacts on growth (Byrd, et. al., 2014; De Stephanis, et. al., 2013) immune response, fertility and reproduction as well as altered cell functions and behaviors in the impacted species; with levels of harm directly related to exposure concentrations (Prokić, et. al., 2019). **Smothering:** Plastic pollution deprives corals, sponges and bottom- dwelling animals of light, food and oxygen, making sediment oxygen- deficient and reducing the numbers of organisms in the sediment (Green, et. al., 2015; Balestri, et. al., 2017). This can negatively affect ecosystems and give pathogens a foothold, which can have detrimental effects on marine life. Smothering is particularly harmful in coral reefs and mangroves (Fig. 2.4).

Chemical pollution: Not all the ingredients in plastics are harmful, but many of them are and can leach from plastics into the marine environment. The smallest plastic particles can cross into the body cells and some of them can even reach the brains of marine animals (Rochman, 2015;Mattsson, et. al., 2017).

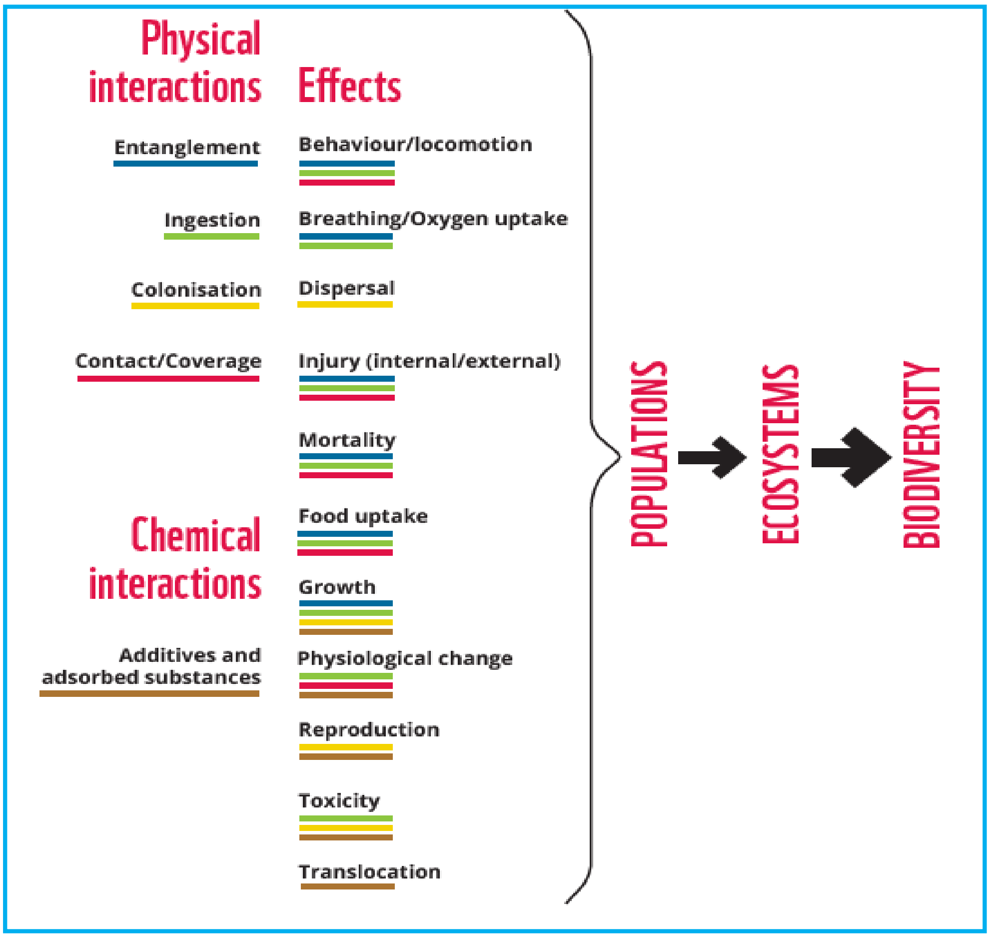


Fig. 2.4: Diagram of the most frequently reported interactions and theireffects on organisms (LITTERBASE). The colors represent the respective interactions, (Green, et. al., 2015).

2.5 *Occurrence of microplastics in water*

Microplastics are ubiquitous in the environment and have been detected in marine water, wastewater, fresh water, food, air and drinking- water, both bottled and tap water.

Microplastics enter freshwater environments in a number of ways: primarily from surface run-off and wastewater effluent (both treated and untreated), but also from combined sewer overflows, industrial effluent, degraded plastic waste and atmospheric deposition. However, there are limited data to quantify the contribution of each the different inputs and their upstream sources. Further, the limited evidence indicates that some microplastics found in drinking-water may come from treatment and distribution systems for tap water and/or bottling of bottled water.

A recent systematic review of the literature identified 50 studies detecting microplastics in fresh water, drinking-water or wastewater (Koelmans et al., 2019). The lack of standard methods for sampling and analysing microplastics in the environment means that comparisons across studies are difficult. In addition, few studies were considered fully reliable. Nevertheless, some initial conclusions can be drawn.

In fresh water, the frequency of microplastic particles by polymer type was consistent with plastic production volumes and plastic densities. A wide range of shapes and sizes were found.

For both freshwater and drinking-water studies, the smallest particles detected were often determined by the size of the mesh used in sampling, which varied significantly across studies. Particle counts ranged from around 0 to 10^3 particles/L in fresh water. In drinking-water, where smaller mesh sizes are typically applied, concentrations in individual samples ranged from 0 to 10^4 particles/L and mean values ranged from 10^3 to 10^3 particles/L. The smallest particle size detected was 1 μm, but this result is constrained by current methods. In most cases, freshwater studies targeted larger particles, using mesh sizes that were an order of magnitude larger than those used in drinking-water studies. Thus, direct comparisons between data from freshwater and drinking-water studies cannot be made. Microplastics are frequently present in the freshwater environment, with studies reporting particle counts ranging from around 0 to 103 particles/L (Koelmans et al., 2019). The relative concentration of microplastics detected is dependent on the location, the sampling technique (mesh size) and method of analysis. Larger sieves will not retain small particles, so total particle numbers will be lower from samples taken with large sieves than from small sieves.

In Koelmans et al. (2019), 31 studies of microplastics in fresh waterwere identified and the six highest-scoring studies in terms of methodological quality (studies with a total accumulated score (TAS), greater than nine or 50% of the maximum value), are summarized in Table 2.2 below. The reported concentrations of microplastics ranged from averages of 4.7 particles/L in the study using a 48 µm sieve down to 0.00026 particles/L in a study using a 333 µm sieve. Not surprisingly, the one groundwater study found low concentrations of microplastics relative to other water types despite using a very small sieve size (3 µm). A priori, low particle counts would be expected in many groundwaters because of the protection afforded by the overlying strata.

Table 2.2: Summary of reported microplastic particle numbers fromfreshwater studies that scored highest for quality

Location	Results reported (particles/L)	Sieve size (µm)	Study	Quality Score (TAS)[a]
Groundwater, Germany	Average:b 0.7 × 10-3 Range:b 0 –7 × 10-3	3	Mintenig et al., 2019	14
Three Gorges Reservoir, China	Average: 4.7 Range: 1.6–12.6	48	Di and Wang, 2018	10
Dongting Lake and Hong Lake, China	Averages: 1.2 and 2.3 Ranges: 0.9–2.8 and 1.3–4.7	50	Wang et al., 2018	15
Wuhan, China	Range: 1.6–8.9	50	Wang et al., 2017	10
Rhine river, Switzerland, France, Germany, Netherlands	Average: 0.0056	300	Mani et al., 2015	10
Western Lake Superior, USA	Average: 0.00026	333	Hendrickson, Minor, and Schreiner, 2018	11

a TAS = total accumulated score, as reported in Koelmans et al. (2019). The maximum scoreis 18 and is calculated by adding scores for nine quality criteria, where for each criterion, a score of 0, 1 or 2 is assigned. See the annex for an overview of the nine quality criteria and for each study, the individual scores against each criteria. TAS values are underlined when all underlying scores are non-zero.

b These values include samples of groundwater and drinking-water derived from groundwater subject to further filtration.

A direct comparison of data between studies of fresh water and drinking-water, cannot be made because in most cases freshwater studies targeted larger particles, using mesh sizes that were an order of magnitude larger than those used in drinking-water studies. Consequently, the small particles detected in drinking-water are not detected in most freshwater studies.

Only in the last few years have scientists begun to study how microplastics are affecting freshwaters. According to Eerkes-Medrano et al., (2015) this pollutant has been detected in freshwater systems in North America, Europe, and Asia, with the first biotic studies revealing that microplastic particles are indeed being ingested by a range of freshwater fauna. In their 2015 review, the authors summarize where many of the recent studies have found microplastic debris in both freshwater and its sediment:

Microplastics have been found in: North America, in the Los Angeles basin (Moore et al., 2011), the North Shore Channel of Chicago (Hoellein et al., 2014), the St. Lawrence River (Castañeda et al., 2014) and the Great Lakes (Zbyszewski et al., 2014) in Europe, in Lake Geneva (Faure et al., 2012), the Italian Lake Guarda (Imhof et al., 2013), the Austrian Danube river, the German Elbe, Mosel, Neckar, and Rhine rivers (Wagner et al., 2014), and the UK Tamar estuary (Sadri and Thompson, 2014); and in Asia, in Lake Hovsgol, Mongolia (Free et al., 2014). (Eerkes-Medrano et al., 2015).

To date, freshwater microplastic studies have varied greatly in their magnitude and research focus, with little standardization in sampling methods and laboratory analysis. Results generally provide plastic count, concentration, and/or plastic weight; with the pieces often sorted by type (foam, line, pellet, bead, fragment, etc.), chemical composition, color, and/or size.

2.6 Possible human health risks associated with micro plasticsin drinking-water

The human health risk from microplastics in drinking-water is a function of both hazard and exposure. Potential hazards associated with microplastics come in three forms: the particles themselves which present a physical hazard, chemicals (unbound monomers, additives, and sorbed chemicals from the environment), and microorganisms that may attach and colonize on microplastics, known as biofilms. Based on the limited evidence available, chemicals and microbial pathogens associated with microplastics in drinking-water pose a low concern for human health. Although there is insufficient information to draw firm conclusions on the toxicity of nanoparticles, no reliable information suggests it is a concern.

Interest in microplastics in drinking-water was stimulated initially by studies (Kosuth, Mason and Wattenberg, 2018; Mason, Welch and Neratko, 2018) that reported the presence of microplastics in tap water and in bottled water. Since then, several additional studies have been published (Oßmann et al., 2018; Pivokonsky et al., 2018; Schymanski et al., 2018; Strand et al., 2018; Uhl, Eftekhardadkhah and Svendsen, 2018; Mintenig et al., 2019) leading to genuine questions and concerns about whether the presence of microplastics in drinking-water poses a risk to human health.

Besides their entanglement in larger plastic materials, aquatic organisms often mistake plastic particles as food. The physical consequences of swallowing plastics are impaired food ingestion and altered feeding behaviour (Gregory, 2009). Moreover, sharp-edged particles can cause mechanical injuries in the intestinal tract, blocking or reducing ingestion and causing indigestion (Wright et al., 2013). The ingestion of plastic particles depends on the ratio of particles to organisms and on the feeding behaviour of those organisms (Setälä et al., 2016).

The uptake of plastic particles has been reported for more than 250 species, particularly for marine species, whereas freshwater biota has been addressed in only a few studies so far. It should be noted that many organisms are able to egest unintentionally ingested plastic materials (McCormick et al., 2014).

In addition to physical effects, immune reactions due to the ingestion of plastics (and perhaps tissue transfer) and/or mechanical injuries have been described (Wegner et al., 2012), together with physiological effects due to a reduced energy supply (Cole et al., 2013).

Toxic effects could be caused by additives (e.g. phthalate-based plastizisers and bisphenol A (BPA)) originally being in the plastic particles or by substances gradually adsorbing to the plastic particles in the environment due to hydrophobic interactions depending on the type of plastics (Bakir et al., 2012). Additives including persistent pollutants can be ingested together with plastic particles depending on the polymer type, plastic additives and on the sorption properties of environmental chemicals. For example, polyethylene accumulates a larger spectrum of organic pollutants than polypropylene or polyvinyl chloride. Persistent organic pollutants can cause hepatic damage when in liver tissue (Rochman et al., 2013).

Toxic or endocrine disrupting effects due to polymers and/or additivessuch as nonylphenolethoxylate have already been described in the literature. In the environment nonylphenolethoxylate is degraded to nonylphenol. (Browne et al., 2013) reported that nonylphenol desorbing from polyvinyl chloride accumulates in the tissue of the lugworm Arenicola marina and affects the phagocytic activity. Contrary to that, a study based on a biodynamic model indicates that nonylphenol leached from polyethylene has not been significantly ingested by Arenicola marina. This example emphasises the fact that our current knowledge is thus far too limited to enable reliable risk assessments. Plastic particles can act as sources and as sinks of pollutants depending on the adsorption/desorption balance. There is evidence showing that adsorbed pollutants may be desorbed in the intestinal tract of organisms due to altered milieu conditions (pH, temperature) and become bioavailable (Bakir et al., 2014). Indeed, bioaccumulative effects and the trophic transfer of microplastics (and of the sorbed pollutants) have already been reported (Setälä et al., 2014). A further concern is that macro- and microplastics could serve as vectors for invasive species (Gregory, 2009), harmful algal bloom (HAB) species (Masó et al., 2003) and opportunistic pathogens (McCormick et al., 2014). These organisms may be additional stressors for an-thropogenically influenced ecosystems or enter more pristine regions with floating plastic particles.

2.7 *Microplastic concentrations reported in drinking-water*

A total of nine studies measured microplastics in drinking-water. Particle concentrations reported in individual samples ranged from 0 to over 10^4 particles/L and mean values ranged from roughly 10^{-3} particles/L in a study of drinking-water derived from groundwater to over 10^3 particles/L. In general, groundwaters are well protected from particulate contamination. Similarly, conventional drinking-water treatment is expected to provide an effective barrier for a wide range of particle sizes

Different studies looked at different ranges of particle sizes and had different cut-offs for reporting particle sizes. Studies typically used a filter with a mesh or pore size of less than 10 μm and in some cases characterized particles as small as 1 μm. In the context of drinking-water, an arbitrary cut-off of 0.1 mm in length was suggested to distinguish large particle from small particles (Koelmans et al., 2019). Where studies looked at both large and small particles, small particles tended to be more numerous. Since there were so few studies, each is described in more detail in the annex. They are described in the annex in order of decreasing study quality, as determined by the TAS quantitative assessment, primarily from Koelmans et al. (2019). Some key results of the eight of the nine scored studies are summarized in Table 2.3. There was insufficient detail to fully assess the study quality of one of the studies, and therefore, is not summarized in the below table, but a short description is included in the annex for completeness.

Table 2.3: Summary of reported microplastic or microplastic-like particle numbers a and particle characteristics from drinking-water studies

Author	Water type	Lower size bound (µm)	Particles/L in sample (average)	Particles/L in sample (average)
Oßmann et al. (2018)	Bottled (mineralwater) • Glass • Single use PET • Reusable PET	1	3074–6292 2649 4889	384
Pivokonsky et al. (2018)	DWTP from surface water sources (3 sites)	1	628 338 369	< 5% of counts insamples
Schymanski et al. (2018)	Bottled Single use Returnable Glass Beverage carton	5–20	14 118 50 11	14 ± 13
Mason, Welch andNeratko (2018)	Bottled	6.5–100 lower bound based on microscope and software	315	23.5
Strand et al. (2018)	Tap from ground-water sources	10–100	0.2, 0.8 and 0.0 (LoD = 0.3)d	Unknown
Mintenig et al. (2019)	Tap from ground-water sources	20	0.0007	0.67 particles/L 0.3 fibres/L
Uhl, Eftekhardadkhah,and Svendsen (2018)	Tap form 24 sources	60	Average not reported since only a single result above LoQ (that result was 5.5)	0.5 (LoQ = 4.1 LoDd = 0.9)

Mason, Welch andNeratko (2018)	Bottled	>100	10.4	4.15
Strand et al. (2018)	Tap from ground-water sources	>100 (10 µm sievesize)	0.312 (LoD = 0.58)	0.26
Kosuth, Mason and Wattenberg (2018)	Tap from unspecified sources	100 lowest reported	5.45	0.33 (based on 5 particles in 30 blanks (ea. 500 mL)

[a] For details on whether particles identified were confirmed to be microplastics, see summaries of the individual studies in the annex.

[b] TAS = total accumulated score. The maximum score is 18 and is calculated by adding scores for nine quality criteria; for each criterion, a score of 0, 1 or 2 is assigned. TAS values are underlined when all underlying scores are non-zero.

c While the Mintenig et al. study was the highest rated study, the Mason et al. study was the only drinking-water study that had a non-zero score in all key quality criteria. Aside from Strand et al. and Uhl et al., the reported TAS values are from Koelmans et al. (2019). Strand et al. and Uhl et al. were assessed separately by one of the authors of the Koelmans et al. (2019) publication. See the annex for an overview of the individual scores against each quality criteria, including scores for Strand et al. and Uhl et al.

d LoD/LoQ = Limit of detection/Limit of quantification.

2.8 *Particle size, shape and polymer type in fresh water and Drinking water*

Size, shape and polymer type are characteristics of microplastics that may influence how they are transported in water and may have implications for toxicity and efficacy of drinking-water treatment. Below is a brief summary of the characteristics of microplastic particles found in freshwater and drinking water samples.

The smallest particle sizes measured in fresh water are determined by the mesh size of the trawl net, which is typically 300 µm, although a few studies have been conducted with finer meshes. Higher particle countstend to be reported when smaller particles are measured (Koelmans et al., 2019). Based on current analytical capabilities, particle sizes measured in drinking-water can be as low as 1 µm. This does not imply that particles smaller than 1 µm are not in drinking-water, rather detection is limited by currently available methods.

In freshwater studies, the shape of particles varied widely, with fragments, fibres, film, foam and pellets being the shapes most often reported (Koelmans et al., 2019). Fragments and fibres were the predominant particle types found in drinking-water. However, as stated by the authors, this data on shapes should be interpreted with caution, as they represent only a subset of isolated particles, which may or may not represent all particles. Also, each study focused on specific sizes and defined ambiguous shapes differently such as nurdle, pellet, pre-production pellet, resin or granule, making it difficult to compare across studies. Studies also differed in the extent to which their water samples represented the studied water systems or water type, which varied over space and time. PP, PE, PS, PVC and PET were the polymers detected most frequently, an order that agrees roughly with production volumes. As described above, in addition to the production volume, polymer density is a key factor in determining which particles might be detected in studies that sample the upper layer of water. In bottled water, there is some evidence that microplastic occurrence appears to be at least partially attributable to the bottling process and/or packaging.

Only limited quantified data are available to demonstrate the efficacy of microplastic removal across drinking-water treatment processes. However, conventional drinking-water treatment (coagulation, sedimentation and filtration) is designed to remove particulates and is therefore expected to effectively remove microplastics, particularly when optimized to produce treated water of low turbidity. When optimized, conventional treatment can remove particles smaller than a micrometre. Advanced treatment using membranes would be expected to achieve 100% removal of microplastics larger than 0.001 to 1 µm, with removal capabilities >0.001 µm for nanofiltration, >0.01 µm for ultrafiltration and >1 µm for microfiltration. Since microplastics removed through both wastewater and drinking-water treatment will be incorporated into sludge or other waste streams, there is potential for particles to return to the environment depending on the use or disposal practices.

2.9 *Collecting water samples*

Collection of water samples is the first step of microplastics sampling methodologies (Table 2.4). The choice between sampled medium is dependent on available equipment but also the objective of the work. For instance, sampling the water column may be the most adequate medium if the objective is to determine the exposure of pelagic organisms. However, microplastic distribution is largely influenced by meteorological, temporal and geographical factors that may compromise reproducibility of the results. On the other hand, methodology and quantity of sampled material may influence representativeness of results. Results are usually expressed as total microplastics per unit of sample (e.g. L^{-1}, in water), sometimes providing detailed classifications of size classes, color and shape (e.g. fiber, particle, and fragment) (Prata et al., 2019).

Several methods were described suggesting different procedures for sampling, sample treatment and sample identification of micro-, meso- and macroplastics. However, no harmonised procedures have been established up to now (Reifferscheid et al., 2017).

Depending on the compartment investigated several bulk or volume reduced sampling methods have been commonly used for microplastics so far. Volume-reduced sampling methods were generally employed for water samples: manta or plankton nets with individual mesh sizes for the water surface; and various nets, which are fixed on riverbeds (e.g. eel nets, drift nets) or flow-through centrifugation in combination with cascade sieves for layers below the water surface (Reifferscheid et al., 2017).

Table 2.4: Methods of sample collection in water (Prata et al., 2019)

Sample	Type	Advantages	Disadvantage
Water	Neuston and Mantanets	Easy to use; Sample large volumes of water; Largely used (good to compare between locations); Produces large numbers of microplastics for further testing.	Expensive equipment;Requires boat; Time-consuming; Potential contamination by vessel andtow ropes; Lower limit of detection is 333 mm.
	Planktonnet	Easy to use; Lowest limit of detection 100 mm; Quick to use; Samples medium volumes of water.	Expensive equipment;Requires boat; Static sampling requires water flow;May become clogged or break; Sampling of lower volumes of water than Manta trawl.
	Sieving	Does not require specialized equipment nor boat; Easy to collect samples.	Laborious and time consuming;Samples medium volumes; Manual transfer of water with buckets
	Pumps	Samples large volumes of water; Effortless; Allows choice of mesh size.	Requires equipment; Requires energy to work; Potential contamination by the apparatus; May be difficult to carry between sampling locations.
	Filtration or Sievingex situ	Easy to collect samples; Known volume of water; Allows choice of mesh size.	Sampling of low volumes; Transportation of water samples to thelab; Potential contamination by the apparatus; Time consuming depending on mesh size.

Table 2.5 presents an overview on sampling, sample treatment and identification methods applied in relevant monitoring studies on microplastics (Hohenblum, 2016).

The majority of sample treatment methods include enzymatic digestion over several weeks with constant stirring (AWI et al., 2014). Alternative or additional methods include the use of oxidizing agents such as H_2O_2 (Mani et al., 2015), and (AWI et al., 2014) or acids such as HNO_3 are applied to remove organic materials. However, there is risk of damaging the plastics, either by oxidation, or in the case of the former, by mechanical friction.

Table 2.5: Overview on methods of sampling, sample treatment andidentification of microplastics (Hohenblum, 2016).

	AWI et al., (2014)	Hohenblum et al., (2015a)	Löder et al., (2015)	Mani et al., (2015)
Sampling location	sewage water treatment plant outlet	Danube River	North Sea	Rhine River
Sampling device	cartridge filter 10 μm	nets 500-, 250-, 41 μm	net 500 μm	net 300 μm
Density separation	ZnCl2	NaCl	sieving 500 μm	NaCl
Organic digestion	Sodium dodecyl sulphate, enzymatic degradation, H2O2	None	enzymatic degradation	enzymatic degradation
Water removal	sieving, filtration on aluminium oxide filter	thermal drying	filtration on aluminium oxide filter	sieving 300 μm
Detection/ identification	Identification: > 500 μm: ATR-FT-IR < 500 μm: FPA-μFT-IR	detection: visual identification: ATR-IR of sub-samples (particles > 2 mm)	identificatio: μFT-IR	detection: visual (particles > 300 μm identification: random controls by FT-IR)

2.10 *Composition, properties, and uses*

2.10.1 *Composition*

All plastic materials are formed from long-chain polymers of very high molecular weight, often measured in the hundreds of thousands of kilodaltons. Synthetic polymers first appeared in the early 20th century, leading to the manufacture of plastic products such as Bakelite and nylon (commercial name for polyamides). Since then, polymer science has evolved, with a greater mechanistic understanding of the interrelationships between polymer structure, morphology, and physical and mechanical behavior. This has resulted in the production of a myriad of plastic materials with varying physical and chemical properties. Polymerization, the synthesis of polymers, can occur following one of two main processes:

chain polymerization or stepwise polymerization. The process used to form polymers greatly influences their physical properties. Common chain polymer structures include polyethylene (PE), polypropylene (PP), polystyrene (PS), and polyvinyl chloride (PVC), whereas common stepwise polymers include nylons, polyethylene terephthalate (PET), polycarbonate (PC), and polyurethane (PU). These represent many of the most common forms of plastics typically found as environmental contaminants.

2.10.2 *Physical properties*

The physical properties of plastic, such as rigidity, flexibility, and elasticity, are influenced by the polymer's molecular weight distribution and organization of polymer chains (Verschoor, 2015). Generally, high molecular-weight polymers with a complex organization that leads to strong covalent bonds between the polymers can result in the formation of a rigid plastic with a high melting point. In contrast, linear polymer organization with low molecular-weight distribution results in a more flexible plastic with a lower melting point. Combinations of different molecular weight distributions, different polymer chain organization, and/or blends of different types of polymers can produce a material that will be effective for its intended use (Sperling, 2006).

Microplastics can exist as fibres, fragments, spheres, pellets, films, and foams, as shown in Fig. 2.4. In general; certain shapes of microplastics originate from certain plastic products. For example, fibres are typically shed from fabrics, such as clothing and upholstery, whereas pellets are typically from industrial feedstock (Rochman et al., 2019).

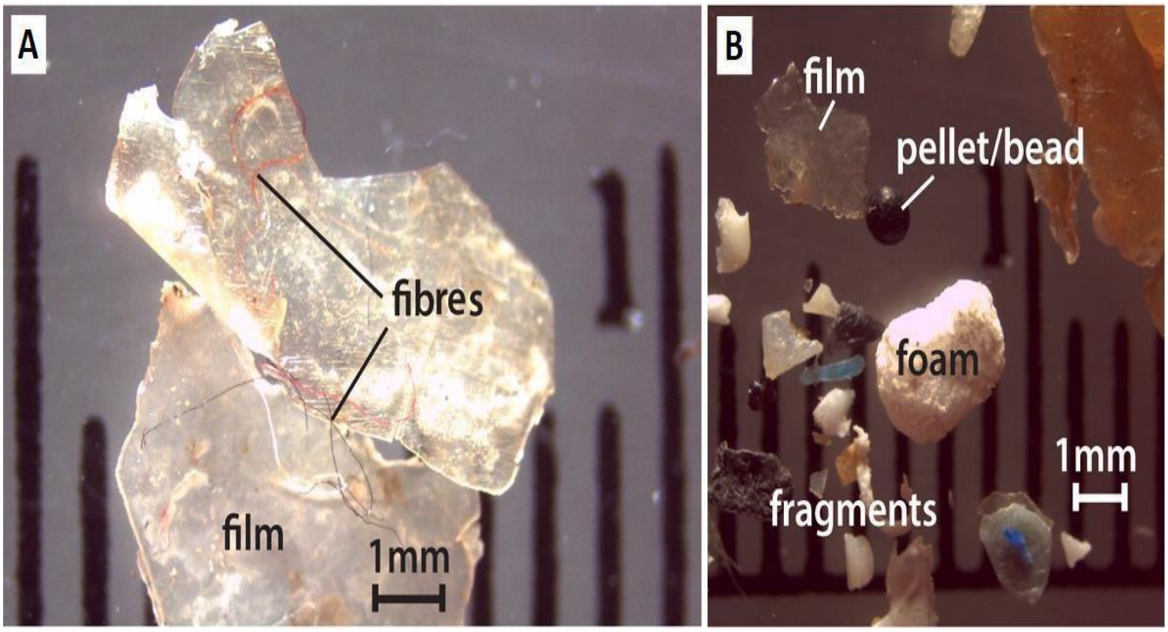


Fig. 2.4: Microplastics found in the environment: (A) Contains film and fibres, (B) Contains film, pellet/bead, fragments, and foam. (Baldwin et al., 2016).

2.10.3 *Density*

Density is a key property that influences the environmental fate of plastics. Densities of plastic polymers such as PE, PS and PVC can range from 0.9 to 2.3 g/cm^3 (Rochman et al., 2019).

Table 2.6 presents the densities of various plastic polymers. Polymerswith a density greater than 1 are denser than water and are expected to sink, while those with a density less than 1 are expected to float. By analogy, the environmental fate and transport of macroplastics or microplastics released to the atmosphere are also likely to be influenced by their density. For example, denser microplastics are less likely to be readily dispersed by the wind. The density of plastics and their buoyancy in water can also be influenced by the coating of plastics with microorganisms, algae, or plants (i.e., biofilms). Other factors, such as shape and size, can also govern the fate of plastics in the environment, (Rochman et al., 2019).

Table 2.6: Selected polymer densities, (Rochman et al., 2019)

Name	Density (g/cm^3)
Polyethylene	0.965 – 0.971
Polypropylene	0.90 – 0.91
Polystyrene	1.04 – 1.10
Polyamides (nylon)	1.02 - 1.05
Acrylic	1.09 – 1.20
Polyvinylchloride	1.16 – 1.58
Poly methylacrylate	1.17 – 1.20
Polyurethane	1.20
Polyester	1.23 – 2.3
Polyethylene terephthalate – a specific type of polyester	1.37 – 1.45

2.11 *Occurrence in drinking water*

A limited number of studies have measured microplastics in tap water, and even fewer are considered reliable due to concerns with quality assurance measures. Average microplastic particle concentrations in tap water have been reported to range from 0.0007 to 628 particles/L, and microplastics as small as 1 µm in size have been measured in drinking water (Pivokonsky et al., 2018). Due to the limitations of existing detection techniques, no information is available on the occurrence of particles below 1 µm in size. The most predominant polymer types detected were PET and PP in the form of fibers and fragments (WHO, 2019).

2.12 *Environmental entry pathways*

Fig. 2.5 shows the various pathways by which plastics enter the environment. There are various reports on the exposure of freshwater environments with effluent from public and /or industrial waste water treatment plants. Pollution of surface waters by plastic materials can also occur by atmospheric inputs. For instance, plastic litter or microplastics, respectively, can be transported by the wind from areas of infrastructure, agriculture and industry, from uncovered landfills or during waste collection (Schwaiger et al., 2016).

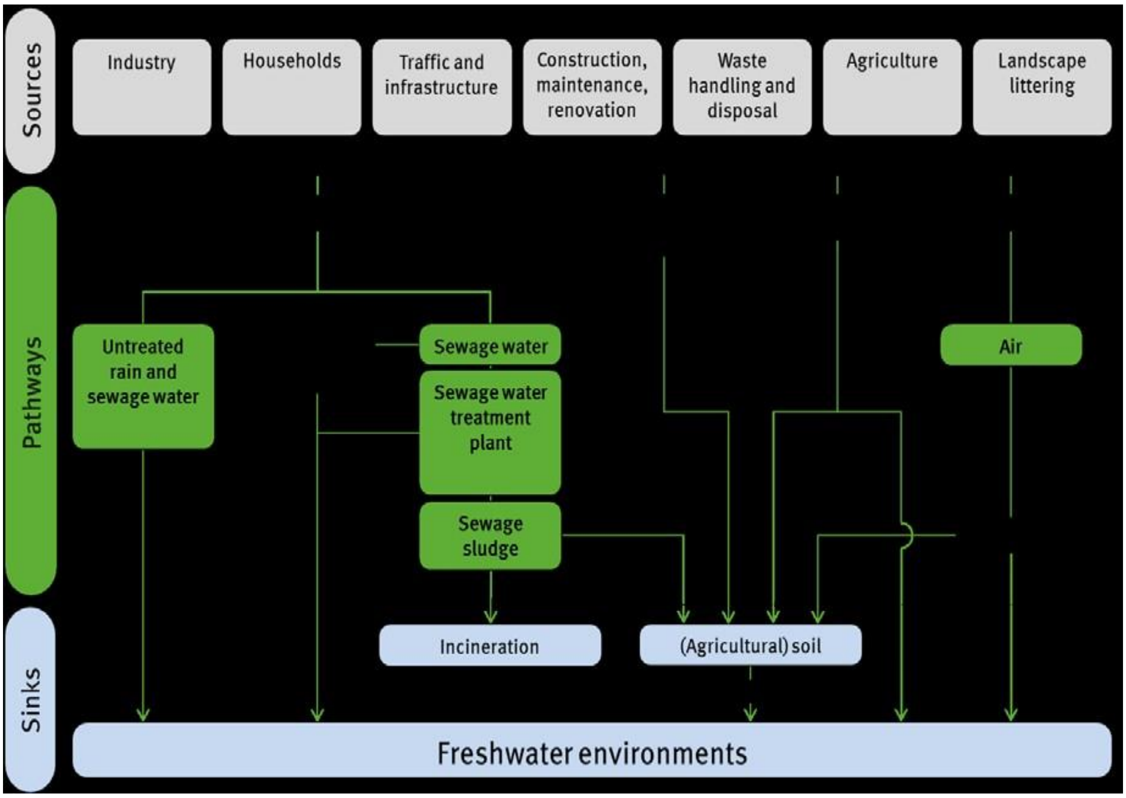


Fig. 2.5: Possible exposure pathways of microplastics into freshwater environments and catchment areas, (Schwaiger et al., 2016).

2.13 *Effects in the environment*

Results from laboratory studies have shown that microplastics may alter freshwater communities, with effects on the individual or population level and of physical or chemical nature. To date, most effect studies have focused on acute toxicity to organisms living in the water column whereas very little is known about toxicity behaviors and sub-lethal effects. Furthermore, the impacts on sediments have been often overlooked, even though microplastic pollution may pose a particular threat to sediment-dwelling organisms. Also of concern is the fact that plastic particles can act as vectors for invasive species, pathogens and hydrophobic pollutants in aquatic systems. For example, stabilizers and additives such as flame retardants, anti-oxidants and UV-blockers can leach into the environment or be ingested by biota together with the plastic (Beate Bänsch-Baltruschat, et al., 2017).

2.14 *Fourier transform infrared spectrometer (FTIR)*

Fourier transform infrared spectrometer is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. An FTIR spectrometer simultaneously collects high-resolution spectral data over a wide spectral range. This confers a significant advantage over a dispersive spectrometer, which measures intensity over a narrow range of wavelengths at a time (Charu Dwivedi et al., 2017).

The term Fourier-transform infrared spectroscopy originates from the fact that a Fourier transform (a mathematical process) is required to convert the raw data into the actual spectrum. The goal of absorption spectroscopy techniques (FTIR, ultraviolet - visible ("UV - vis") spectroscopy, etc.) is to measure how much light a sample absorbs at each wavelength. The most straightforward way to do this, the "dispersive spectroscopy" technique, is to shine a monochromatic light beam at a sample, measure how much of the light is absorbed, and repeat for each different wavelength. Fourier transform spectroscopy is a less intuitive way to obtain the same information. Rather than shining a monochromatic beam of light (a beam composed of only a single wavelength) at the sample, this technique shines a beam containing many frequencies of light at once and measures how much of that beam is absorbed by the sample. Next, the beam is modified to contain a different combination of frequencies, giving a second data point. This process is rapidly repeated many times over a short time span. Afterwards, a computer takes all this data and works backward to infer what the absorption is at each wavelength. The beam described above is generated by starting with a broadband light source one containing the full spectrum of wavelengths to be measured. The light shines into a Michelson interferometer a certain configuration of mirrors, one of which is moved by a motor. As this mirror moves, each wavelength of light in the beam is periodically blocked, transmitted, blocked, transmitted, by the interferometer, due to wave interference. Different wavelengths are modulated at different rates, so that at each moment or mirror position the beam coming out of the interferometer has a different spectrum. As mentioned, computer processing is required to turn the raw data (light absorption for each mirror position) into the desired result (light absorption for each wavelength). The processing required turns out to be a common algorithm called the Fourier transform. The Fourier transform converts one domain (in this case displacement of the mirror in cm) into its inverse domain (wavenumbers in cm^{-1}). The raw data is called an "interferogram"(https://en.wikipedia.org/wiki/Fouriertransform_infrared_spectroscopy).

2.15 *Micro-Raman spectrometer (MRS)*

Raman spectroscopy is a molecular vibrational spectroscopy which is complementary to infra-red spectroscopy. The energy difference between the incident and scattered photons is characteristic of the vibrational modes of the material and are given in wave number scale (v — v'/c) with respect to the exciting radiation. Raman spectroscopic analysis of fluid inclusions is possible using an optical microscope with a high numerical aperture objective to focus the exciting radiation (the laser beam) onto the sample. The objective also collects the scattered light.

In the earlier generation of Raman microprobes (type MOLE). A double monochromatic scatters the different radiations. By rotation of the gratings, radiations are focused through the exit slit, then onto the photocathode of a photomultiplier for the measurement of the intensity of the Raman signal. Multi-channel detection based on the spectrograph principle is now possible thanks to the recent improvements in photodiode array detectors. A foremonochromator, made of two gratings coupled in subtractive mode, selects a band pass which is scattered and analyses by a multichannel detector consisting of an array of 512, 700 or 1024 intensified photodiodes. For a given integration time per channel, the signal-to-noise ratio obtained with multi-channel detection is increased by around one order of magnitude compare with single-channel detection. (https://www.sciencedirect.com/topics/chemistry/micro-raman-spectroscopy)

2.16 *Scanning Electron Microscope (SEM)*

Scanning electron microscopy (SEM), which is also recognized as SEM analysis or SEM technique, has been used worldwide in manydisciplines. It can be regarded as an effective method in analysis of organic and inorganic materials on a nanometer to micrometer (μm) scale. SEM works at a high magnification reaches to 300,000x and even 1000000 (in some modern models) in producing images very precisely of wide range of materials. Energy Dispersive X-ray Spectroscopy (EDS) works together with SEM to provide qualitative and semi-quantitative results. Both techniques, together, have the potential to introduce fundamental information on material composition of scanned specimens, which could not be provided by the common laboratory tests. The analysis is done through SEM equipment, which is a very developed SEM Quanta device for materials science. The device consists of variable pressure system with the ability to hold any samples (even wet or samples with minimum preparation). The device allows to analyze samples with diameter up to 200 mm and height of 80 mm. Magnification of the device range from 5x to 300,000 x. Materials that can be used in SEM is organic and solid inorganic materials including metals and polymers .

2.17 *Completed studies*

According to the results of the European survey, monitoring studies were completed in Austria, (Danube River), Belgium (Leie River), Germany (Rivers Weser, Rhine and its tributaries) and the Netherlands (Lake Ijssel, Rivers Meuse and Rhine), together with an international investigation covering rivers in Italy (Po), the Netherlands (Rhine), Romania (Danube) and Sweden (Dalålven).

In a preliminary study of the Danube River (Austria), the abundance of microplastics (> 500 μm) in the water column was evaluated by Hohenblum et al., (2015a) on behalf of the Federal Environmental Agency, Austria. The study included the development of a method that takes into account the vertical, horizontal and temporal variability of plastic transport in the water column. According to the authors, plastic transport and the annual plastic load in the river can be reliably calculated only by multi-point sampling. The study was conducted at two sampling sites: in Aschach, close to the river's entrance into Austria, and in Hainburg, at its exit to Slovakia. Concentration ranges of 0.039–0.205 mg/m^3 and 0.029–0.516 mg/m^3, respectively, were reported. The majority of the sampled plastic particles were PE and PP polymers, as determined by attenuated total reflectance infrared spectroscopy (ATR-IR). Over 50 % of the extracted plastic particles consisted of fragments, 4–10 % were pellets and 2.1–2.8 % were green lenticular flakes (Hohenblum et al., 2015a).

In Belgium, two sampling campaigns were conducted in 2014 with the purpose of investigating the overall litter problem in the Leie River. In that campaigns floating litter (> 5 mm) was investigated. The scope of the study was extended to cover the investigation of microplastics as well (Craenenbroeck et al., 2014).

In the context of a study by the University of Bayreuth (Germany) the abundance of microplastics and mesoplastics in the Rhine River, four Rhine tributaries (Rivers Ruhr, Lippe, Sieg, Wupper and Emscher) and the Weser River were investigated. At three sampling points along the Rhine, the total plastic amounts ranged from 0.928 to 4.45 particles/m^3. The highest concentration was measured near Düsseldorf in the Rhine-Ruhr metropolitan area. Among the Rhine tributaries, the highest concentration was measured in the Emscher River close to where it joins the Rhine. The plastics concentration of 15.7 particles/m^3 (total plastic amount) was expected due to high wastewater content of the sample. Measurements up-stream and down-stream a wastewater treatment plant (WWTP) at the Ruhr River near Duisburg showed a considerably higher concentration of total plastics at down-stream than at up-stream sampling site (up-stream site: 4.81 particles/m^3, down-stream site: 166 particles/m^3). Along the Weser River the total amount of plastic particles was 0.487 particles/m^3. The sampled particles were identified by means of ATR-FTIR spectroscopy. In many samples the common polymers polyethylenes, polypropylene und polystyrene were detected (Laforsch, 2015).

Transboundary monitoring studies including Netherlands freshwaters:

Mani et al., (2015) evaluated the abundance and composition of microplastics (0.3 mm - 5 mm) in the surface layer of the Rhine River along an 820 km stretch between Basel and Rotterdam. The mean concentration from 11 sampling sites located in Switzerland, France, Germany and the

Nether-lands was 0.893 particles/m^2. Microplastic concentrations reached a peak in the Rhine-Ruhr metropolitan area, with a maximal concentration of 3.9 particles/m^2 at Rees. In fact, sampling points at Rees and Duisburg accounted for >66 % of all particles recovered during the entire campaign. The microplastics consisted of opaque spherules (45.2%), fragments (37.5 %), transparent spherules (13.2 %), fibers (2.5 %) and others (1.1 %). Polystyrene (29.7 %) and polypropylene (16.9 %) were the most dominant polymers. The profile study along the river demon-stated considerable pollution of the Rhine River.

During research of a master thesis, Urgert, (2015) investigated the abundance and composition of microplastics of the European rivers Meuse and Rhine in 2014. Over a period of less than 6 months weekly samples were taken at each of the three monitoring locations (Netherlands: Eijsden, Lobith; Germany: Bimmen) and a single sample at one additional sampling site (Germany: Bad Honnef). Mean concentrations of microplastics (size range: 0.125 - 5 mm) in the water column were higher in the Rhine River (0.56 mg/m^3, 56 particles/m^3) than in Meuse River (0.14 mg/m^3, 9.7 particles/m^3). Visually identified microplastic particles were classified into the following groups: films, miscellaneous microplastics, white and transparent spherules and scrubs. Polymers were identified by means of Raman- and FT-IR spectroscopy. In the Meuse River, PE, comprising films, miscellaneous microplastics and scrubs were predominantly identified whereas in the Rhine River the predominant polymers were PE and polystyrene.

The comprehensive study by Hohenblum et al., (2015b) included preliminary monitoring of plastics in the Dalälven River (Älvkarleby, Sweden), Rhine River (Rotterdam, Netherlands), Po River (Ferra-ra, Italy) and Danube River (Galati, Romania) in addition to an assessment of riverine plastic litter inputs into the marine environment, the abundance of both micro- and macroplastics was evaluated using various sampling methods simultaneously (man-ta net, waste free water (WFW) sampler and pump-manta net method) in order to consider the size range of 0.3 - 25 mm and to test the feasibility of the monitoring approach. The pump method was applied in the Dalålven River out of necessity, because of local limitations in setting up other sampling equipment. To enable comparisons, the manta-pump method was also applied at the sampling point of the Po River. Concentrations at the sampling point of the Rhine River were measured two times (Hohenblum et al., 2015b). The results of the study are summarised in Table 2.6.

In the context of two studies (Brandsma et al., 2013, 2015) investigated SPM in the Rivers Meuse (Netherlands) and Rhine (Germany and Netherlands) as well as Lake Ijssel (Netherlands) in regard to abundance of microplastics in the size categories 1 - 300 μm and 300 - 5000 μm. In 2013, mean microplastic concentrations of 1400 particles/kg dw (Meuse River) and 1700 – 4900 particles/kg dw (Rhine River, two sampling points) were derived. Whereas, 1800-6880 particles/kg dw (Meuse River, 3 sampling points), 990 particles/kg dw (Rhine River, single sample) and 2000 particles/kg dw (Lake Ijssel, single sample) were foundin 2015. The majority of microplastics detected in both sampling years were in the size category < 0.3 mm, with fibres and spheres rather than foils as the most frequently detected shapes.

Table 2.6: Comparison of plastic concentrations in the rivers Dalålven, Rhine, Po and Danube by using different sampling methods (Hohenblum et al., 2016)

River	Manta net (micro sized particles < 5 µm)*		Waste free water sampler (meso sized particles > 5 µm)*	
	Manta trawl	Pump manta net	Surface	Suspension
Dalålven		4.5 p/m^3 2 mg/m^3		
Rhine I (measurement1)	1.77 p/m^2 2.45 mg/m^2		0.01 p/m^2 15.86 mg/m^2 0.05 p/$m^3$79 mg/m^3	0.01 p/m^3 0.24 mg/m^3
Rhine II (measurement2)	0.31 p/ m^2 0.04 mg/ m^2		0.01 p/ m^2 1.54 mg/ m^2 0.04 p/ m^3 7.7 mg/ m^3	0.002 p/ m^3 0.8 mg/ m^3
Po	2.04 p/ m^2 0.78 mg/ m^2	20.3 p/ m^3 0.5 mg/ m^3	0.01 p/ m^2 0.75 mg/ m^2 0.03 p/ m^3 3.8 mg/ m^3	0.03 p/ m^3 2.5 mg/ m^3
Danube	1.06 p/ m^2 0.12 mg/ m^2		0.07 p/ m^2 7.55 mg/ m^2 0.37 p/ $m^3$38 mg/ m^3	0.24 p/ m^3 5.3 mg/ m^3

2.18 Standards and definitions

An important prerequisite of obtaining consistent monitoring results is broadly accepted definitions. According to the ISO, plastic is “a material which contains as an essential ingredient a high polymer and which, at some stage in its processing into finished products, can be shaped by flow”. The chemical legislation defines a polymer material as a substance in which >50% of its weight consists of polymer. Size classes for the plastic particles found in the environment have been suggested by the EU Working Group on Good Environmental Status under the Marine Strategy Framework Directive and by the International Union of Pure and Applied

Chemistry, among others. While the upper limits of macro-, meso- and microplastic particle sizes are broadly accepted, the borderline be-tween micro- and nanoplastics remains poorly defined. In most studies the lower size limit was set by the limit of detection. At the conference, it was agreed that, for environmental research a clear identification of a microplastic particle requires an assessment not only of its size but also of its chemical composition, shape and physico-chemical parameters. Furthermore, the methods used in micro-plastic identification should be harmonized, as the variability in sampling, sample treatment, polymer identification and data reporting (e.g. particle per km^2, particle per m^3, mass per km^2, mass per m^3) makes it difficult to compare the results of the different studies, (Beate Bänsch-Baltruschat., 2017).

2.19 Literature review

When wastewater containing plastics from domestic, commercial, and industrial sources passes through wastewater treatment systems (WWTSs), most of the plastics are removed prior to discharge to the aquatic environment.

The research interest in plastic contamination has increased in recentyears. A quick search in the Aquatic Sciences and Fisheries Database (Ferraz et al., 2020) using the term “micro plastic” generates 804 hits for theperiod between 2003 and 2020. A total of 713 (88%) of all articles werepublished in the last five yearsbetween 2015 and 2020. These publications address the quantification of particles suspended in the water column (Faure, F et al., 2012), in the sediment (Castañeda, R.A et al., 2014), in invertebrates (Brennecke, D., et al., 2015), in stomach contents of fish (Silva-Cavalcanti, J.S et al., 2017), and in aquatic bacteria.

Based on a review of several published studies, (Sun et al., 2019) reported significant reductions in microplastic concentrations when comparing influent and effluent in various WWTSs: concentrations ranged from 1 to 10044 particles/L for influent and from to 0 to 447 particles/L for effluent. While large variations in microplastic concentrations can be observed between WWTSs, this may be due to differences in sample collection and analysis methods, as there are currently no standardized methods for the detection and quantification of microplastics in water. Other factors, such as catchment size, population served, wastewater source (residential, commercial, or industrial), and treatment technology, may also contribute to variations in influent and effluent concentrations and treatment efficiencies estimated that 50% to 98% of microplastics can be removed during primary treatment, which involves skimming processes and settling stages, with larger particles being preferentially removed. Secondary treatment, which typically involves biological treatment to remove organic compounds, can increase microplastic removal to approximately 86% to 99.8% of microplastics. The addition of tertiary treatment can lead to the removal of 98% to 99.8% of microplastics, but removal efficiency is dependent on the type of treatment technology used.

According to available data on the microplastic removal efficiencies of wastewater treatment systems (WWTSs), standard WWTSs using primary and secondary treatment processes can effectively remove most microplastics from the effluent before it is released to receiving waters (WHO, 2019).

A study recently conducted by Lobelle and Cunliffe, (2011) tested polyethylene food bags, submerged in Queen Anne’s Battery (Plymouth, UK) to gain more information about how early microbial biofilm formation occurs. They found that after two weeks, the plastic began to float below the water’s surface, and by three weeks, the plastic started sinking down below the surface, showing signs of neutral buoyancy.

Castañeda et al., (2014) have been identified microplastic pollution has only recently been documented in freshwater environments, almost exclusively in surface waters. They reported microplastics (polyethylene microbeads, 0.40– 2.16 mm diameter) in the sediments of the St. Lawrence River. They sampled 10 freshwater sites along a 320 km section from Lake St. Francis to Québec City by passing sediment collected from a benthic grab through a 500 μm sieve. Median and mean (±1 SE) densities across sites were 52 microbeads·m−2 and 13 832 (±13 677) microbeads·m−2, respectively. The highest site density was 1.4 × 105 microbeads·m−2 (or 103 microbeads· L−1), which is similar in magnitude to microplastic concentrations found in the world's most contaminated marine sediments. Mean diameter of microbeads was smaller at sites receiving municipal or industrial effluent (0.70 ± 0.01 mm) than at non-effluent sites (0.98 ± 0.01 mm), perhaps suggesting differential origins. Given the prevalence and locally high densities of microplastics in St. Lawrence River sediments, their ingestion by benthivorous fishes and macroinvertebrates warrants investigation.

Murphy et al., (2016) found that the large volumes of effluent water leaving a WWTS, even a small fraction of microplastics remaining in the effluent water after treatment can translate into high absolute numbers of particles being released to the environment. Effluent discharges have therefore been identified as an important pathway for the entry of microplastics into freshwater sources.

Talvitie et al., (2017); Lares et al., (2018) showed that advanced technologies such as rapid-sand filters, membrane bioreactors, and dissolved-air flotation can remove 95% to 99.9% of microplastics greater than 20 µm. Mintenig et al., (2019) observed complete removal of microplastics >500 µm and 95% of microplastics <500 µm using tertiary filtration.

In Brazil, studies related to MPs follow the global pattern with most publications on coastal andmarine ecosystems (Castro; da Silva, M.L et al., 2018). Gomes, (1973) conducted the first study about plastic particles in the environment in Brazil. He counted a mean of 31.8 pellets m^{-2} on beaches of the northern coast of Rio Grande do Sul. Other studies in Brazilfound MPs on the beaches of Guanabara Bay (Rio de Janeiro) where the concentrations ranged from 12 to 1300 p m^{-2} (De Carvalho et al., 2016). Surface water concentrations ranged from 0.0014 to 0.0213 p. L^{-1} at the same location (Olivatto et al., 2019). Out of a sample of 2332 fish from a tropical Brazilian estuary 9% of the examined individuals contained a mean particle count of 1.06 ± 0.3, independently of size or trophic guild (Vendel, Bessa et al., 2017). In a tropical Brazilian river, Silva-Cavalcanti et al., (2017) examined 48 individuals Hoplosternum littorale, a siluriformbottom dwelling species. They quantified a total of 176 MPs in the guts of40 individuals (min. = 1 and max. = 24 per fish), 46.6% of the particles were fibers. Another study by Andrade et al., 2019), examining the stomach contents of 172 specimens from 16 serrasalmid species in the Xingu River basin, found 96 MPs in 46 specimens. The authors analyzed species of three trophic guilds (omnivorous, herbivorous and carnivorous fish), but did not find significant differences. The work was the first recordof microplastics in fish belonging to the Amazon basin.

Carney et al., (2018), counted the number of microfibres released from different types of fabric under different laundering conditions. Both studies found that the use of a detergent increases the number of fibres released during washing. Powdered detergents, which often contain insoluble compounds that are able to create friction with the fabric, enablean even greater number of fibres to be released.

Biber et al., (2019) studied the deterioration of different plastics in air and seawater. Macro-sized pieces of PE, PS, PET, and a commercial material marketed as biodegradable plastic were exposed to environmental conditions in air and water. All materials deteriorated more slowly in seawater than in air, likely due to reduced exposure to light and thus reduced photooxidation in seawater. They are found that PS showed the most rapid deterioration and is likely to break down into microplastics faster than the other materials evaluated, but that all materials tested did deteriorate to microplastics. Given the requirements for breakdown, it is expected that plastic items likely remain in seawater and that the formation of microplastics would occur in areas where plastic pollution is exposed to oxygen and UV radiation, such as intertidal habitats and at the water surface.

The recent review by Koelmans et al., (2019) listed 50 studies with 55 records of MP concentrations in tap and bottled drinking water, as well as in ground, surface, and wastewater. All studies reported the presence of MP in the samples with concentrations covering a 10 billion-fold magnitude (10^{-5} p.L^{-1} – 10^{5} p.L^{-1}). Given the omnipresence of MP on a global scale, the potential health hazards caused by artificial polymers, and the importance of the Sinos River as a drinking water resource for approximately 1.3 million inhabitants, this study analyzed MP concentrations in raw and treated raw water.

Kadhum et al., (2020), were studies microplastics abundance, distribution, and polymer types in surface sediment from Euphrates River were reported for the first time. Microplastics were found in all sediment sites along the river, and abundance ranging from 29.6 to 120.0 particles/kg dry, with an average 86.7 particles/kg dry. Fiber particles were the most predominant form (57.1%), followed by films (29.6.2%) and fragments (13.1%). The polymers identified using Fourier Transform Infrared Spectroscopy (FTIR) which was dominantly polyethylene (44.1%), polyethylene terephthalate (35.0%) and nylon (20.2%). It is clear that the microplastics levels in sediment from Euphrates river were polluted and these data will be useful for manage and rehabilitation the Iraqi rivers to reduce possible microplastics pollution.

2.20 Rationale for this Study

Plastic pollution is a globally recognized problem, with some regions acknowledged as being more heavily impacted than others. It has often been stated that countries in Asia like Iraq are the most significant polluters globally, in terms of plastic pollution inputs to the environment. The reasons for these inputs are numerous, but include factors such as insufficient waste management infrastructure, lack of education around waste management, economic reasons leading to greater use of single use plastics (such as single-portion sachets) and global imports. Additionally, population growth and increasing urbanization are leading to increased volumes of plastics utilized; where waste management is insufficient or ineffective for controlling the large volumes of plastics used and discarded, these end up becoming distributed on land, and within rivers. Here they may accumulate or be transported to the streams, depending on local and seasonal environmental conditions. It has been suggested that even with ambitious efforts to reduce and manage plastic waste, millions of tones will continue to enter the environment (Lyons, et al., 2020).

The importance of this study lies in:-

- It is the first study conducted on evaluating the concentration abundance, distribution, and composition of microplastics in drinking water of some districts of Al-Hilla city/ Iraq.
- On the other hand, it is concentrated on the assessment of microplastics in raw water of AL-Hilla River, identifying the sources of this pollution, and showing its impact on the population of the city of AL-Hilla, which depends entirely on the river for all its water purposes, No studies to date have addressed the presence of microplastics along the Al-Hilla River, either those flushed downstream, those deposited in the river's sediments or those carried into the estuary.

- Enhanced aggregation can cause microplastic to become negatively buoyant and sink into sediments, where they may accumulate owing to very slow rates of degradation.

Chapter Three

Experimental Work

CHAPTER THREE

EXPERIMENTAL WORK

3.1 Introduction

Microplastics are becoming an increasingly dominant threat to water. The ubiquity of microplastics is one of the main problems; small plastic particles have been found from the water surface, the water column, and floor sediments, or even ingested by organisms. This study focuses on evaluating the concentration of microplastics (plastic particles <5 mm) in raw and drinking water, of Al-Hilla River/ Babylon Governorate/ Iraq. Al-Hilla River is the main source for drinking purposes in the governorate. It is also a tourist and recreational area for the residents of the governorate, and therefore it is also considered a cause of litter in the river. This chapter, identified the study areas and also analyzed the microplastics found in water samples.

Water samples were collected from Al-Hilla River and water supply network of some district of Al-Hilla city. Some parameters of raw and drinking water were tested. The analyzing and testing locations of microplastic for raw and drinking water samples were conducted as shown in table 3.1.

Table 3.1: Analyzing and testing locations of microplastic for raw and drinking water samples.

Objective	Device	Laboratory
Raw Water	The analysis of microplastics for raw water	AL-Mustaqbal University / Iraq
Raw Water &Drinking Water	Micro-Raman Spectrometer (MRS)	Tehran University / Iran
Raw Water	Scanning Electrons Microscope (SEM)	Tehran University / Iran
Drinking Water	Scanning Electrons Microscope (SEM)	Department of Polymer Engineering and Petrochemical Industries/ Faculty of Materials engineering/ University of Babylon /Iraq

Raw Water	Fourier Infrared spectrometer (FTIR)	Department of Polymer Engineering and Petrochemical Industries/ Faculty of Materials engineering/ University of Babylon/ Iraq
Drinking Water	Fourier Infrared spectrometer (FTIR)	Tehran University / Iran

3.2. Description of the study site

This study was conducted from August to December of 2022 using the garbage trap method, with monthly sampling. 5 sample were taken with 30 days intervals between each. The sampling sites consisted of five stations for raw water and five stations for drinking water.

Al-Hilla city is densely populated and industrialized. Its length is about 101 km (https://ar.wikipedia.org/wiki), and its borders start from the northern border of the governorate to the governorate of Al-Diwaniyah (Fig. 3.1). Al-Hilla River provides drinking water for 2.6 x 10^6 capita. During September 2022, five of the most important locations along 101 km freshwater section of Al-Hilla River were chosen to take samples of raw water in order to cover different geographical areas. These locations had a high population density, as Sadat Al-Hindiya, Al-Athar area were a tourist area in addition to being an agricultural area and feeding the surrounding area with water, Al-Sayeyd Al-Farisy area which is an important, as it is located in the center of the city of Al-Hilla, and Al- Hashimiya area was also chosen because it is considered an agricultural and rural area as a source of financing for the residents of the region.

The locations for drinking water, samples was chosen one in Al-Bakarly area, two points in Al-Khusrawiyah neighborhood, and two points in Al-Tayyara neighborhood. For all samples, preventive measures were taken to avoid contamination: sampling team should wear clothing made from natural fibers rather than synthetic clothing owing in to the containers, air exposure to samples limited as much as possible, clean and monitor all tools and containers with a stereo tax before using them. Some plastic fragments found after observation that resemble plastic in the containers were discarded to avoid any contamination through scraping of the containers.

3.3 Stations selection

The selected stations were chosen due to the lack of current studies, in addition, Al-Hilla River contains urban, suburban, and rural sub-watersheds. This study was conducted for raw water (five stations) as shown in Fig. 3.1 and drinking water (five stations) as shown in Fig. 3.2.

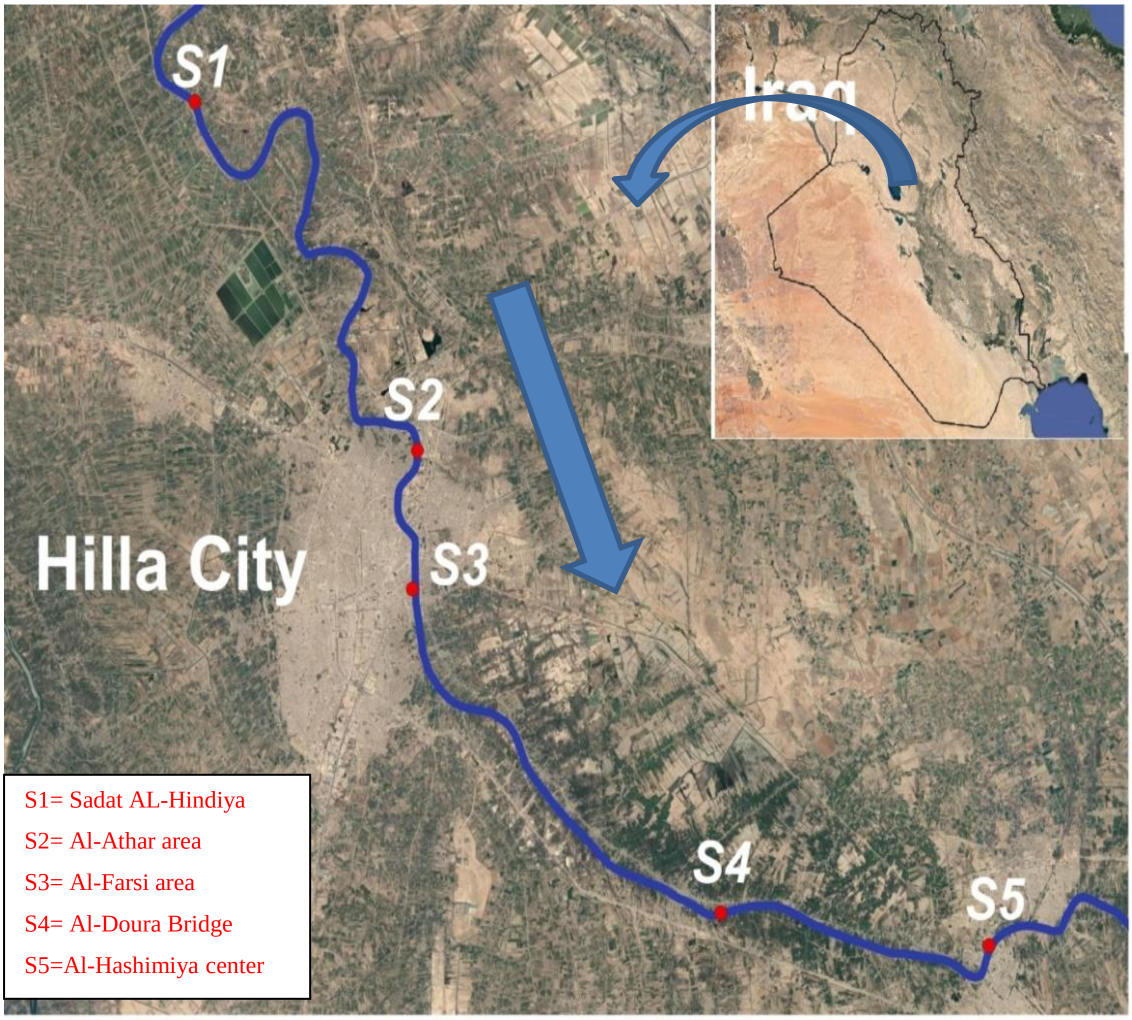


Fig. 3.1: Map of Al-Hilla River illustration sampling stations.

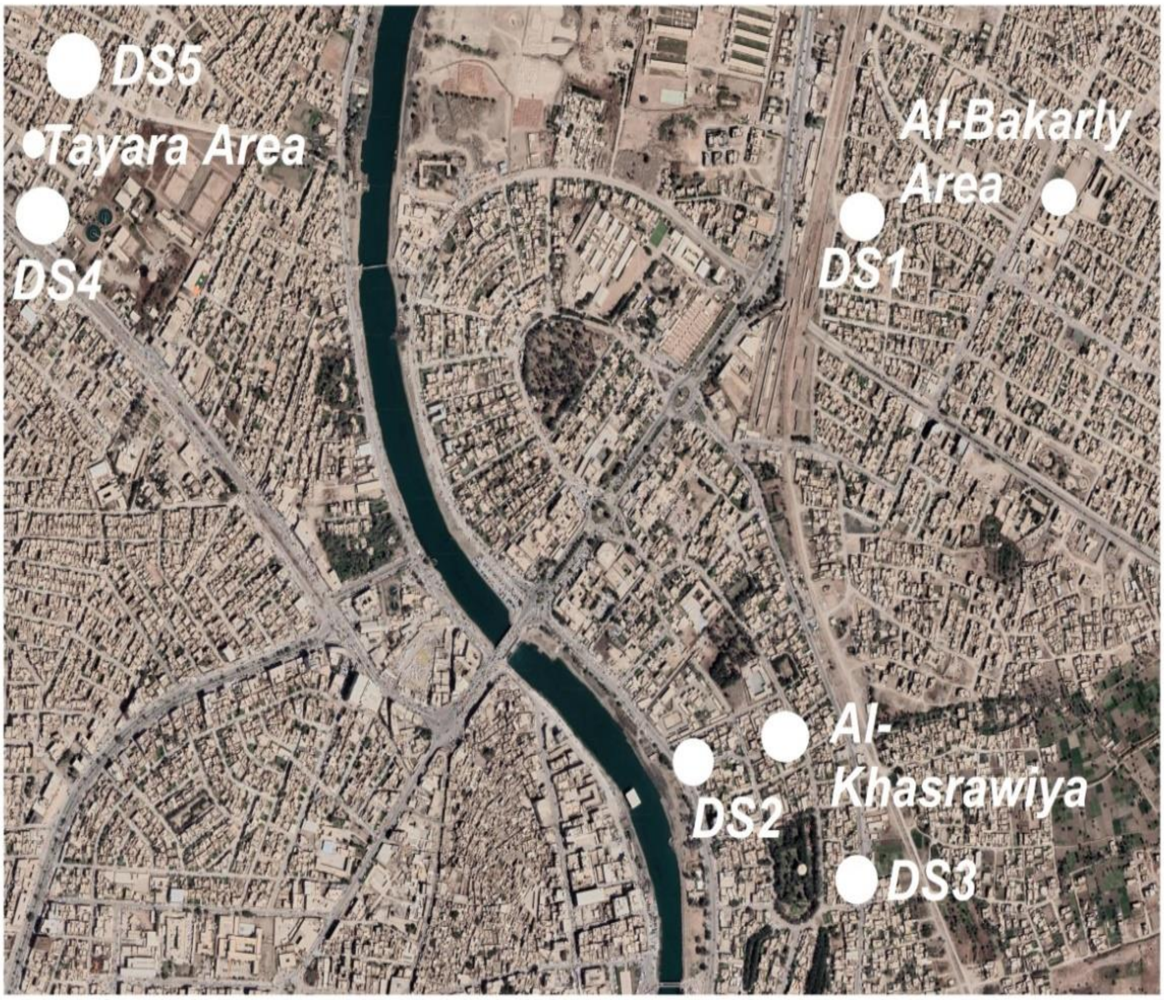


Fig. 3.2: Map of Al-Hilla city illustration drinking water sampling sites.

3.3.1 Stations of raw water

These locations are coded:-

- Station 1 (S1), Sadat AL-Hindiya: Is located in the center of Iraq within the Middle Euphrates region, south of Baghdad in the province of Babylon and administratively affiliated to the Musayyab district. The reason for choosing this site is due to its importance and responsibility for providing the residents of the region with water.
- Station 2 (S2), Al-Athar area: It is considered one of the tourist areas in Babylon Governorate due to the presence of antiquities that are a

reason to attract tourists to it. Livestock are raised by the inhabitants of the area along the river, and therefore the water is affected by it.

- Station 3 (S3), Al-Farsi area: This area is located in the province of Babylon. Because of the unique location of this area in the center of the city, near the market and the waste that market owners throw in the river. Therefore, this is the main reason for the pollution of this area with plastic bags and waste dumped in the river.
- Station 4 (S4), Al-Doura Bridge in the Al-Hashimiya District. The area in general has picturesque nature, agricultural lands, and orchards, which is considered rural and agricultural, so environmental awareness is low and there is no control over plastic materials.
- Station 5 (S5), center of Al-Hashimiya: It is located near water treatment plant, as it is considered the main and only source of drinking water for the residential area. This area is generally characterized by picturesque nature, agricultural lands, and orchards, and it is considered a rural area, which affects cultural awareness in terms of throwing plastic waste into the water.

3.3.2 Stations of drinking water

These locations are coded:-

- Station 1 (DS1): Al-Bakarly area: This area was approved in the study because of its proximity to Al- Hilla River and its population density. It is one of the important areas in Babylon Governorate, as it contains markets, schools, and the police station, in addition to the railway station, which made it one of the vital areas of the governorate.
- Station 2 and station 3 (DS2 and DS3): Al-Khusrawiya area: This region has been selected in the study Because of its proximity to Al-Hilla River and its population density. It is considered one of the urban

areas in Babylon Governorate, as it contains markets, schools, and hospitals, in addition to the health center.

- Station 4 and station 5 (DS4 and DS5), Al-Tayyara area: This area was adopted in the study due to its close proximity to Al-Hilla River and its population density. It is considered one of the vital areas of Babylon Governorate, as it contains the Al-Tayyara district water treatment plant, as well as markets and schools. It is considered a link area for Babylon Governorate with the neighboring governorates.

3.4 Water sampling

Microplastics are distributed in the water column dependent on their properties, such as density, shape, size, adsorption of chemicals and biofouling, and on environmental conditions such as water density, wind, currents and waves. Thus, quantity and quality of microplastics recovered are highly dependent on sampling location and depth. Sampling and processing methods are similar for both fresh and tap water samples. Differences can be found in the distribution of microplastics in each system, influenced by environmental characteristics, such as hydrodynamic profiles, as well as density.

The sampling was conducted across sites (five raw water sampling sites, and five drinking water sampling sites) and seasons (rainy and dry season). The tools used in this study were nets, roller meters, digital scales (accuracy of 1 gram), trash bags, camera, GPS (global positioning system) and stationery. The garbage trap method was used to collect macroplastic waste in the water river. The net as a garbage trap was placed vertically across the body water for one hour. All plastic wastes that entered the trap were collected and cleaned, then put into a trash bag and taken to the laboratory for further analysis. Plastic waste taken from the field is then dried (under the sun), grouped by type and weighed using digital scales.

3.5 Apparatus and materials

- ***Apparatus***

1. Stainless steel sieves.
2. An oven (90 ^{o}C) used for sample drying.
3. Density separator, which is assembled by using a glass funnel (122mm in diameter), fitted with a 50mm segment of latex tubing on the bottom of the stem and a pinch clamp is attached to control liquid flow from the funnel.

- ***Materials***

All chemical reagents used in this study were given in Table 3.2.

Table 3.2: Chemical reagent used in this study.

Chemical materials	Chemical Formula
Sulfuric acid	H_2SO_4
Iron(II) sulfate	$FeSO_4$
Hydrogen peroxide	H_2O_2

3.6 The analysis of microplastics of water samples

The methods used for the analyses of plastic debris as suspended solids in water samples collected by a surface net were done according to Masura et al., (2015). Plastics include hard plastics, soft plastics (e.g. foams), films, line, fibers, and sheets. The method involves the filtration of solids that obtained from five stations (S1, S2, S3, S4 and S5) through a 0.335 mm surface sampling net (e.g. a manta net for surface water tows) through 5.6mm and/or 0.3mm sieves to isolate the solid material of the appropriate size. The sieved material is dried to determine the solids mass in the sample. The solids are subjected to wet peroxide oxidation in the presence of a $FeSo_4$ catalyst to digest organic matter, as shown in Figs. 3.3 and 3.4. The plastic debris remains unaltered. The wet peroxide oxidation mixture is subjected to density separation in NaCl to isolate the plastic debris through flotation. The floating solids are separated from the denser undigested mineral components using a density separator. The floating plastic debris is collected in the density separator using a custom 0.3mm filter, air-dried, and plastic material is removed and weighed to determine the microplastics concentration.

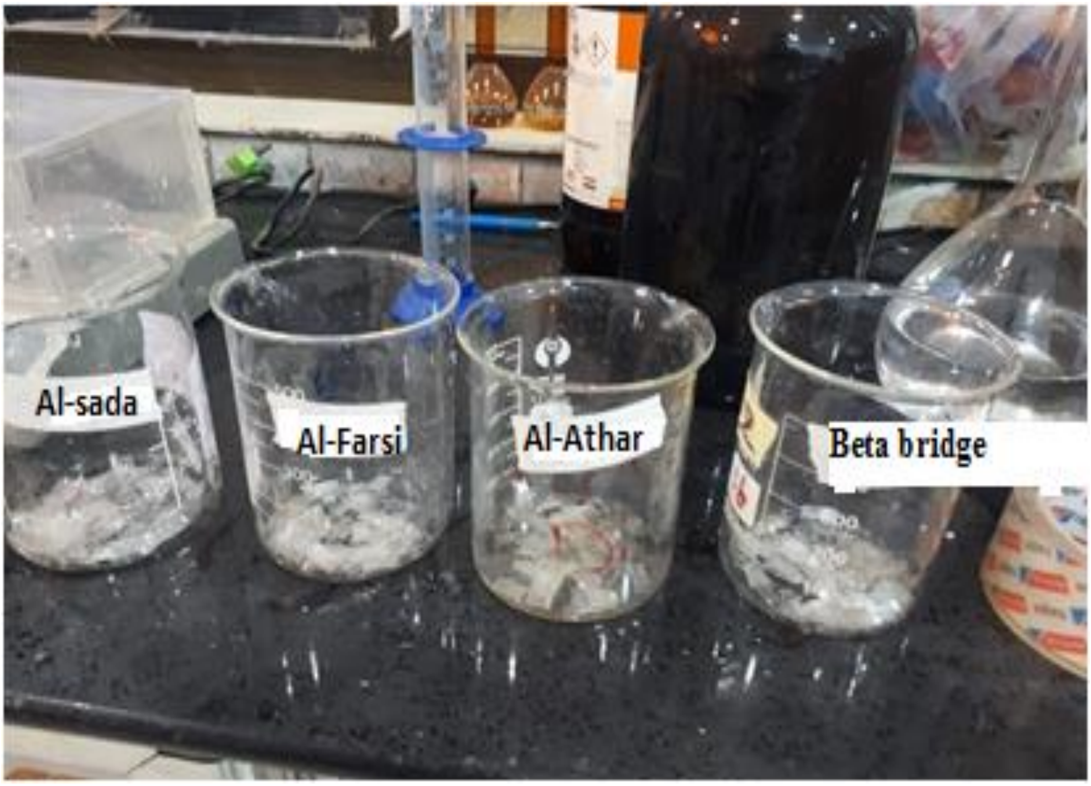


Fig. 3.3: Water sample before processing.

Fig. 3.4: Sieving and rinsing field samples.

The plastic debris analyzed by this method is microplastic and ranges in size from 5 mm to 0.3 mm. Microplastic debris is operationally defined by this method as any solid material in the appropriate size range that is resistant to wet peroxide oxidation.

3.7 Testing Procedure

3.7.1 Wet sieving

Wet sieving, is a particle size gradation technique involving the use of a liquid substance. The process of sieving water samples is commonly applied in microplastic research. This method involves applying water to the test sample and allowing fine particles to pass through the sieve while also breaking down agglomerates. The sample is then dried and assessed. Wet sieving is also used to prepare the granular sample for further particle size analysis (e.g., vibration sieving). Like other sieving methods, wet sieving is used to separate or measure particles by their size (Masura et al., 2015).

- Pour the sample through a stacked arrangement of 5.6mm and 0.3mm stainless steel mesh sieves.
- Rinse sample with squirt bottle filled with distilled water to transfer all residual solids to the sieves. This also removes salts from the field sample. Repeat as necessary.
- Rinse sieves thoroughly using distilled water. Ensure all material has been well washed, drained, and sorted.
- Discard or archive material retained on 5mm sieve.

3.7.2 Transfer sieved solids

The sieve physically captures the microplastics and allows the water to be lost from the sample. This method is well documented and has been used in numerous studies to separate microplastics from water the size of the mesh used in the sieve depends upon the size of the microplastics that are desired to be collected. It is important to note that the mesh size must not exceed 5 mm, otherwise the size of the pieces of plastic collected would be outside the size-range of microplastics.

- Weigh a clean and dry 500mL beaker.
- Transfer solids collected in the 0.3mm sieve into the tared beaker using a spatula and minimal rinsing with a squirt bottle containing distilled water.
- Ensure all solids are transferred into the beaker.
- Place beaker in 90^{o}C drying oven for 24 hours or longer to sample dryness.

The mass of total solids was determine by using the following equation (3.1), (Masura et al., 2015).

$$C = b - a \tag{3.1}$$

Where:

a= mass of beaker (mg).
b= mass of the beaker with dried solids (mg).
C= mass of total solids (mg).

- ***Wet Peroxide Oxidation (WPO)***
  - Add 20 mL of aqueous 0.05 M $FeSo_4$ solution to the beaker containing the 0.3 mm size fraction of collected solids.
  - Add 20 mL of 30% hydrogen peroxide.
  - Let mixture stand on lab bench at room temperature for five minutes prior to proceeding to the next step.
  - Add a stir bar to the beaker and cover with a watch glass.
  - Heat to 75 ^{o}C on a hotplate.
  - As soon as gas bubbles are observed at the surface, remove the beaker from the hotplate and place it in the fume hood until boiling subsides. If reaction appears to have the potential to overflow the beaker, add distilled water to slow the reaction.
  - Heat to 75 ^{o}C for an additional 30 minutes.
  - If natural organic material is visible, add another 20 mL of 30% hydrogen peroxide.
  - Repeat until no natural organic material is visible.
  - Add about 6 g of salt (NaCl) per 20 mL of sample to increase the density of the aqueous solution (about 5 M NaCl).
  - Heat mixture to 75 ^{o}C until the salt dissolves.

3.7 Density separation

Transfer the WPO solution from section 3.6.4 to the density separator as shown in Fig. 3.5.

- Rinse the WPO beaker with distilled water to transfer all remaining solids to the density separator.
- Cover loosely with aluminum foil.
- Allow solids to settle overnight.
- Visually inspect settled solids for any microplastics. If any are present, drain the settled solids from the separator and remove microplastics using forceps. Archive or discard.
- Drain settled solids from the separator and discard.
- Collect floating solids in a clean 0.3mm custom sieve, as shown in Fig. 3.6.
- Rinse the density separator several times with distilled water to transfer all solids to the 0.3mm sieve.
- Allow the sieve to air dry while loosely covered with aluminum foil for 24 hours Fig. 3.7.

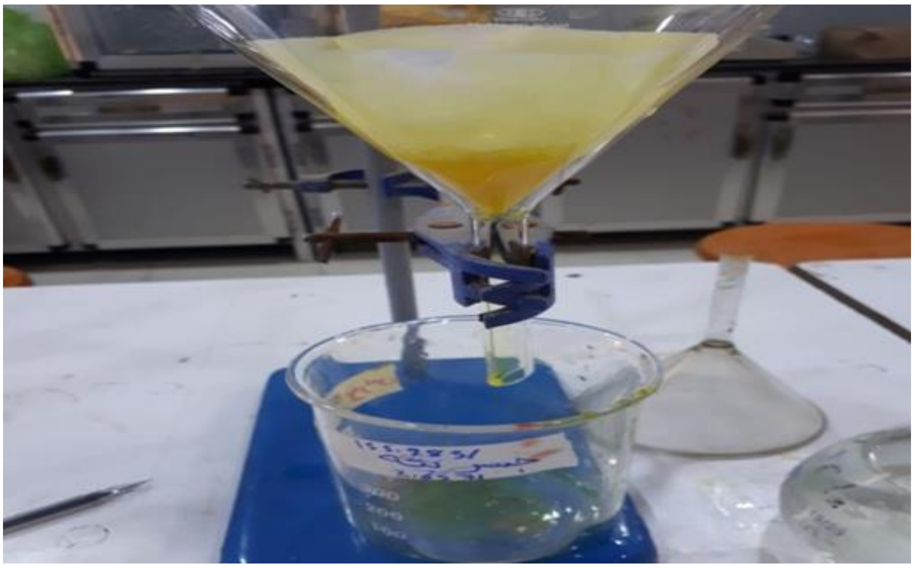


Fig. 3.5: A sample is depicted in a glass funnel to separate plastic.

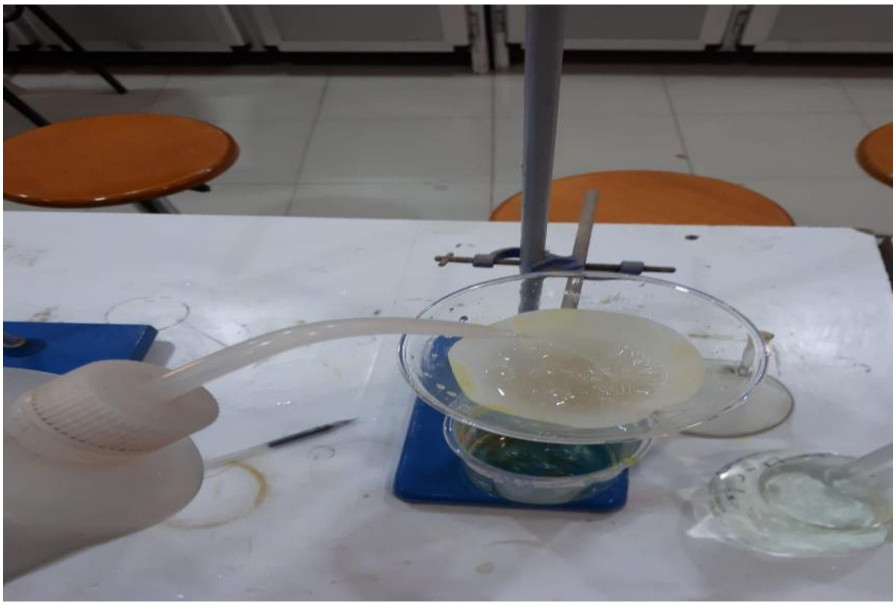


Fig. 3.6: Floating material is drained into a custom sieve.

Fig. 3.7: A prepared sample is ready for microscope examination.

3.8 Gravimetric analysis

Gravimetric analysis is one of the most accurate and precise methods of macro-quantitative analysis. In this process the analytic is selectively converted to an insoluble form.

$$F = e - d \tag{3.2}$$

Where:-

d= mass of the tared vial, (mg).

e= weigh the mass of the vial and microplastics, (mg).

F= mass of microplastics, (mg).

3.9 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) can provide high-resolution images of the microplastic. SEM imaging is performed in the laboratory of University of Tehran/Iran (Quanta 450 Environmental Scanning Electron Microscope (FE-ESEM)) with resolution ($5*10^{-6}$ mm) and magnification (0.001 mm and 200 nm) for this purpose.

3.10 Fourier Infrared Spectrometer (FTIR)

A Fourier Transform Infrared Spectrometer collects and processes infrared wavelength absorption or transmission into spectra. These spectra are generated when a molecule converts infrared radiation into molecular vibrations. These vibrational motions create bands in the spectrum that occur at certain wavelengths (cm^{-1}). Each wavelength then depends on a number of other problems that can be used to help determine what types of bonds are present in the test sample.

3.11 The Micro-Raman spectrometer (MRS)

Micro-Raman spectroscopy (MRS) is a non-destructive analytical method based on the effect of inelastic light scattering on molecules. MRS offers the advantage of being insensitive to water, which allows the analysis of MP in aqueous and wet samples. The spectral range for the Raman analysis of microplastics has to cover all spectral features of (synthetic) polymers from 50 to 4000 cm^{-1} (best practice, or from 200 to 2000 cm^{-1} as minimum requirement).

Chapter Four

RESULTS AND DISCUSSION

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Introduction

In this chapter, five stations were identified for raw and five for drinking water. Samples were taken from these stations representing the raw water of Al-Hilla River, such as: Sadat Al-Hindiya, Al-Athar area, Al-Sayyid Al-Farsi area, Al-Dora Bridge in the area and Al-Hashimiyya Center. Five samples were taken from water distribution network, such as Al- Bakarly arly area (one sample), Al-Khusrawiya (two samples) and Al-Tayyara area (two samples).

In order to detect the microplastic particles in the raw water and drinking water samples using a series of testing techniques such as: Infrared Fourier Transform Spectrometry, Electron Microscopy and Micro-Raman Spectrometry. The microplastic in raw and drinking water samples were tested at Tahran University laboratories, and Al- Mustakbel University laboratories.

4.2 The analysis of water samples

Raw water samples were taken from five locations covered Al-Hilla River, where these areas were Sadat Al-Hindiya, Al-Athar area, Al-Sayyid Al-Farsi area, Al-Dora Bridge and Al-Hashimiyya area. The main sources of microplastics in the river coming from urban, storm water, runoff, sewer overflows, inadequate waste disposal and management industrial activities and illegal dumping.

Table 4.1 shows the mass of total solids after washing and mass of microplastic for the samples covering the river line during the study time (Aug. – Dec.). The most abundant area of microplastic materials was, Al-Dora Bridge, with a wet mass of 2.811 mg, followed by the Sadat Al-

Hindiya with 2.771 mg, followed by Al-Sayyid Al-Farsi area with 2.763 gm, then Al-Athar area with a 2.657 gm, and the last of which is Al-Hashimiyya area with 2.638. The reason for the difference in the percentage for each region can be attributed to the increase in water pollution, the lack of cultural awareness, and the lack of permanent cleaning of the river water.

Table 4.1: Mass of total solids and mass of microplastic.

Station	Wet Mass of total solid after washing, (gm)	Wet density, kg/m^3, *10^{-3}	Dry Mass of microplastic (gm)	Dry density, kg/m^3, *10^{-3}
S1	2.771	2.771	2.073	2.073
S2	2.657	2.657	2.646	2.646
S3	2.763	2.763	2.115	2.115
S4	2.811	2.811	2.533	2.533
S5	2.638	2.638	2.413	2.413

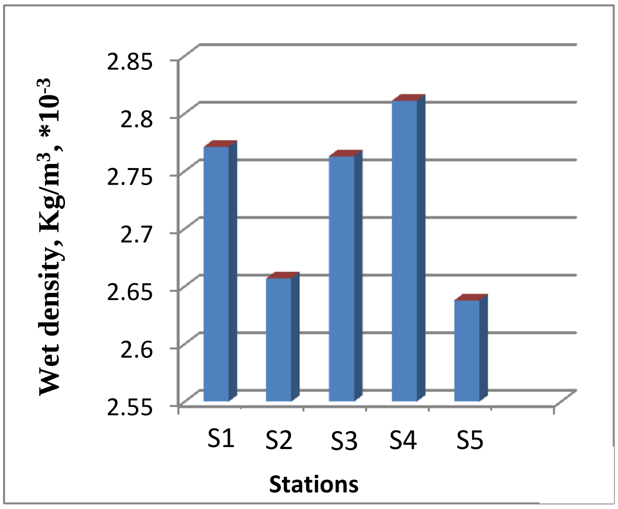


Fig. 4.1: Variation of wet density for raw water samples in five stations.

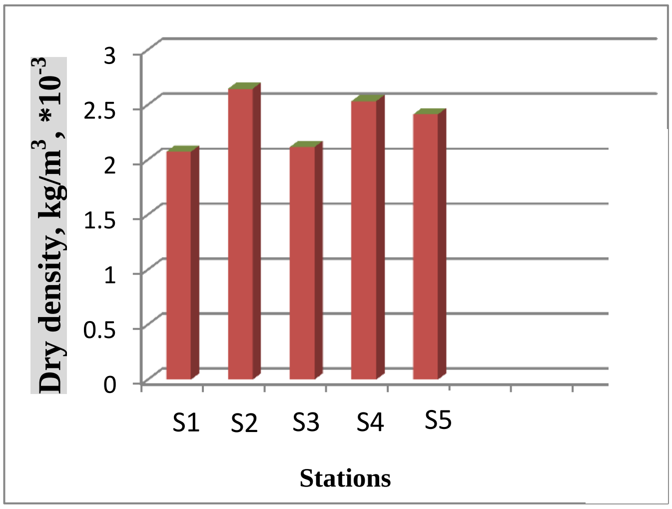


Fig. 4.2: Variation of dry density for raw water samples in five stations.

4.3. Microplastics density

During the study, a total of five macroplastic categories were collected with a lowest wet density 2.638×10^{-3} kg/m^3 at station S5, (Table 4.1) and highest wet density equal 2.811×10^{-3} kg/m^3 at station S4. While the lowest dry density is at S1 station with an average value of 2.073×10^{-3} kg/m^3, and highest dry density equal 2.646×10^{-3} kg/m^3 at station S4. According to National Oceanic and Atmospheric Administration, microplastic density with an average of 0.1–1 item/m^2 shows that these waters have complex environmental problems and have high turbidity levels. The geographical and topographic conditions of the Al-Hilla River lead to the speed of the river flow which greatly affects the microplastic distribution process from upstream to downstream area as shown in table 4.2.

Table 4.2: Characteristics of Al-Hilla River at sampling locations.

Station	Water flow (m^3/s)	Depth, m	Width, m
S1	100	8	45
S2	95	3.90	77
S3	87	3.76	104
S4	79	4.97	58
S5	75	5.20	62

Microplastic density significantly vary in each station, due to the different factors of each station. Residents who lived around of S1, S2, and S4 station chose to dump garbage directly into the river because there is no temporary waste disposal site (WDS) in the nearby, while at S3 station already had a WDS but had not been fully operated. S5 station is one of five stations that operate a temporary WDS properly, so the microplastic percentage in the waters and the surrounding environment is the smallest than others. In all of the stations, the 3R (reuse, reduce, and recycle) is not yet implemented at the household level and the public building. The recreational areas without temporary WDS or improper waste management potentially increase volume of waste discharged to water rivers.

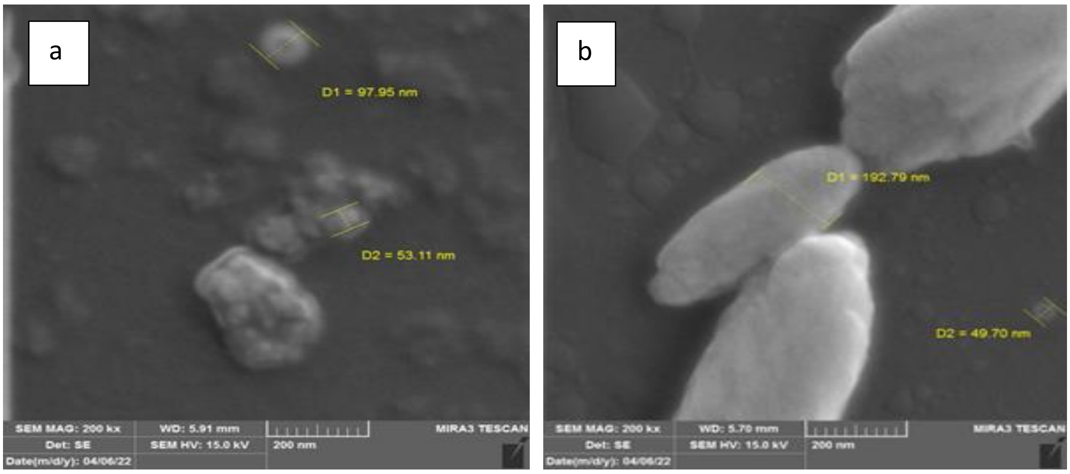
4.3.2. Microplastic composition

Based on the types of plastic found in the field, macroplastic is divided into nine main groups, namely plastic bags, product packaging, styrofoam boxes, plastic bottles, sponges (cigarette butts, head protector foam), plastic utensils and drinks (spoons, cups, straws), baby equipment (diapers, verbs, towels), slippers and others. Based on the data collected, the product packaging (15 items) is the most dominant. This plastic packaging is applied for snacks, kitchen ingredients, shampoo, and detergent. The next rank is in order by plastic bags (18 items), styrofoam boxes (15 items), foam (14 items), plastic bottles (13 items), food utensils (10 items), baby equipment (7 items), other slippers 5 items), and other (4 item). From the results of this grouping, it can be illustrated that household waste dominates the types of macroplastic that have been collected in the Al-Hilla River.

The macroplastic collected then classified according to the type of polymer constituents. According to secondary data and observation at label packages signs, five types of polymers were identified, namely polyethylene terephthalate, polystyrene, high-density polyethylene, polypropylene, low-density polyethylene and other. The most commonly found polymer is low-density polyethylene (67%). Low-density polyethylene is a constituent polymer of plastic bags and packaging. Polystyrene is found at 25% which is a constituent of boxes made of styrofoam and cigarette butts. Polyethylene 8%.

4.4. Microplastic analysis for raw water samples by using SEM

The resulte from SEM images appear in the Fig. (4.3: a, b, c, d and e) shows the presence of microplastic materials with irregular shapes and sizes under the power of magnification of 200 nm. The sampling stations along Al-Hilla river show different diameters and shapes of microplastic particels. The reason for these differense were according to the varians of pollutants. The most area appeared in SEM containing the proportion of microplastic materials was Al- Hashimiyya area (S5), with diameter of the largest microplastic particles was 191.02 nm and the smallest was 28.77 nm, and the shape was white gelatinous forms. Al-Sayyid Al-Farsi area (S3) shows that the largest diameter was 178.19 nm and the smallest clear diameter under the electron microscope was 47.15 nm, and its shape was interconnected crystalline particles. Al-Athar area (S2) with the largest diameter was 192.79 nm and a smaller diameter was 49.70 nm in the form of long sticks overlapping with each other. Followed by Al-Dora Bridge area (S4) with a diameter of 105.62 nm and a smaller diameter was 45.21 nm, and the shape was similar to the shape of the particles in Al- Hashimiyya area. Finally, Sadat Al-Hindiya area (S1) has microplastic diameter range from 95.97 nm to 53.11 nm. This is agreed with the findings conducted by *Andrady*, 2011.



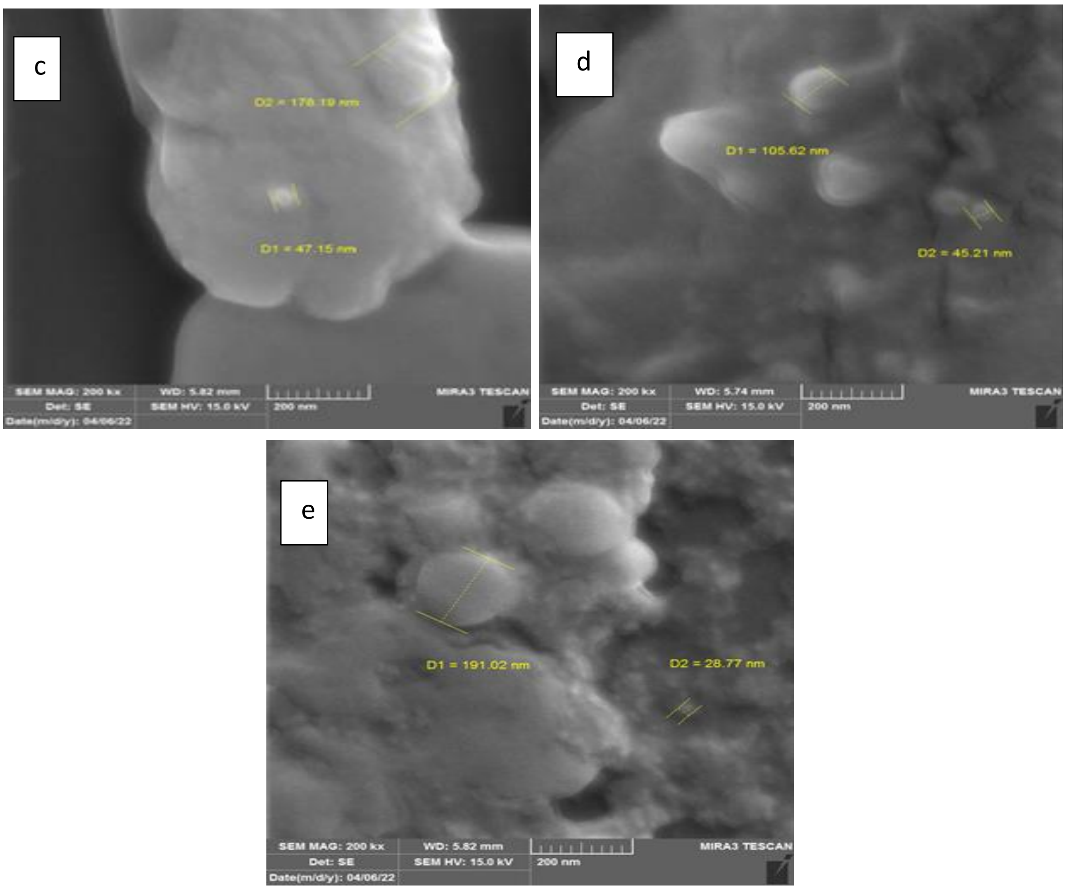
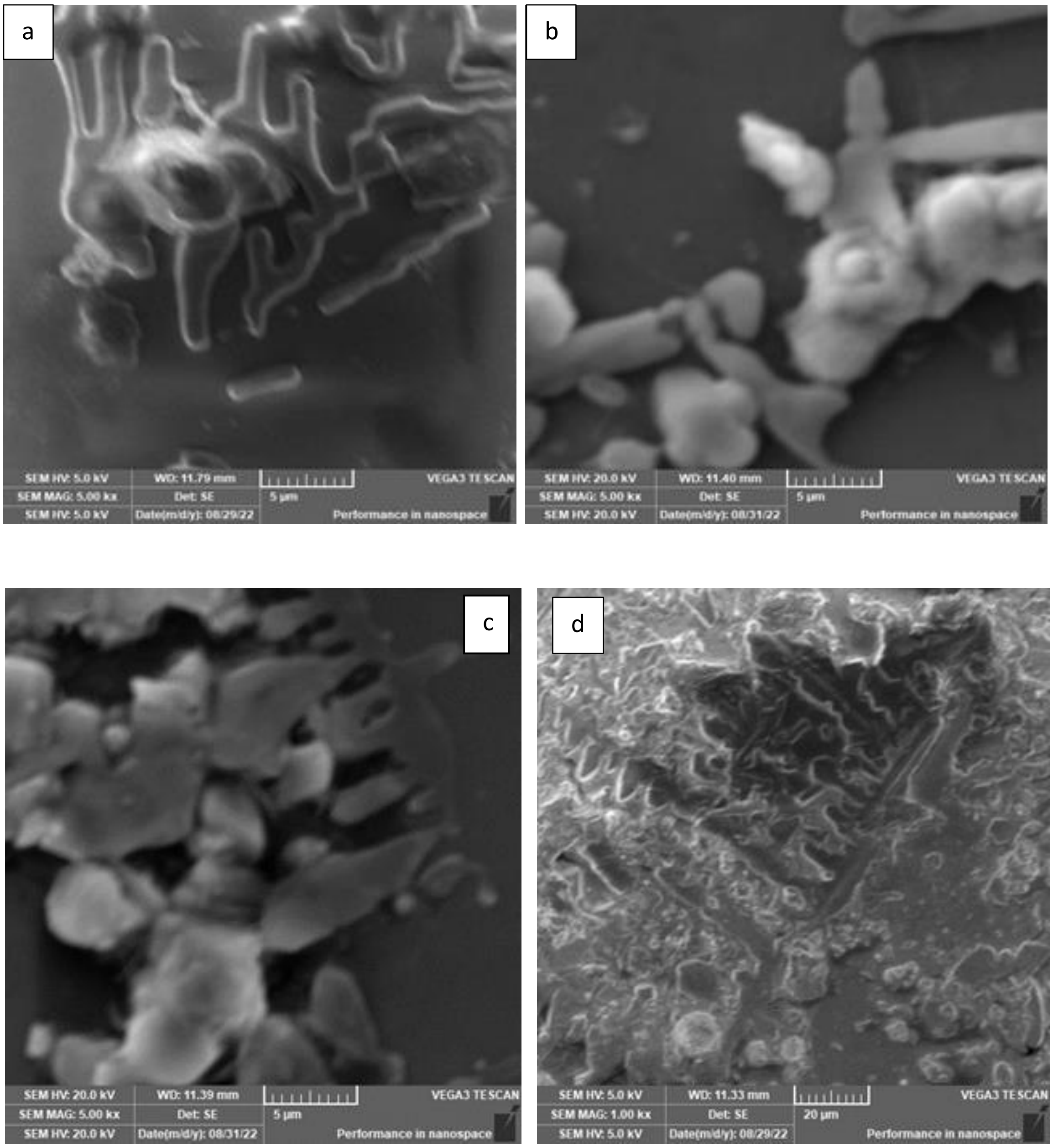


Fig. 4.3: SEM test of microplastic in raw water at a) Sadat Al-Hindiya, b) Al-Athar area, c) Al-Sayyid Al-Farsi area, d) Al- Dora Bridge station, e) Al-Hashimiyya center.

4.5 Microplastic analysis for Drinking water samples by using SEM

The morphological characteristics of drinking water samples collected from several locations along Al-Hilla River are displayed in Fig. 4.4, Fig (4.4, a) represents the drinking water sample of Al-Bakarly rly area (DS1) under a magnification of 5 mm. The image demonstrates that the chain-like microplastic particles range in diameter from $1*10^{-3}$ mm to $4*10^{-3}$ mm. Two stations were taken in the Al-Khusrawiya area; the first station (DS2) is shown in Fig. (4.4, b), and the second station (DS3) is shown in Fig (4.4, c), the two stations were magnified by a factor of 5 mm). The remaining two stations for the Al-Tayyara area are depicted by Fig. (4.4, d and e). Station (DS4), the microplastic particles' sizes ranged from ($0.5*10^{-4}$ mm to $2.5*10^{-3}$mm) and it is represented in the Fig. (4.4, d), while the second station (DS5) the diameters of microplastics ranged from ($1.3*10^{-3}$ mm to 3.510^{-3} mm). These findings were in line with those of an earlier study Andrady, 2011.



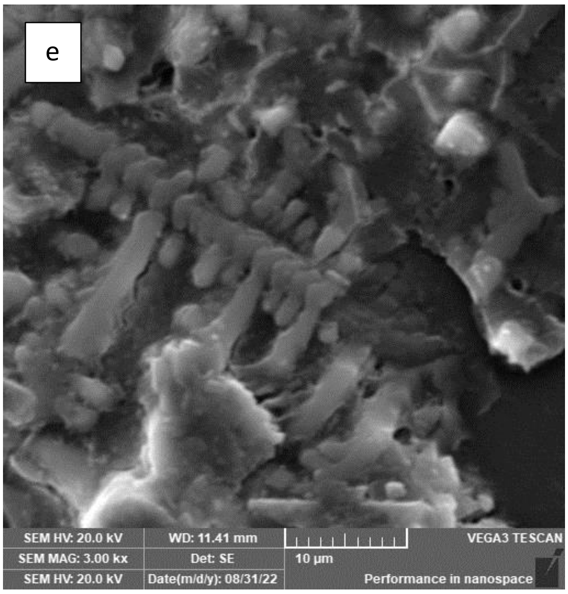


Fig. 4.4: SEM test of microplastic in drinking water sample at a) Al-Bakarly rly area, b) Al-Khusrawiya area first station, c) Al-Khusrawiya area second station, d) Al-Tayyara area first station, e) Al-Tayyara area second station.

4.6 Microplastic analysis for Raw water samples by using FTIR

Fourier transformation the collection and conversion of infrared wavelength absorption or transmission into spectra is done using an infrared spectrometer. When a molecule transforms infrared energy into molecular vibrations, these spectra are produced. In the spectrum, these vibrational vibrations produce bands that appear at particular wavelengths (cm^{-1}). Then, each wavelength is dependent on a variety of additional issues that can be utilized to identify the different bond types in the test sample. In order to determine the functional groups and chemical structure of the solid particles at Sadat Al-Hindiya (S1), infrared spectroscopy was used, as shown in Fig. 4.5. The band of polyvinyl chloride can be identified as the band at 601.791 cm^{-1}, which corresponds to the band of metal oxides at 470.63 cm^{-1} (PVC). Groups of C-O or C-N can be attributed to the band at 1126.43 cm^{-1}. While the band at 1627.92

cm^{-1} of the bond NH of amide and 1760 cm^{-1} is the stretching deformation of C=O. A polyamide polymer, such as Nylon6,6, was indicated by the presence of an amide and carbonyl group. The C-H aliphatic structure may vibrate, producing the 2850 cm^{-1} band. The skeletal O-H bond of cellulose are represented by the band 3410.15. This is agreed with the findings conducted by Murase, H., 2003.

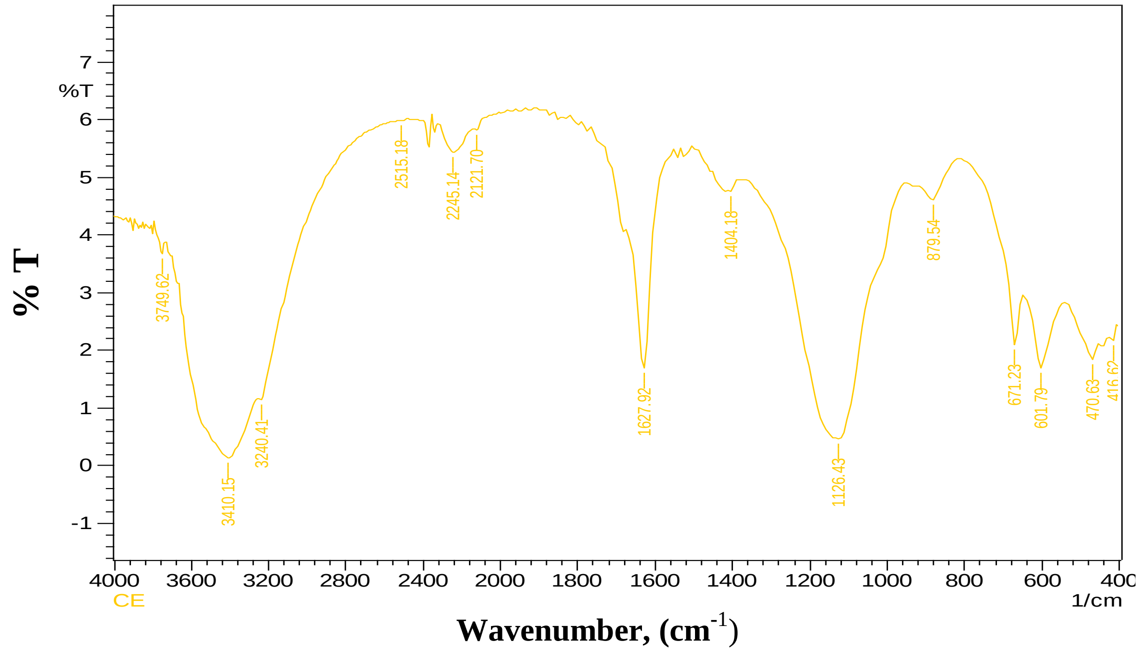


Fig. 4.5: FTIR for a raw water sample of Sadat Al-Hindiya.

Fig. 4.6 illustrates infrared spectroscopy of the solid particles at Al-Athar area (S2) to identify the functional groups and chemical structure. The band of metal oxides at 416.62, the band C–Cl groups can be corresponded to the band at 470.63 - 671.23 cm^{-1}. The band at 1118.71 cm^{-1} was attributed to groups of C-O or C-N. While the band at 1635.64 cm^{-1} of the bond NH of amide. The 2507.46 cm^{-1} band can result from vibrations of C-H aliphatic structure. This is agreed with the findings conducted by Murase, H., 2003.

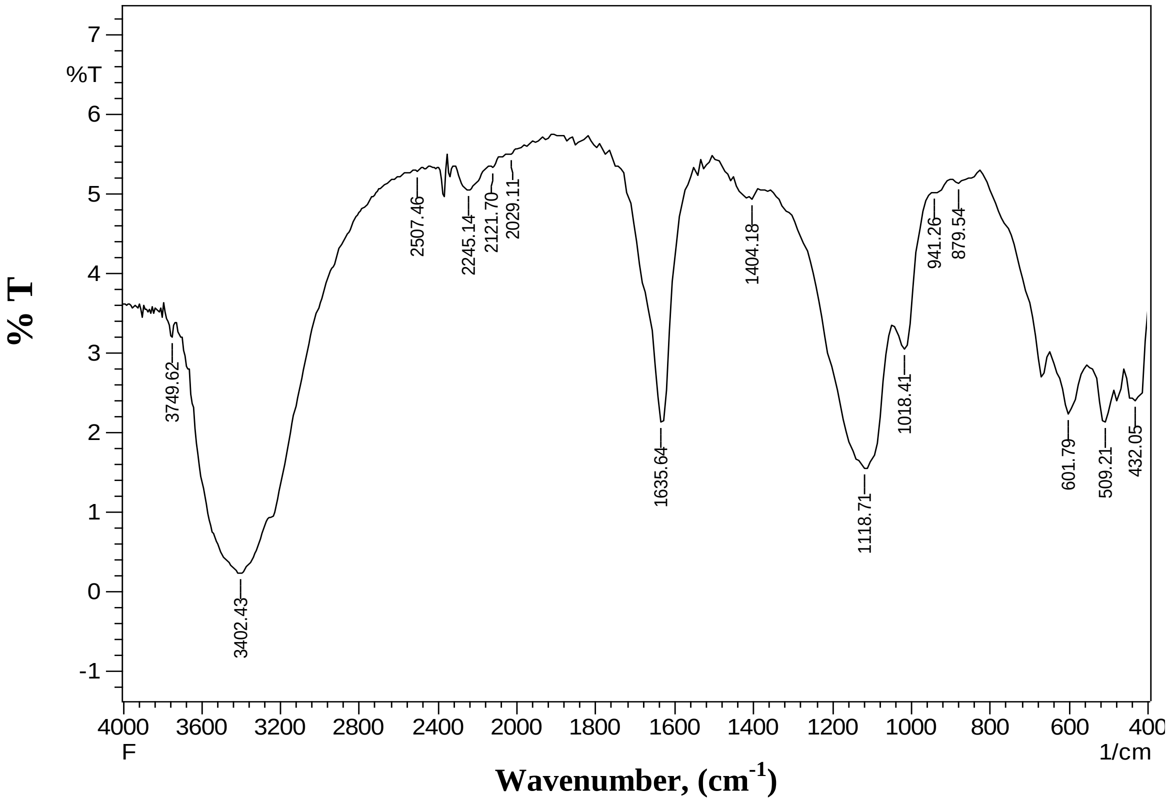


Fig. 4.6: FTIR for a raw water sample of Al-Athar area.

In order to determine the functional groups and chemical structure of the solid particles at Al-Sayyid Al-Farsi area (S3), infrared spectroscopy is shown in Fig. 4.7. The band of metal oxides at 401.10 cm^{-1}, the band C–Cl groups can be corresponded to the band at 432.05 - 601.79 cm^{-1}. The band at 1118.71 cm^{-1} was attributed to groups of C-O or C-N. While the band at 1635.64 cm^{-1} of the bond NH of amide. The 2507.46 cm^{-1} band can result from vibrations of C-H aliphatic structure. The bands 3248.13 and 3402.43 cm^{-1} belong to the bonds of O-H and C-C skeletal of cellulose. This is agreed with the findings conducted by Murase, H., 2003.

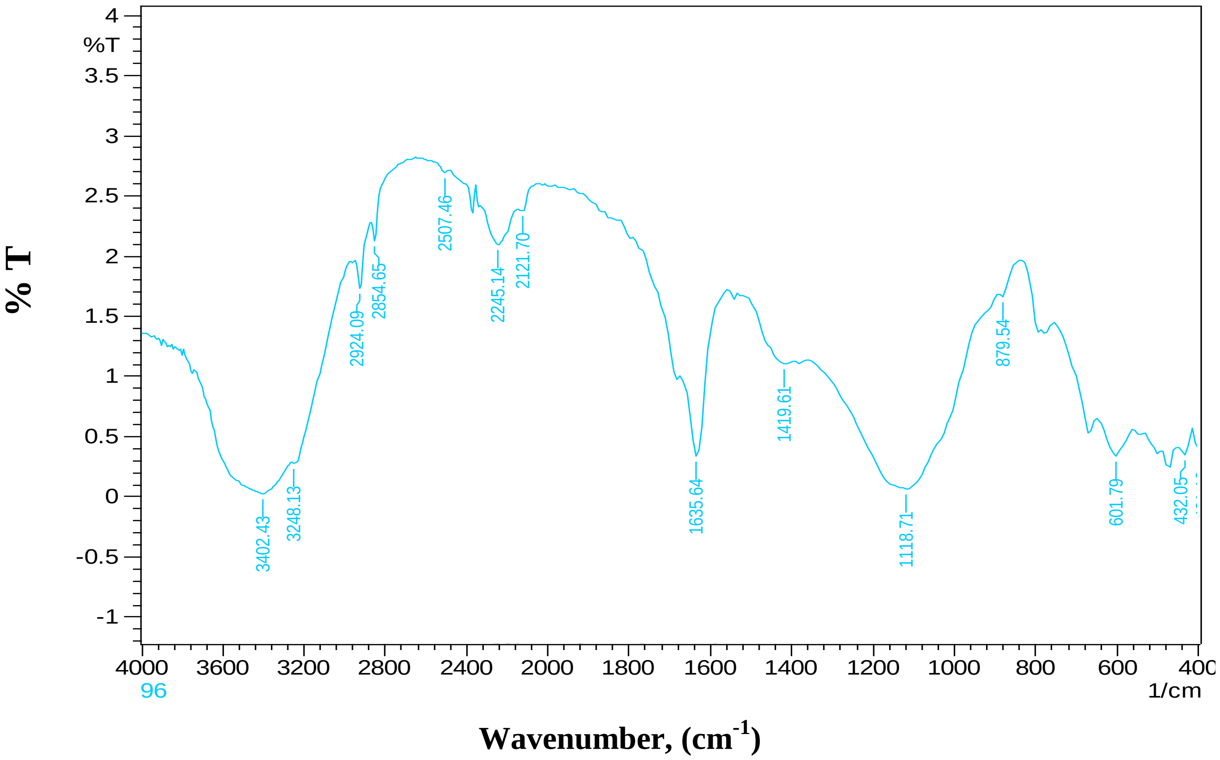


Fig. 4.7: FTIR for raw water sample at Al-Sayyid Al-Farsi area.

Figs. 4.8 and 4.9 show FTIR of the samples for Al-Dora Bridge area (S4) and Al-Hashimiyya station (S5).

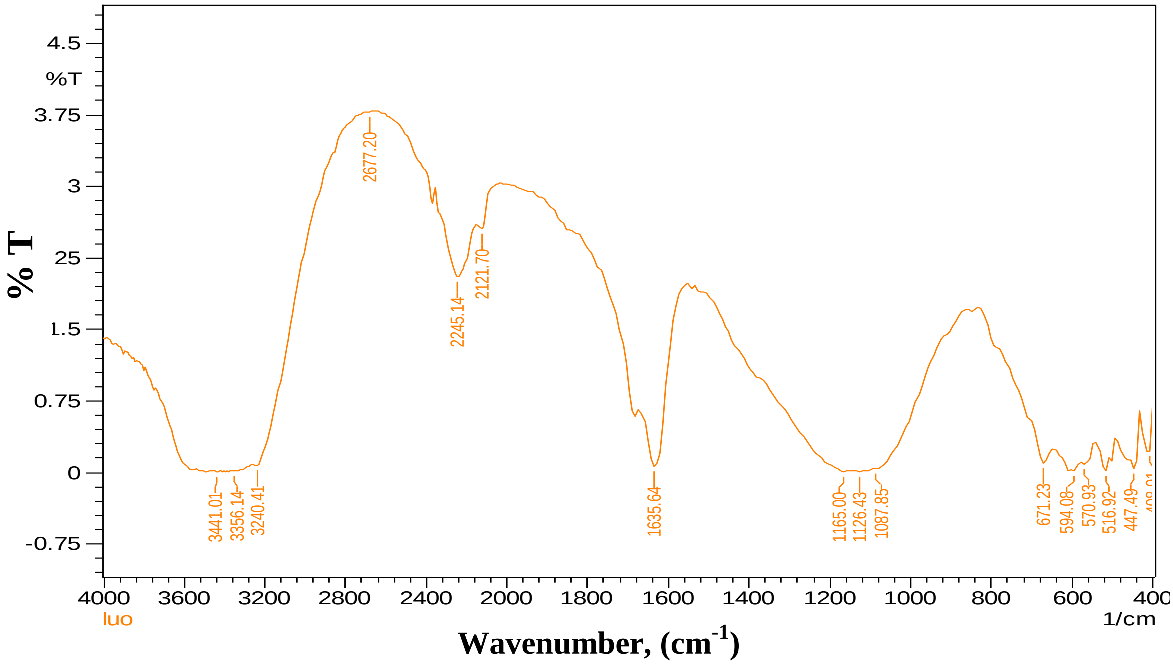


Fig. 4.8: FTIR for raw water sample at Al-Dora Bridge

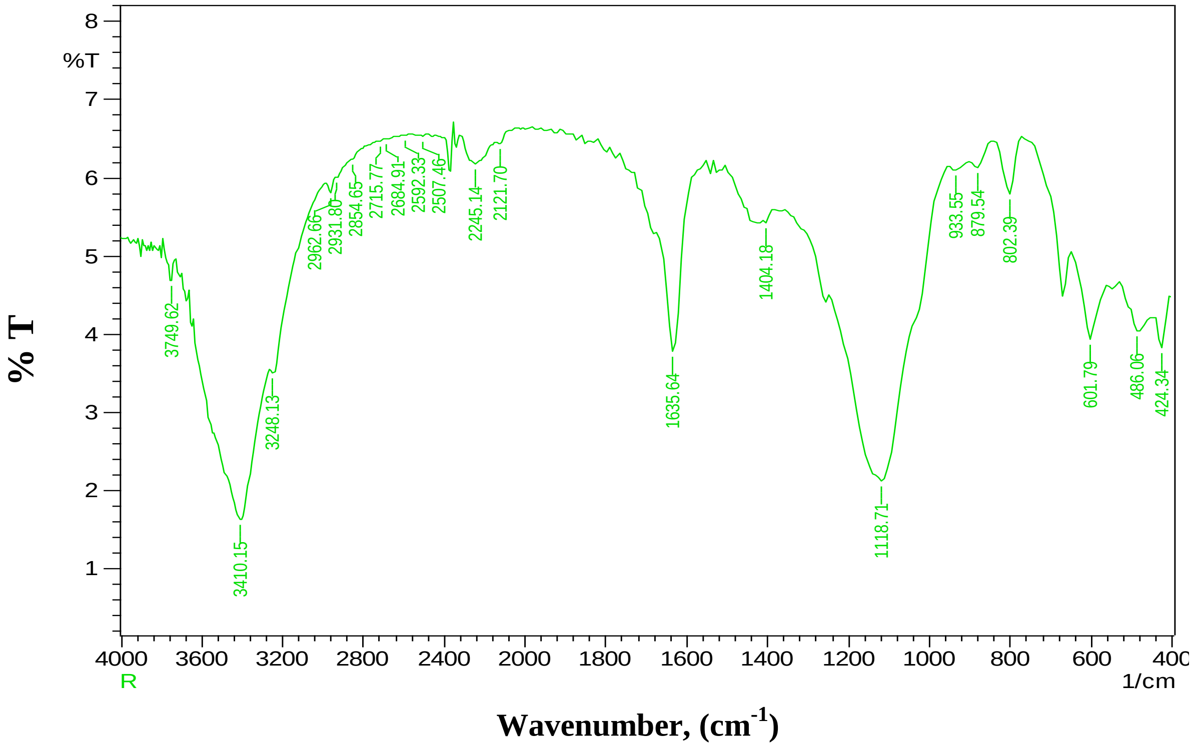


Fig. 4.9: FTIR for raw water sample at Al-Hashimiyya Center.

All raw water samples for these stations contain metal oxides, Poly Vinyl Chloride (PVC)**,** polyamides polymer like Nylon6,6 and skeletal of cellulose, and this reason is common because of the use of these materials containing such polymers, and therefore they are thrown into Al-Hilla River, which causes water pollution with microplastic materials. The obtained results were interpreted on the basis of the links in each of the five regions, and each link was represented in Excel format.

Figs. 4.10 – 4.16 shows the variation of bonds such as metal oxides, C-Cl, C-O or C-N, C=O, C-H, O-H and NH or NH2 for raw water samples in the five stations.

For example Fig. 4.10 explain the variation of metal oxides for raw water samples in the five stations. The average value of metal oxides for stations shows that (S1) has the highest value, followed by (S5, S4, S3) and finally (S2). The reasons of differences in average values of metal oxides could be appear because of the nature of study area and the diversity of the area from residential to agricultural to industrial, as well as the pollutants excreted through waterways or thrown into river water.

The reasons for the difference in the bond between the stations can appear because of the difference in the study area, the distance of each station from the other, the diversity of the stations' areas from residential to agricultural to industrial, as well as pollutants that are excreted through waterways or that are dumped into river water, and the difference in the depth of the water column from one region to another.

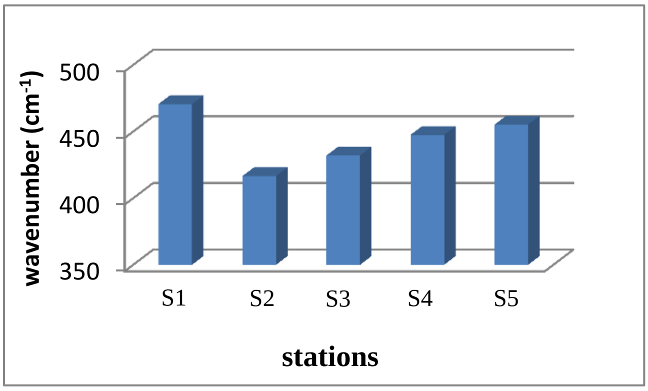


Fig. 4.10: Variation of metal Oxides for raw water samples in five stations.

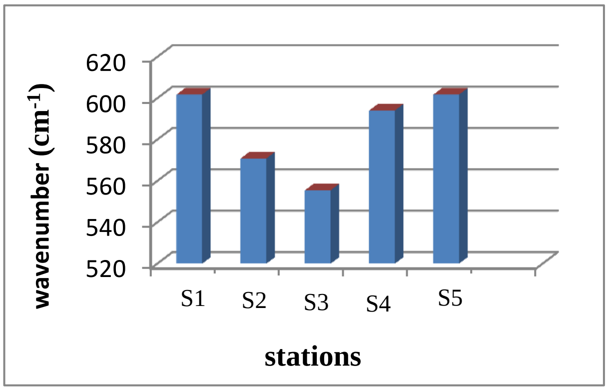


Fig. 4.11: Variation of C-Cl for raw water samples in five stations.

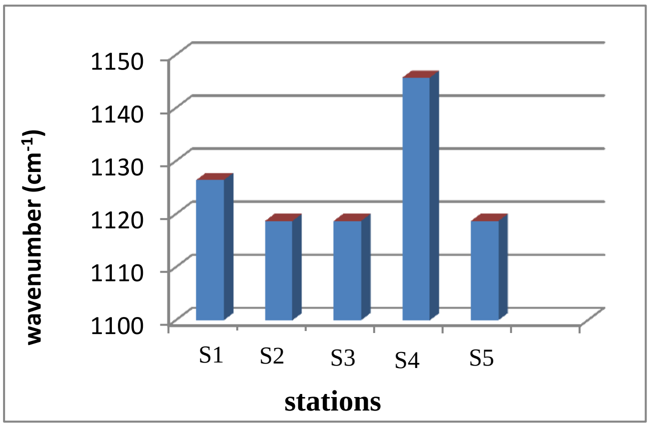


Fig. 4.12: Variation of C-O or C-N for raw water samples in five stations.

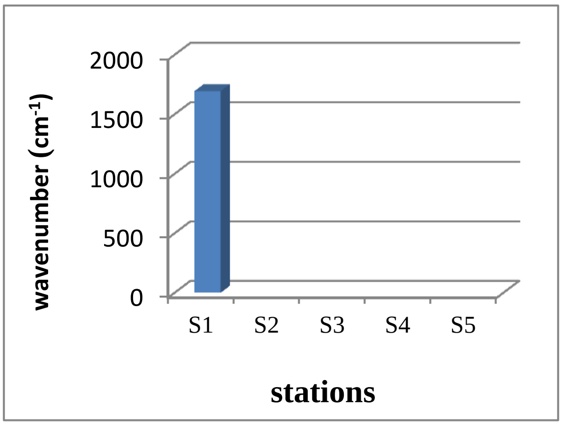


Fig. 4.13: Variation of C=O or for raw water samples in five stations.

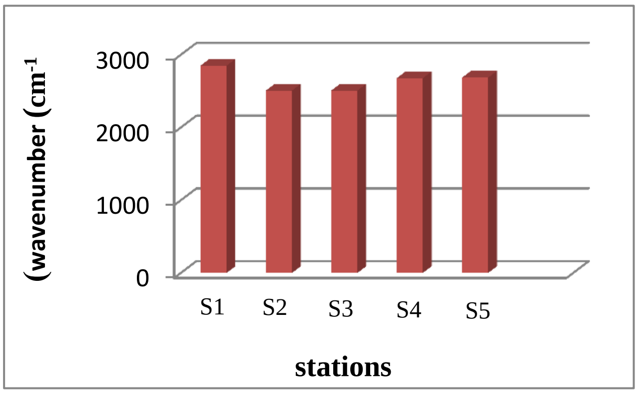


Fig. 4.14: Variation of C-H for raw water samples in five stations.

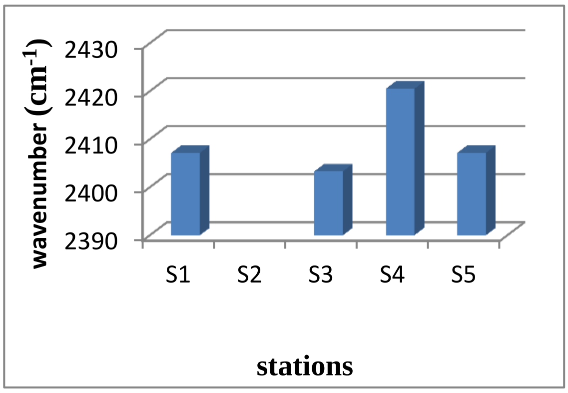


Fig. 4.15: Variation of O-H for raw water samples in five stations.

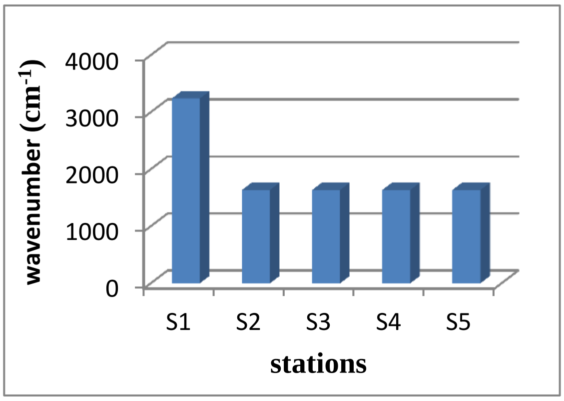


Fig. 4.16: Variation of NH or NH_2 for raw water samples in five stations.

4.5.2 Microplastic analysis for Drinking water samples by using FTIR

In Al-Bakarly arly area (DS1), by interpreting the results obtained during the FTIR examination (Fig. 4.17), a set of chemical bonds was obtained (424.34 – 486.06) cm^{-1} metal oxides, the band C–Cl groups can be corresponded to the band at 601.79 cm^{-1} and this can be revealed that of Poly Vinyl Chloride (PVC). The band at 1118.71 cm^{-1} can be attributed to groups of C-O or C-N. While the band at 1627.92 cm^{-1} of the bond NH of amide. The bands 1427.32 and 3402.43 cm^{-1} belong to the bonds of O-H and C-C skeletal of cellulose. This is agreed with the findings conducted by Ana Alonso-Simón, 2011.

The results of FTIR for the other locations were illustrated in Figs. 4.18 to 4.21.

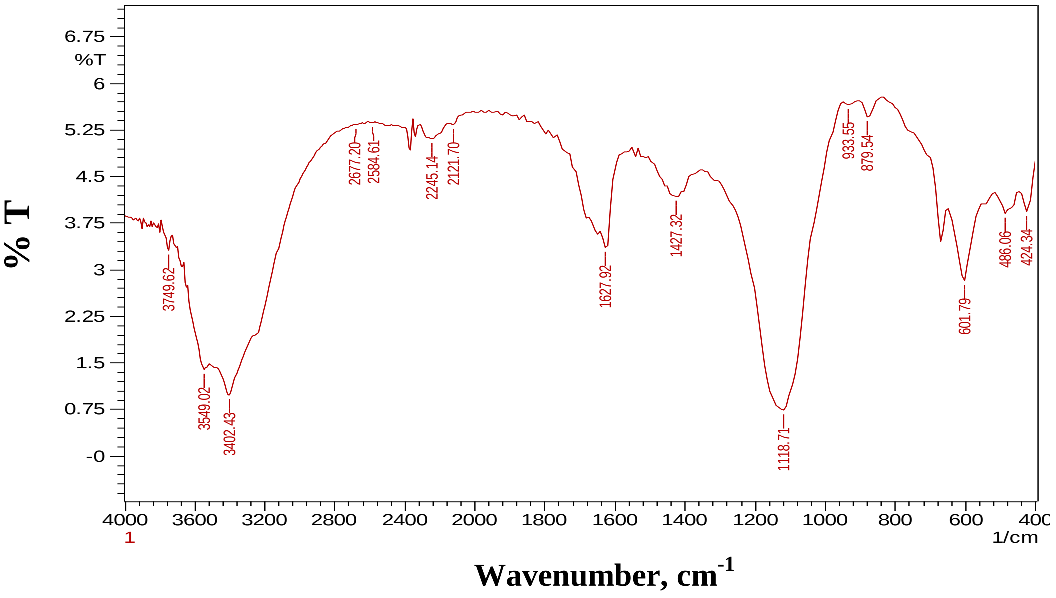


Fig. 4.17: FTIR test of drinking water sample at AL- Bakarly area.

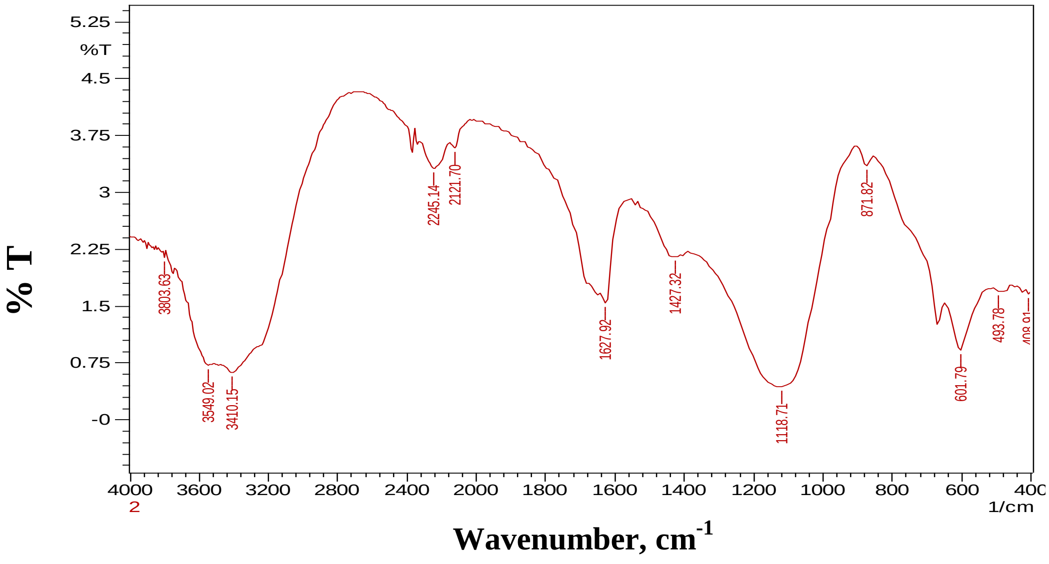


Fig. 4.18: FTIR test of drinking water sample at Al-Khusrawiya area (first location).

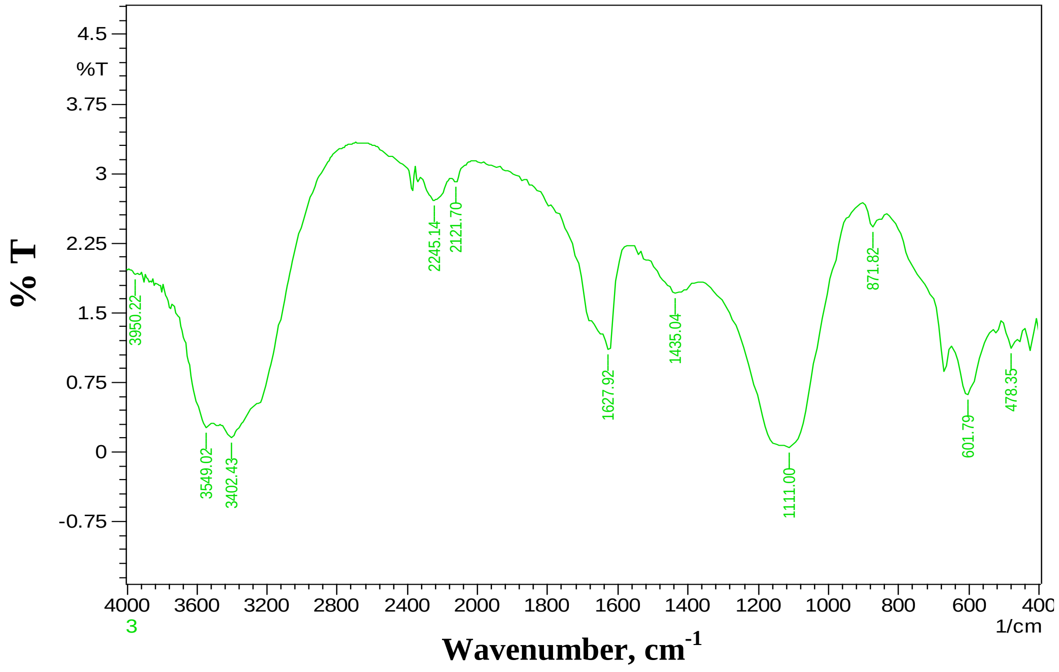


Fig. 4.19: FTIR test of drinking water sample at Al-Khusrawiya area (second location).

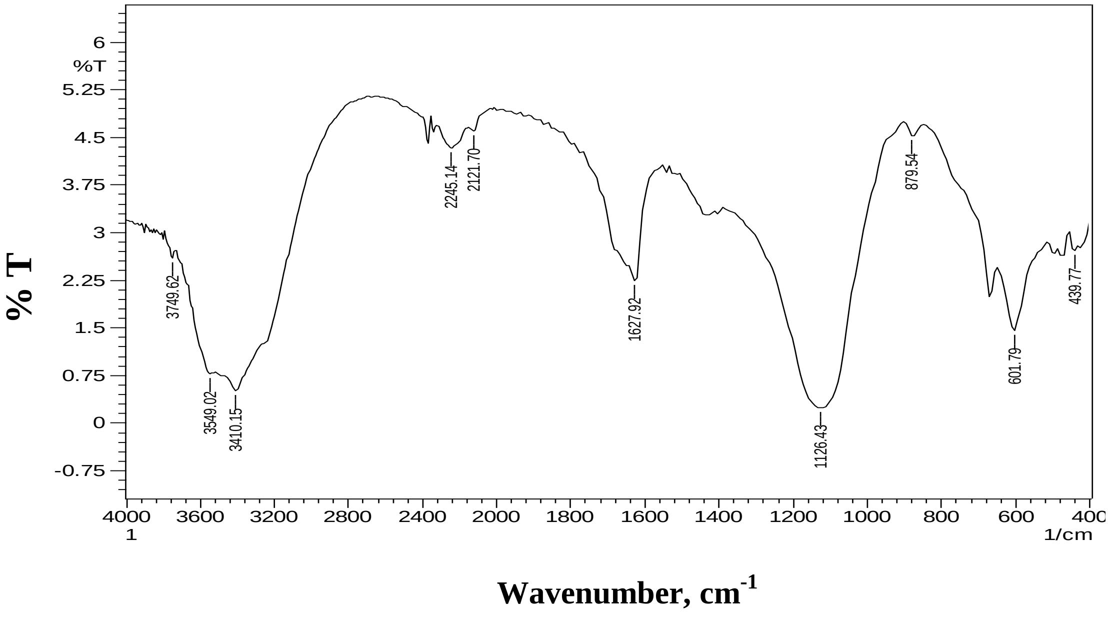


Fig. 4.20: FTIR test of drinking water sample at Al-Tayyara area (first location).

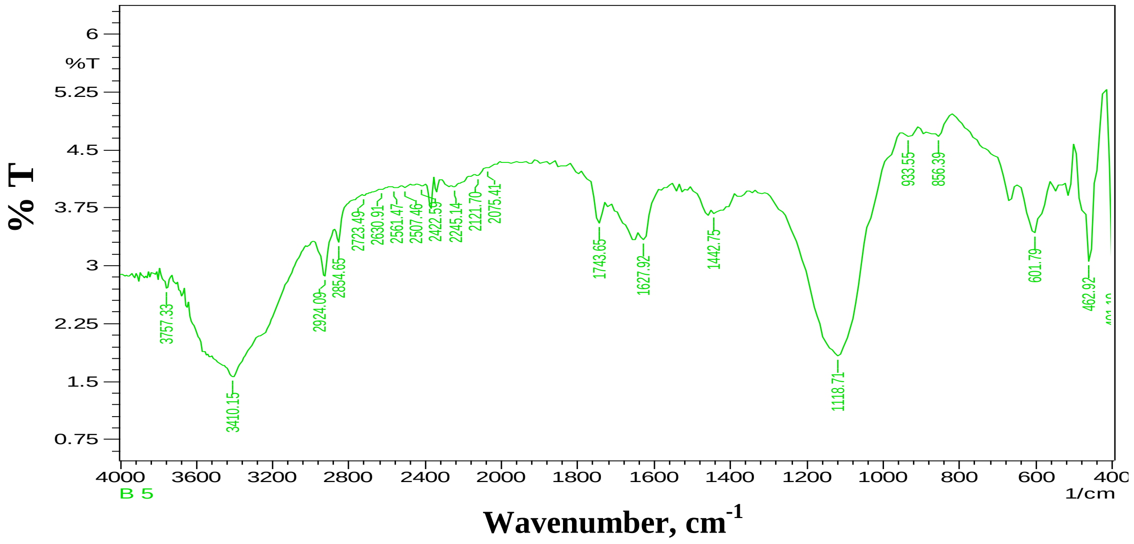


Fig. 4.21: FTIR test of drinking water sample at Al- Tayyara area (second location).

All the drinking water sampling stations for the study area contain metal oxides, polyvinyl chloride (PVC), polyamide polymer such as nylon 6,6 and structural cellulose.

Fig. 4.22 explain the variation of metal oxides for drinking water samples in the five stations. The average value of metal oxides for stations shows that (DS3) has the highest value, followed by (DS5, DS1, and DS2) and finally (DS4).

Figs. 4.23 – 4.28 shows the variation of bonds such as metal oxides, C-Cl, C-O or C-N, C=O, C-H, O-H and NH or NH_2 for drinking water samples in five stations.

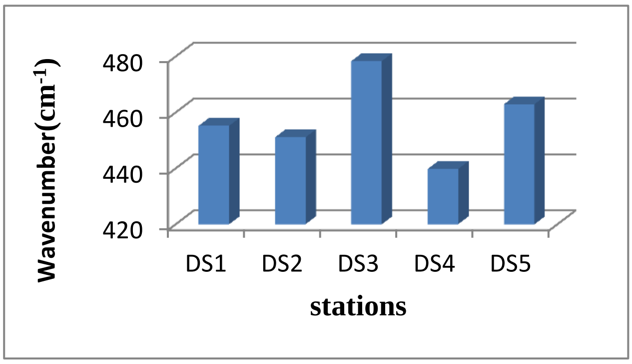


Fig. 4.22: Variation of Metal oxides for drinking water samples in five stations.

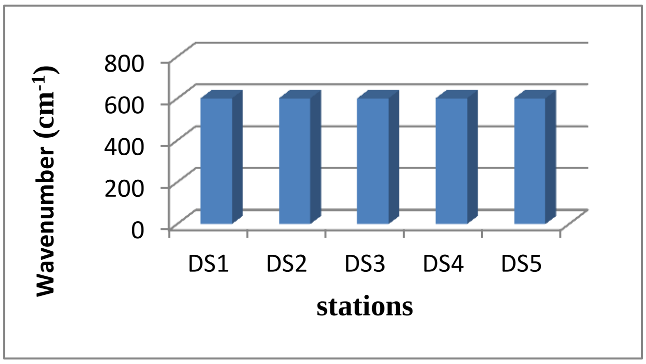


Fig. 4.23: Variation of C-Cl for drinking water samples in five stations.

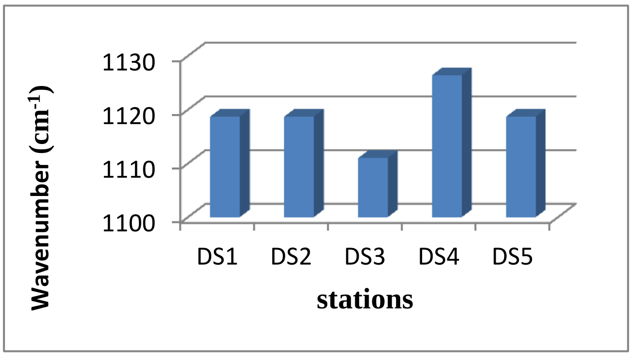


Fig. 4.24: Variation of C-O or C-N for drinking water samples in five stations.

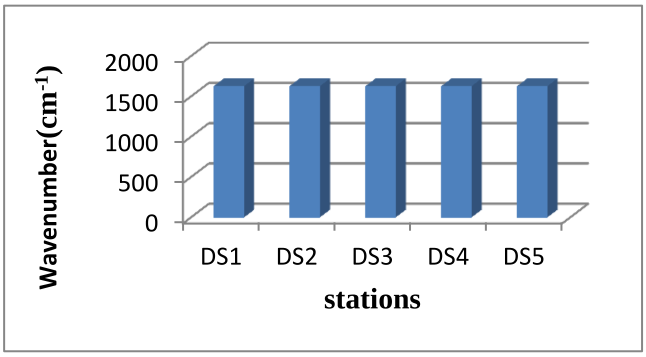


Fig. 4.25: Variation of C=O for drinking water samples in five stations.

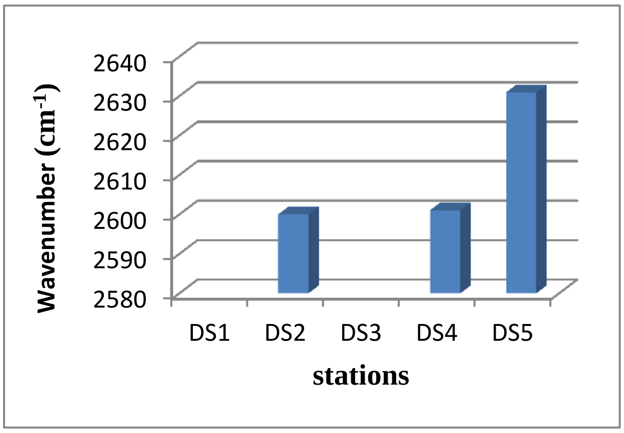


Fig. 4.26: Variation of C-H for drinking water samples in five stations.

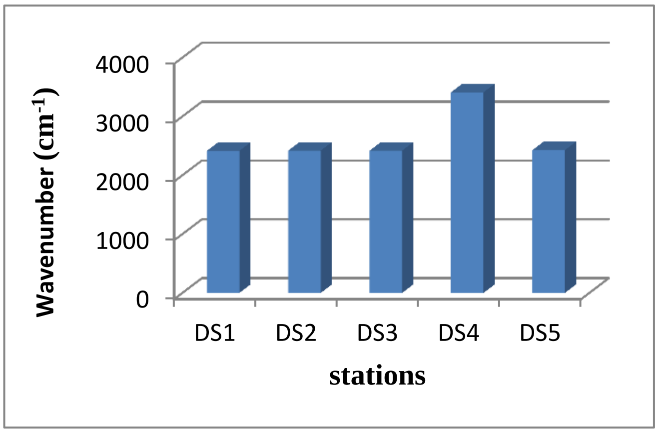


Fig. 4.27: Variation of O-H or C-C for drinking water samples in five stations.

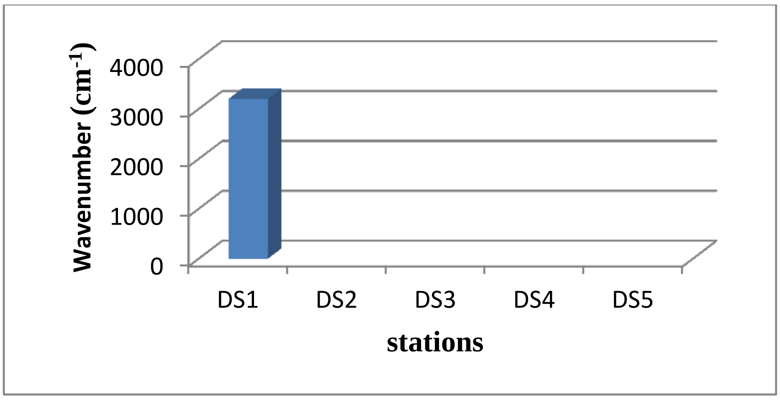


Fig. 4.28: Variation of NH or NH_2 for drinking water samples in five stations.

4.6 Microplastic analysis in Raw Water Samples by MRS test

The Micro-Raman spectrometer (MRS) test of raw water samples for five stations are illustrated in Figs 4.29 – 4.33.

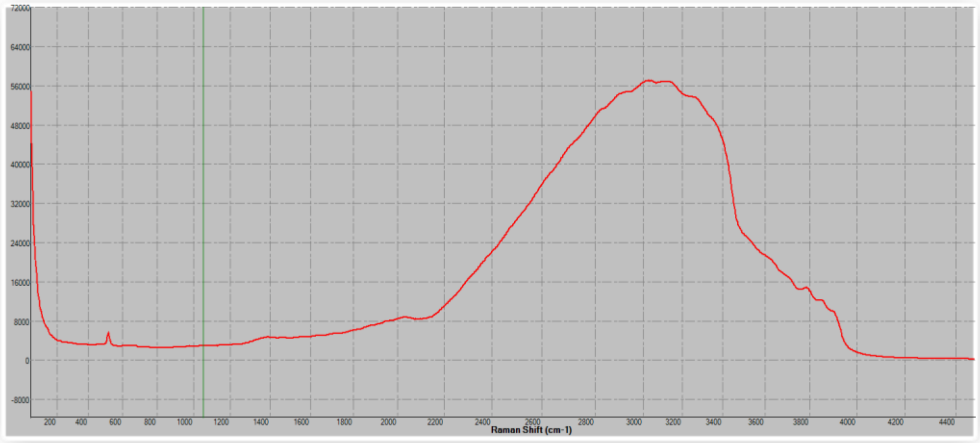


Fig. 4.29: MRS test for raw water sample at Sadat Al-Hindiya.

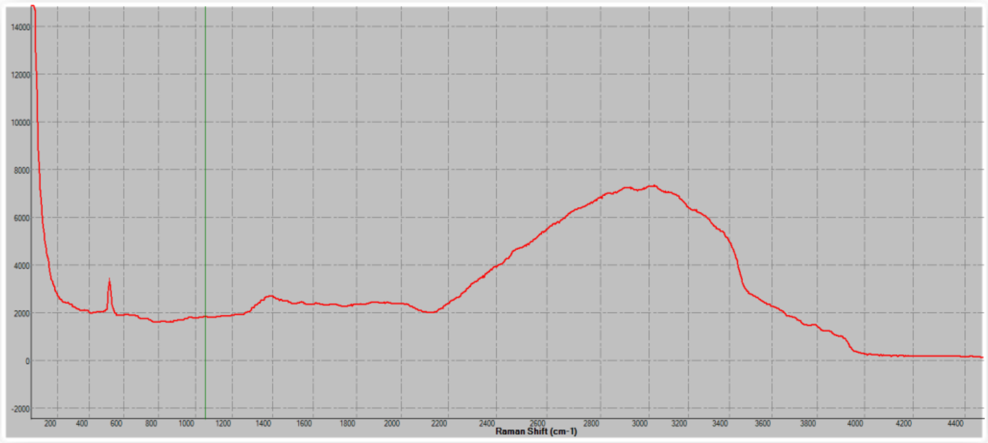


Fig. 4.30: MRS test for raw water at Al-Athar area.

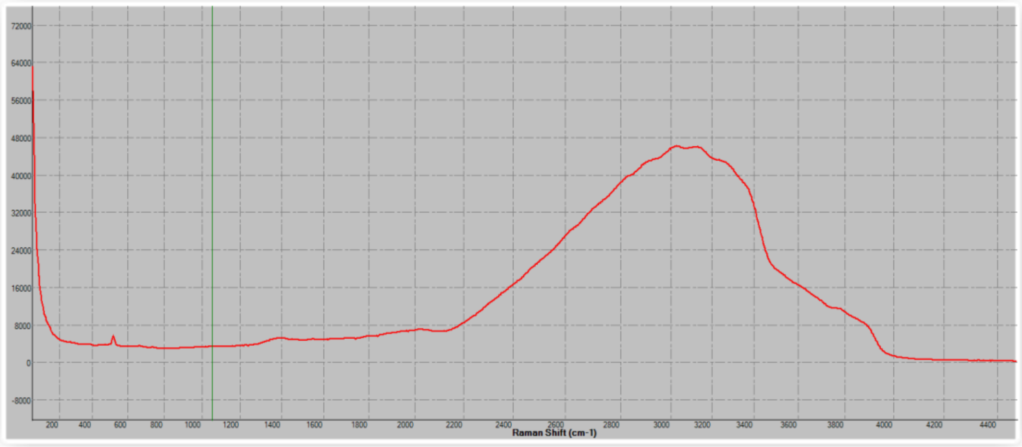


Fig. 4.31: MRS test for raw water at Al-Sayyed Al-Farsi location.

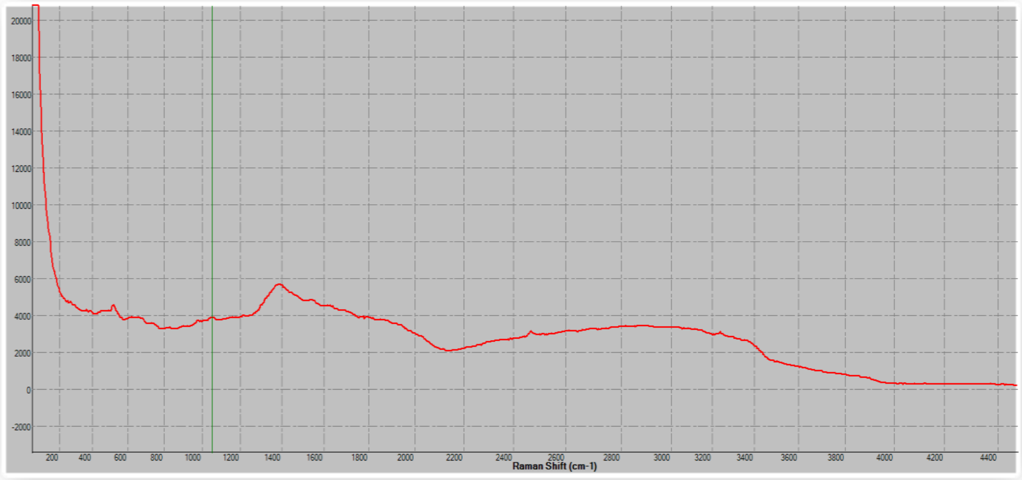


Fig. 4.32: MRS test for raw water at Al-Dora Bridge.

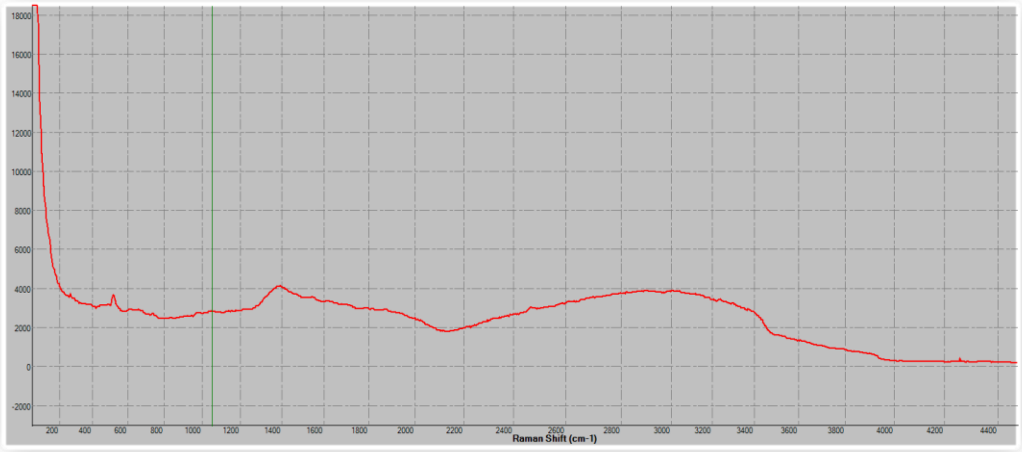


Fig. 4.33: MRS test for raw water sample at Al-Hashmmiya location.

These Figs. of Raman spectra of polyethylene (PE) show main vibrations at (1512, 1450, 1740, 1855, 2400, 2250, 3325, 3850, and 4220) cm^{-1}. The polymers found in aquatic systems, i.e., polyethylene, polypropylene, polystyrene, polyamide, polyesters, and polyvinyl chloride, get a better understanding of the chemical fingerprint of these polymers.

The reason for these variations in Raman spectra tests are due to that the Raman spectroscopy is sensitive to changes in the molecular structure level of PE, such as the degree of crystallinity, which is the key determining factor of PE density (i.e., higher the degree of crystallinity, higher the density). Thus, it is argued that Raman spectroscopy may distinguish low-density polyethylene (LDPE) and high-density polyethylene (HDPE). The Raman spectra of LDPE and HDPE are very similar, but differences are reported in the C–H stretching region. Moreover, the intensity of the symmetric CH_2 stretching mode at 2600 cm^{-1} relative to the asymmetric CH_2 stretching mode at 3100 cm^{-1} appears to be higher for LDPE compared to HDPE. Therefore these two regions, i.e. C-H stretching (2725-3200 cm^{1}) and the CH_2 bending regions (2200-3000 cm^{-1}).

4.7 Drinking water Samples for Micro-Raman Spectrometer

Figs. 4.34 to 4.38 display the spectra for drinking water samples obtained from Al- Bakarly arly, Al-Khusrawiya first and second areas, and Al-Tayyare first and second areas. Polyethylene, Polypropylene, Polycarbonate, and Polyamide were found in the samples tested. Visual differences in the signals from the stretching vibrations of the -CH chain at 2994.918 - 3523.232 cm^{-1} can be used to complete a qualitative examination of the polymers. Two different bands, at 2882 cm^{-1} and 2848 cm^{-1}, which correspond to asymmetric stretching in the crystalline phase and symmetric stretching in the amorphous phase, are visible in the polymer region of these. This was in line with the results that had been by Hiejima, 2018.

The difference between asymmetric vibrations in the crystalline phase and symmetric vibrations in the amorphous phase is what differentiates high-density polyethylene (HDPE) and low density polyethylene (LDPE). The density of the polymer is inversely correlated with the degree of crystallinity. In comparison to amorphous domains, HDPE has more of them than LDPE does. Since the signal from asymmetric stretching vibrations (2882 cm^{-1}) is stronger than the signal from symmetric stretching vibrations, it is highly unlikely that all polyethylene spectra that have been recorded are LLDPE (2848 cm^{-1}). Additionally, the band originating from vibrations of the crystalline group $-CH_2$ has a larger intensity in the 1200–1500 cm^{-1} range than the band originating from vibrations of the group in the liquid phase.

In Fig. 4.34, DS1 station was identified as polypropylene the most common polyamide, exhibits the following bands: C-C deformation at 791.461 cm^{-1}, C-CO stretching at 1038.867 cm^{-1}, C-C skeletal stretching at 1193.495 cm^{-1}, CN stretching and -NH bending of amide III at 1417.707cm^{-1}, $-CH_2$ twisting at 1629.032 cm^{-1}, $-CH_2$ bending at 2043.952, 2355.787 and 2585.152 cm^{-1} and –CH stretching at 2884.101 cm^{-1}.

Spectra for samples collected from four locations (Al-Khusrawiya area first location, Al-Khusrawiya area second location, Al-Tayara area first location and second location) the phenomenon of luminescence occurred. In this identified sample, found polycarbonate, polystyrene and polypropylene.

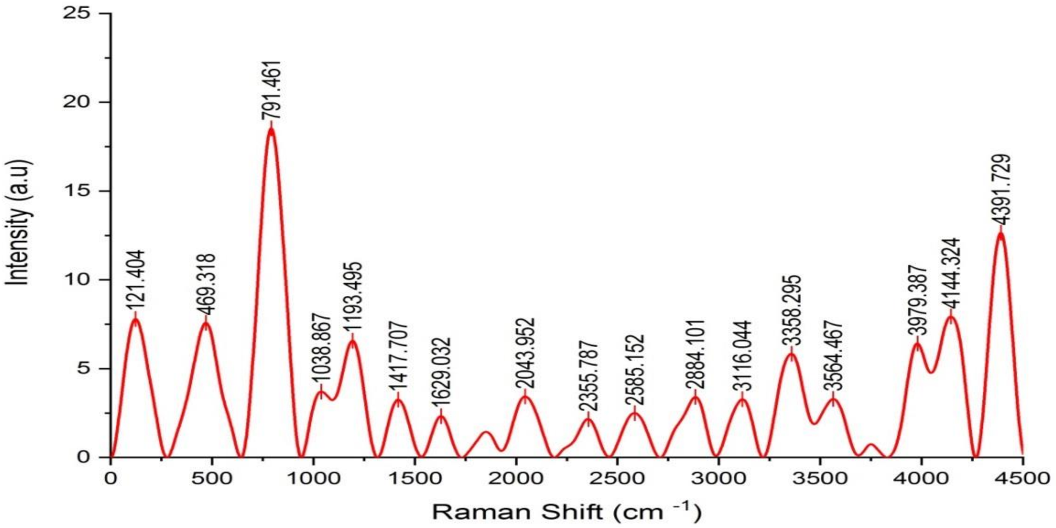


Fig. 4.34: MRS test for raw water sample at Al- Bakarly arly area of the center.

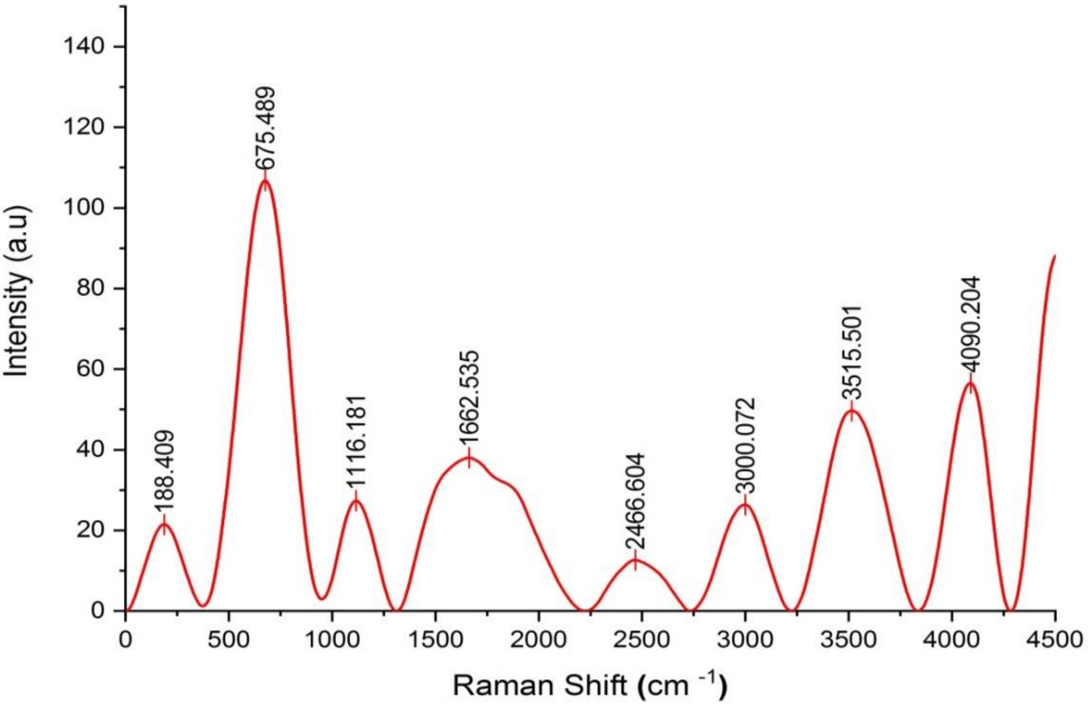


Fig. 4.35: MRS test for raw water sample at Al-Khusrawiya area first location of the center.

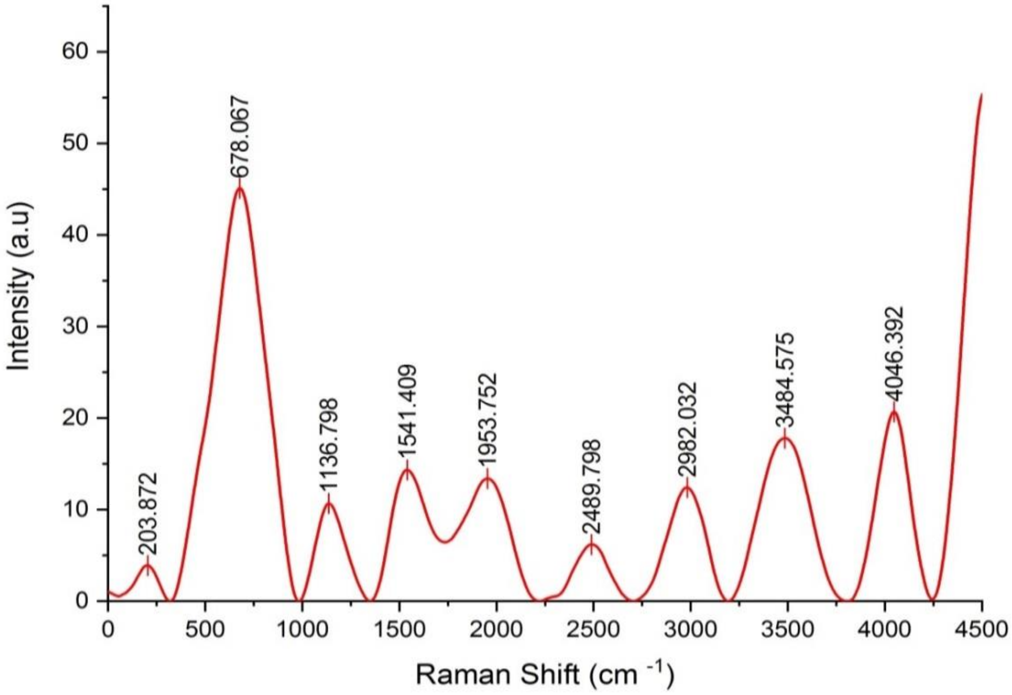


Fig. 4.36: MRS test for raw water sample at Al-Khusrawiya area second location of the center.

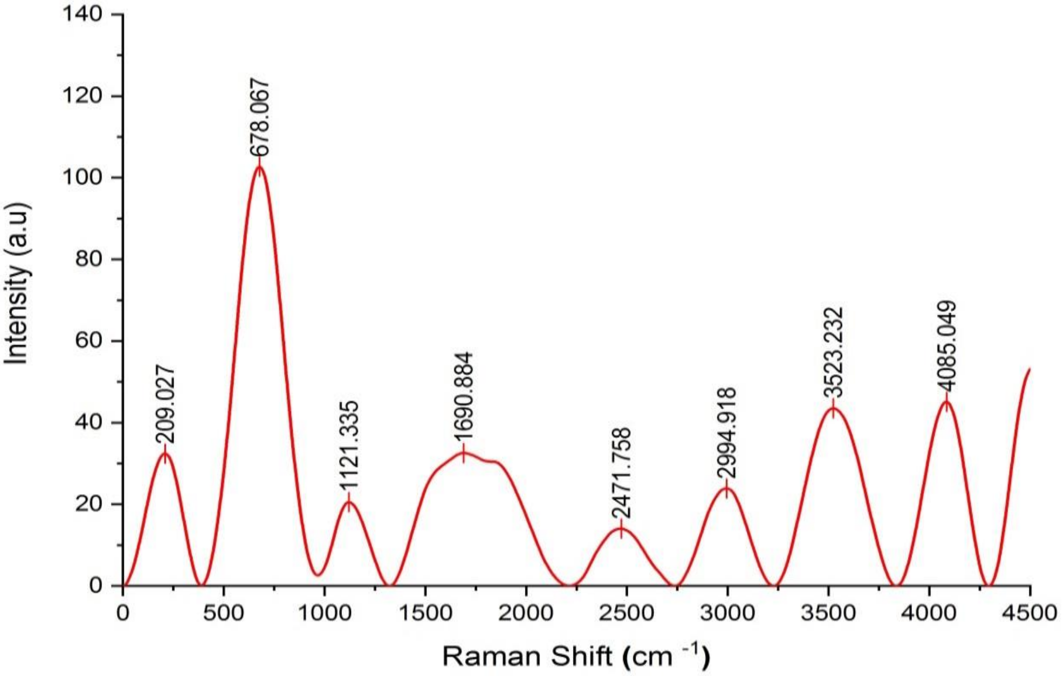


Fig. 4.37: MRS test for raw water sample at Al-Tayyara area first location of the center

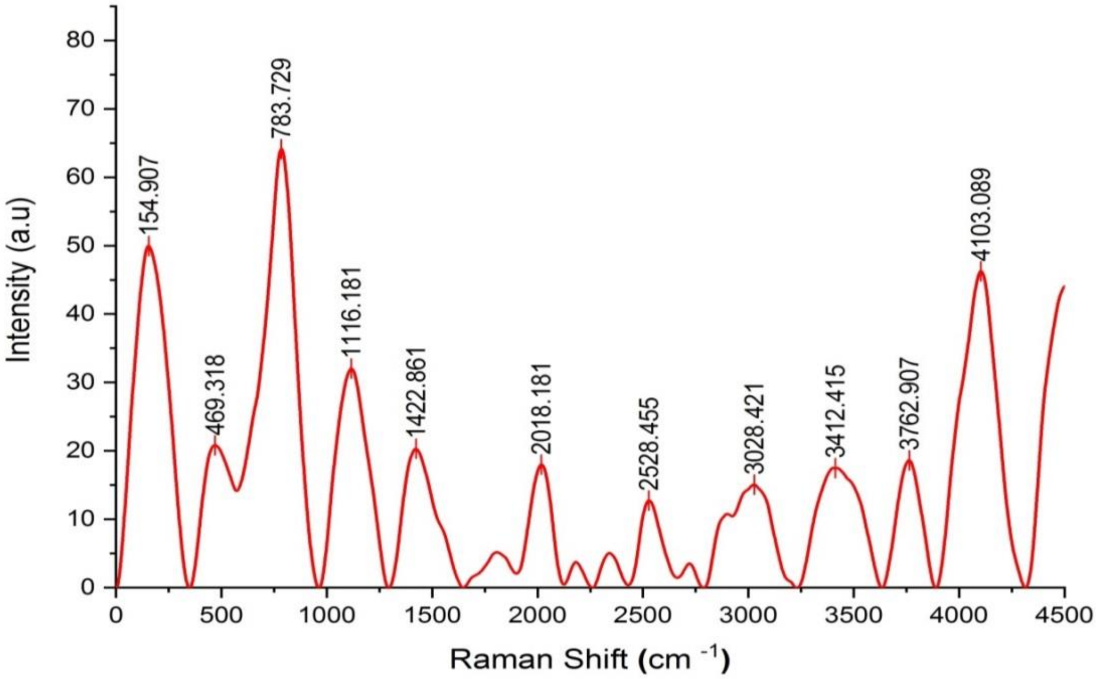


Fig. 4.38: MRS at Al-Tayara area second location of the center.

Spectra for samples collected from four locations (Al-Khusrawiya area first location, Al-Khusrawiya area second location, Al-Tayara area first location and second location) the phenomenon of luminescence occurred. In this identified sample, found polycarbonate, polystyrene and polypropylene.

Chapter Five

Conclusions and Recommendations

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATIONS

5.1 Introduction

This chapter represents the concluding remarks, obtained from this study and recommendations for future studies.

5.2 Conclusions

Based on the results obtained, the following conclusions where be drawn:

1- The most polluted locations by microplastic particle were in stations Al-Dora Bridge and Sadat Al- Hindiya with total plastic mass around 2.811 gm and 2.771 gm, respectively.

2- the Results of density show that the lowest wet density was 2.657×10^{-3} kg/m^3 at station S2, and highest wet density equal 2.811×10^{-3} kg/m^3 at station S4. While the lowest dry density is at S1 station with an average value of 2.073×10^{-3} kg/m^3, and highest dry density equal 2.646×10^{-3} kg/m^3 at station S2.

3- Based on the types of plastic found in the field, macroplastics were divided into nine main groups, namely plastic bags, product packaging, styrofoam boxes, plastic bottles, sponges (cigarette butts, head protector foam), plastic utensils and drinks (spoons, cups, straws), baby equipment (diapers, verbs, towels), slippers and others.

4- The microplastic were classified according to the type of polymer constituents. According to secondary data and observation at label packages signs, five types of polymers were identified, namely polyethylene terephthalate, polystyrene, high-density polyethylene, polypropylene, low-density polyethylene and other. The most commonly found polymer is low-density polyethylene (67%). Low-density polyethylene is a constituent polymer of plastic bags and packaging. Polystyrene is found at 25% which is a constituent of boxes made of styrofoam and cigarette butts. Polyethylene (8%).

5- SEM results of raw and drinking water samples showed the presence of microplastic particles with that size smaller than 5 mm, the range of diameter of the plastic particles in the five locations were between (28.77 - 192.79) nm, while for drinking water ranged between (0.5 - 4.8) nm.

6- FITR results of raw water samples revealed that it mainly consist of metal oxides, Poly Vinyl Chloride (PVC), polyamides polymer like Nylon6,6 and skeletal of cellulose, the main components of microplastic pollutants for drinking water samples are metal oxides, Poly Vinyl Chloride (PVC), polyamides polymer like Nylon6, 6 and skeletal of cellulose.

7- Micro-Raman spectroscopy (MRS) results indicated a variation in polyethylene (PE), and it was between (1450 – 4220) cm^{-1} for raw water samples and between (121.404 – 4391.729) cm^{-1} for samples of drinking water.

5.3 Recommendations for future studies

1. More research is required to monitor and manage pollution source in Al-Hilla River in order to safeguard and improved the quality of water, particularly in the upcoming years since the nation may be facing a water crisis.
2. Expand the investigation from the headwater to the downstream at all seasons, taking into account the extraction of microplastic particles.
3. Study microplastic particles in sediment and their impact on aquatic life in Al-Hilla River.

4. More data are needed on the occurrence of microplastics in drinking-water to assess human exposure from drinking-water adequately.
5. Studies on occurrence of microplastics must use quality-assured methods to determine numbers, shapes, sizes, and composition of the particles found. They should identify whether the microplastics are coming from the freshwater environment or from the abstraction, treatment, distribution or bottling of drinking-water.
6. Further studies could be conducted on the factors that influence the composition and potential specificity of microplastic-associated biofilms, studies could also consider the factors influencing biofilm formation on plastic surfaces, including microplastics, and how these factors vary for different plastic materials, and what organisms more commonly bind to plastic surfaces in freshwater systems.
7. Research could be carried out to better understand the capacity of microplastics to transport pathogenic bacteria longer distances downstream, the rate of degradation in freshwater systems and the relative abundance and transport capacity of microplastics compared with other particles.
8. More research is needed to understand the fate of microplastics across different wastewater and drinking-water treatment processes (such as clarification processes and oxidation) under different operational circumstances, including optimal and sub-optimal operation and the influence of particle size, shape and chemical composition on removal efficacy.
9. Studying the relationships between turbidity (and particle counts) and microplastic concentrations throughout the treatment processes.

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الخلاصة

تعد دراسة توزيع المواد البلاستيكية الدقيقة في المسطحات المائية أمرًا مهمًا نظرًا لإمكانية تحلل جزيئاتها الكيميائية. تتشكل وفرة المواد البلاستيكية الدقيقة من الأنشطة السكانية التي لا تصاحبها أنظمة مناسبة لإدارة النفايات. منذ أن تطورت الأنشطة الصناعية والبشرية بطرق مختلفة في العراق، فقد انخفضت جودة المياه على طول المياه السطحية والجوفية. أجريت هذه الدراسة في الفترة من أغسطس إلى ديسمبر من عام 2022 باستخدام طريقة مصيدة القمامة، وبأخذ العينات الشهرية. تم أخذ خمس عينات بفاصل 30 يومًا بين كل منها. وتتكون مواقع أخذ العينات من خمس محطات للمياه الخام وخمس محطات لمياه الشرب.

تم اختبار عينات المياه التي تم جمعها باستخدام تقنيات مختلفة، بما في ذلك مطياف فورييه للأشعة تحت الحمراء (FTIR)، والمجهر الإلكتروني الماسح (SEM)، ومطياف مايكرو رامان (MRS).

أشارت نتائج التحليل والفحص للمياه الخام إلى أن أكثر المواقع تلوثاً بالجسيمات البلاستيكية كانت في محطتي جسر الدورة وسدة الهندية حيث بلغت الكتلة البلاستيكية الإجمالية حوالي 2.811 غرام و 2.771 غرام على التوالي. أظهرت نتائج FITR لعينات المياه الخام أنها تتكون بشكل رئيسي من أكاسيد المعادن، بولي كلوريد الفينيل (PVC)، بوليمرات البوليمايد مثل النايلون 6،6 وهيكل عظمي من السليلوز. أظهرت نتائج SEM لعينات المياه الخام وجود جزيئات بلاستيكية صغيرة بحجم أصغر من 5 ملم. تراوح مدى قطر حبيبات البلاستيك في المواقع الخمسة بين (28.77 – 192.79) نانومتر.

كما أظهرت محطات أخذ العينات الخمس لمياه الشرب أن المكونات الرئيسية للملوثات البلاستيكية الدقيقة هي أكاسيد المعادن، وبولي فينيل كلورايد (PVC)، وبوليمرات البوليمايد مثل النايلون 6،6 وهيكل السليلوز. أظهرت فحوصات SEM لمياه الشرب وجود جزيئات بلاستيكية دقيقة تراوح حجمها بين (0.5 - 4.8) نانومتر. بالإضافة إلى ذلك، أشارت نتائج مطيافية ميكرورامان (MRS) إلى وجود تباين في جسيمات البولي إيثيلين (PE)، وكان يتراوح بين (1450 – 4220) سم$^{-1}$ لعينات المياه الخام، النطاق الناشئ من اختلافات المجموعة البلورية - CH_2 لديه كثافة أكبر (1500-1200) سم$^{-1}$، من النطاق الناشئ عن اختلافات المجموعة في الطور السائل، وبين (121.404 – 4391.729) سم$^{-1}$ لعينات مياه الشرب، شدة وضع تمديد CH_2 المتماثل عند 2600 سم$^{-1}$ بالنسبة إلى وضع تمديد CH_2 غير المتماثل عند 3100 سم$^{-1}$ يبدو

أنه أعلى بالنسبة للبولي إيثيلين منخفض الكثافة (LDPE) مقارنة بالبولي إيثيلين عالي الكثافة (HDPE) لعينات مياه الشرب.

اقـــــرار المشرف

اشهد ان اعداد هذه الرسالة والموسومة بـــ (تقييم الجسيمات المايكروبلاستيكية في المياه الخام ومياه الشرب لمدينة الحلة) والمقدمة من قبل الطالب / الطالبة (سرى محمد سامي مظلوم) جرت تحت اشرافي في قسم هندسة البيئة / كلية الهندسة / جامعة بابل وهي جزء من متطلبات نيل درجة الماجستير علوم في الهندسة البيئية.

التوقيع :

المشرف : أ.د. علاء حسين الفتلاوي

التاريخ : / /2023

اشهد ان هذه الرسالة المذكورة اعلاه قد استكملت في هندسة البيئة في كلية الهندسة / جامعة بابل

التوقيع :

رئيس القسم :أ.م.د حسين علي مهدي

التاريخ : / /2023

اقرار لجنة المناقشة

نحن أعضاء لجنة المناقشة ، نشهد بأننا اطلعنا على رسالة الماجستير الموسومة (تقييم الجسيمات المايكروبلاستيكية في المياه الخام ومياه الشرب لمدينة الحلة) وقد ناقشنا الطالب / الطالبة في محتوياتها وفيما له علاقة بها ، نؤيد انها جديرة بالقبول لنيل درجة الماجستير علوم في الهندسة البيئية.

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جمهورية العراق
وزارة التعليم العالي والبحث العلمي
جامعة بابل
كلية الهندسة
قسم الهندسة البيئية

تقييم الجسيمات المايكروبلاستيكية في المياه الخام ومياه الشرب لمدينة الحلة

رسالة

مقدمة الى كلية الهندسة في جامعة بابل
كجزء من متطلبات نيل درجة الماجستير في الهندسة /الهندسة البيئية

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