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Investigation of Micro Polymer Origin

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List of abbreviations

MP: Microplastics

MPP: Microplastic particles

NPs: Nanoparticles

HD-PE: High-Density Polyethylene

LD-PE: Low-Density Polyethylene

PP: Polypropylene

PET/PETE: Polyethylene Terephthalate

PPCPS: Pharmaceuticals and Personal Care Products

PVC: Polyvinyl Chloride

POPS: Persistent Organic Pollutants

PCBs: Polychlorinated Biphenyls

MPS: Microplastic Particles

BPA: Bisphenol A

PBDES: Polybrominated Diphenyl Ethers

PAHs: Polycyclic Aromatic Hydrocarbons

TBBPA: Tetrabromobisphenol A

PS: Polystyrene

PE: Polyethylene

PES/PET: Polyethersulfone/Polyethylene Terephthalate

PMMA: Polymethyl Methacrylate

WWTPs: Wastewater Treatment Plants

OM: Organic Matter

FTIR: Fourier Transform Infrared Spectroscopy

Micro-FTIR: Micro Fourier Transform Infrared Spectroscopy

ATR-FTIR: Attenuated total reflectance Fourier transform infrared spectroscopy

SEM: Scanning Electron Microscopy

FPA-Micro –FTIR: Focal plane array Micro Fourier Transform Infrared spectroscopy

GC-MS: Gas Chromatography –Mass Spectrometry

QA/QC: Quality Assurance /Quality Control

1. Introduction

In 1941, Yarsley and Couzens published an article describing plastics, a novel material with the potential to revolutionize modern society (Thompson et al. 2009). Since then, the production and usage of plastics have increased drastically due to their low cost, ease of production, and versatility with a global plastic output estimated to be 359 and 368 million tons in 2018 and 2019, respectively, with predictions of tripling by 2050 (Perez et al. 2022; Kärkkäinen and Sillanpää 2021). The significant increase in plastic production over the years and the improper disposal of plastic waste has resulted in a considerable amount of plastic waste, leading to environmental pollution (Choi et al. 2021, Sajjad et al. 2022). Plastic waste can undergo physical, chemical, and biological degradation, resulting in small particles of less than 5mm known as microplastics and they are capable of degrading into even smaller particles, called nanoplastics.

MPs have been detected in various natural habitats, including oceans, inland waterways, soils, and even in the atmosphere, with indications of their existence in remote areas such as the Arctic and Antarctica, as well as in food (Priya et al. 2022, Šaravanja et al. 2022, Lin et al. 2022). MPs' small size and large surface area-to-volume ratio make them prone to adsorb and release pollutants, which worsens plastic pollution's impact on many species, including humans (De Falco et al. 2018, Treilles et al. 2020). Microplastics are believed to originate from primary sources, such as personal care and cosmetic products, which account for 15-31% of overall production of microplastic pollution, and secondary sources, such as the degradation of larger plastic items or synthetic textiles, which account for 70-80% of microplastics in the environment (Sun et al. 2019, Tiffin et al. 2022). Therefore, this study primarily focuses on secondary microplastics, as they account for the majority of microplastic pollution in the environment.

To mitigate their environmental and health impacts, it is crucial to understand the origin, separation, and segregation cycle of microplastics (Lamichhane et al. 2023). Although researchers have developed new methodologies for monitoring and mitigating environmental pollution caused by microplastics, uncertainties remain regarding their sources and interactions with different materials. Furthermore, To prepare microplastic samples for analysis of their origin, various techniques are required, such as density separation, sieving, and digestion of water, sediment, and living organisms samples (Lee et al., 2022). However, the effect of different chemical agents on

microplastic properties remains unclear, and some digestion treatments fail to eliminate organic matter without damaging the polymers (Savino et al., 2022). To address this gap of knowledge therefore, the first segment of this study aimed to assess the resistance of four commonly encountered microplastic polymers namely high-density polyethylene (HD-PE), low-density polyethylene (LD-PE), polypropylene (PP), and polyethylene terephthalate (PET/PETE) towards acidic and alkaline agents namely sulfuric acid (H_2SO_4), hydrochloric acid (HCl), phosphoric acid (H_3PO_4), and sodium hydroxide (NaOH) under varying reaction conditions (i.e. Temperature and Time). The results were evaluated based on two performance parameters, namely mass loss % and morphology alterations in the microplastic polymers. As textile washing is predicted to be a significant source of microplastic pollution in the future, especially in water bodies. Thus, the second segment of this study is to quantify microplastics released from commercially available synthetic clothes during washing in real-life household laundry machines. The researchers conducted washing trials using 2.5 kg loads of five different types of textile polymers commonly used namely cotton, viscose, elastane, polyamide, and polyester using a synthetic washing program to minimize the effects of the temperature and the washing process. The results were evaluated based on two performance parameters, namely the mass of the effluent filtered and the number and dimensions of microfibers released.

The objective of this research is to enhance the detection and analysis of microplastics by studying how different chemical agents affect the properties of various polymers, especially in aged and damaged particles. Additionally, the effectiveness of commonly used digestion solutions in extracting microplastics without altering the chemical characteristics of the polymer was evaluated. This provides essential insights into the sources of microplastics in the environment and the resistance of different polymers to degradation can inform us which polymer has a higher ability to contribute to the release of microplastics. This information can be used to develop effective strategies to mitigate the impact of microplastics, identify industries or products that significantly contribute to microplastic pollution, and evaluate the associated risks in the environment. The study also examined the contribution of synthetic fabric washing to microplastic pollution, quantified the amount and dimensions of microplastics released during washing, and tested a possible filtration process for their removal. The resulting data can be used to develop mitigation measures and promote the protection of the environment and human well-being.

2. Literature review

2.1Plastic pollution

In 1941, Yarsley and Couzens published an article describing a novel material called plastics, which had the potential to revolutionize modern society (Thompson et al. 2009). Synthetic plastics were first invented in 1907 and gained popularity in the 1930s (De Falco 2018). Today, plastic has become an essential component of our daily lives. Plastics are made up of large, chain-like structures of organic polymers, which are macromolecules consisting of numerous segments connected together. These synthetic organic polymers are commonly referred to as plastics in industrial settings and exhibit a moldable state, called the plastic state, at temperatures above room temperature (Oliveira et al. 2020, Zhang et al. 2021, Chen et al. 2021). Plastics have become ubiquitous due to their lightweight, strength, and resistance to corrosion and chemicals, leading to the replacement of traditional materials such as glass, metal, and paper. Plastics have been widely used to package food and water, ensuring their safety and longevity, while also playing a crucial role in technological advancements such as renewable energy, electronics, and biomedical applications. However, the primary concern with plastics is their appropriate disposal due to their persistence in the environment for extended periods. In 2018 and 2019, global plastic output was estimated to be 359 and 368 million tons, respectively, with this number expected to increase in the future (Kärkkäinen and Sillanpää 2021). By 2050, this production is predicted to triple (Perez et al. 2022). These materials are popular due to their lightweight, flexibility, thermal and electrical insulation, corrosion resistance, and low cost (Zhang et al. 2021). Nonetheless, plastic pollution from these materials has led to the visible buildup of micro plastic particles in various environments, including marine and freshwater, sea ice, sediments, soil, and the atmosphere.

Plastic pollution is a persistent environmental issue, attributed to the large-scale production of plastics that remain intact for extended periods, leading to the contamination of water bodies. The degradation of plastics into micro plastics, particles smaller than 5 μm, or even smaller nano plastics, particles less than 1 μm, further exacerbates this issue. While polymers and plastics are often used interchangeably, it is important to distinguish between them. Polymers are large molecules composed of monomers, with a variety of structures, including linear, branched, or crosslinked. In contrast, plastics are a combination of two polymers or a polymer and low-molecular-weight substances, such as pigments, stabilizers, flame retardants, dyes, or fillers, depending on their intended use. The pervasiveness of micro plastics is not limited to the

environment, as they have been found in a variety of foods, including seafood, honey, salt, beer, sugar, and drinking water (Priya et al. 2022, Sing et al. 2021). To address plastic pollution, several strategies, including reducing plastic consumption, improving waste management and recycling practices, and developing innovative methods to remove plastic waste from the environment, are being implemented. However, the non-biodegradable nature of plastics makes plastic contamination an irreversible problem, and once micro plastic particles are released into water sources, they are challenging to recover (Šaravanja et al.2022).

2.2Microplastic

Microplastics exhibit variable size ranges and definitions across studies. While most studies define microplastics as particles smaller than 5mm, some suggest an upper limit of 10mm, with particles larger than 1mm classified as macro or mesoplastic. Microplastics are categorized into small microplastics (0.33–1μm) and large microplastics (1.0–4.75μm), with the lower limit being undefined and varying based on sample collection and processing methods. To be more inclusive, it is commonly defined that microplastics are plastic particles smaller than 5mm in their smallest dimension (Jiang et al. 2020, Acharya et al. 2021). The potential harm of microplastics to human health, biodiversity, and water security has led to a significant increase in research on microplastics (Jenkins et al. 2022). The strong intermolecular bonds, hydrophobic characteristics, and functional groups of plastics and microplastics make them difficult to break down in the environment. The large surface area of microplastics increases their attraction to other compounds, leading to further degradation, and they also have the potential to accumulate harmful substances, posing significant risks to organisms in the food chain. While scientists are working on developing new methods to identify and measure microplastics in different environments, the lack of an accurate description of microplastics has made it challenging to compare findings across research. Standardization of sampling and analysis methods is required to enable better comparison of microplastic concentrations and effectively address the issue. Although current methods for measuring and analyzing microplastics have limitations in speed and detection, researchers are working to improve these methods to minimize the negative impacts of microplastics on both humans and aquatic wildlife (Park and Park 2021).

2.2.1 Type of microplastic

Two types of microplastics can be identified: primary and secondary. Primary microplastics are directly produced and used in personal care and cosmetic products (PPCPs) (Khalid Ageel et al. 2022) . Secondary microplastics are formed from the degradation of larger plastic waste due to abiotic and biotic environmental factors. (Sun et al. 2019, Singh et al.2021, Zhang et al. 2021). The combined total of primary and secondary microplastics amounts to approximately 8.3 million tonnes per year in the context of global plastic pollution. Household and commercial activities contribute 3.2 million tonnes of primary microplastics annually, with 1.5 million tonnes of this amount ending up in the ocean. On average, this results in the release of around 400 grams of primary microplastics per person each year, which is equivalent to 80 plastic grocery bags, with half of them ultimately reaching the ocean (Eerkes-Medrano et al.2019). In Europe, it is estimated that between 307 and 925 million litter items enter the ocean each year, with plastics accounting for 82% of them. Approximately 176,000 tonnes of microplastics unintentionally enter European surface waters annually due to the weathering and abrasion of plastic products, and an additional 42,000 tonnes of intentionally added microplastics are discharged into the environment through products such as artificial turf pitches, cosmetics, detergents, and fertilizers (Zhang et al. 2021).

2.2.2 Abiotic and biotic degradation of microplastic

The physical or chemical changes in plastics caused by external factors such as light, temperature, air, water, and mechanical forces are referred to as abiotic degradation. Given their limited biodegradability, abiotic degradation is typically the first step in the decomposition of plastics in the environment. The degradation of plastics can occur via various processes, including photo degradation, thermal degradation, and mechanical degradation (Oliveira et al. 2020). Photo degradation, which is initiated by sunlight, is the most significant process and involves free radical-mediated reactions. Thermal degradation, on the other hand, is caused by exposure to high temperatures, leading to thermo-oxidative reactions. Finally, mechanical degradation, also known as mechanical deterioration, results from external forces such as plastics rubbing against rocks and sand from wind and waves or the freezing and thawing of plastics in aquatic conditions. Biotic degradation of plastics refers to the deterioration of plastics by living organisms, either through physical means such as biting, chewing, or digestive fragmentation, or through biological processes such as biochemical reactions (Singh et.al 2021, Zhang et al. 2021). General processes of plastic degradation are illustrated in Figure 1.

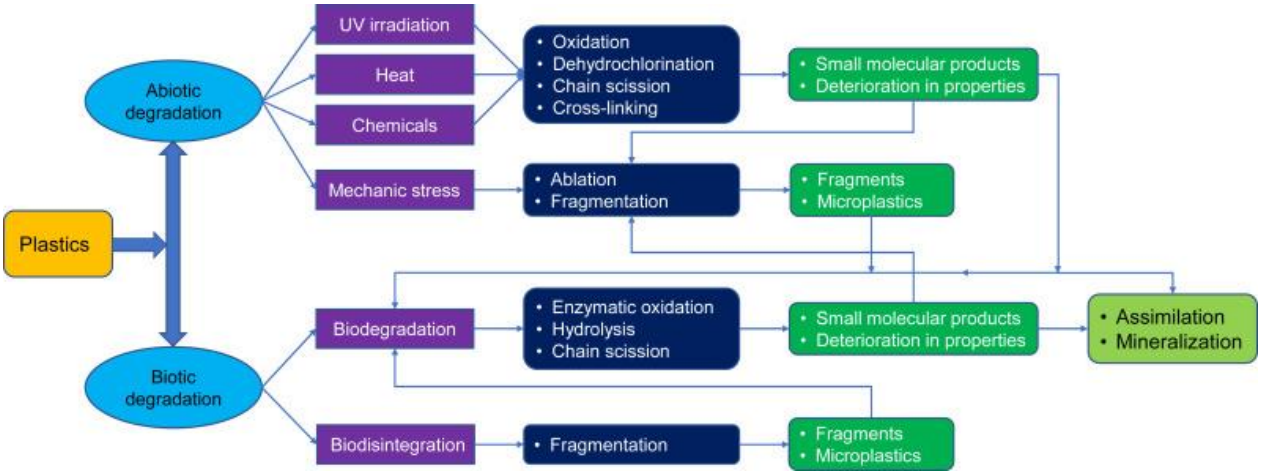


Figure 1 General processes of plastic degradation (Zhang et al. 2021)

2.2.3 Microplastic composition

Microplastics consist of a mixture of substances from both natural and human-made sources, forming a "cocktail of contaminants" (Reimonn et al. 2019). The "cocktail of contaminants" is a useful concept for identifying the potential hazards associated with microplastics, as it highlights that plastic is not just composed of its polymeric constituents, but also contains additives, adsorbed chemical pollutants, and manufacturing byproducts within its matrix Figure 2. It is essential to consider all additional contaminants when assessing the environmental impact of microplastics, as plastic is classified based on its main polymer. Polymeric base substances are the primary materials used to make products such as polyethylene, polycarbonate, or polyvinyl chloride, with some materials like the monomer of PVC, vinyl chloride, being dangerous. They often contain additional chemical additives like phthalates, polybrominated diphenyl ethers (PBDEs), and tetrabromobisphenol A (TBBPA) which can leach out of the plastics after they are released into the environment (Rogers, Kara, 2022). Additionally, plastic particles that have undergone chemical degradation can still be pollutants even after disposal. Production byproducts, such as initiators, catalysts, surfactants, organic solvents, and suspension acids, may also be unintentionally present in the final plastic product. Chemical additives, such as those added to enhance polymer quality, are the most dangerous subset of plastic pollutants as they can leach out of the plastic and

pollute the environment. Because of this, scientists are now using new terms like "microlitter" or "anthropogenic microlitter" to describe all the tiny, harmful materials found in samples, whether they are made of natural or synthetic materials (Acharya et al. 2021).

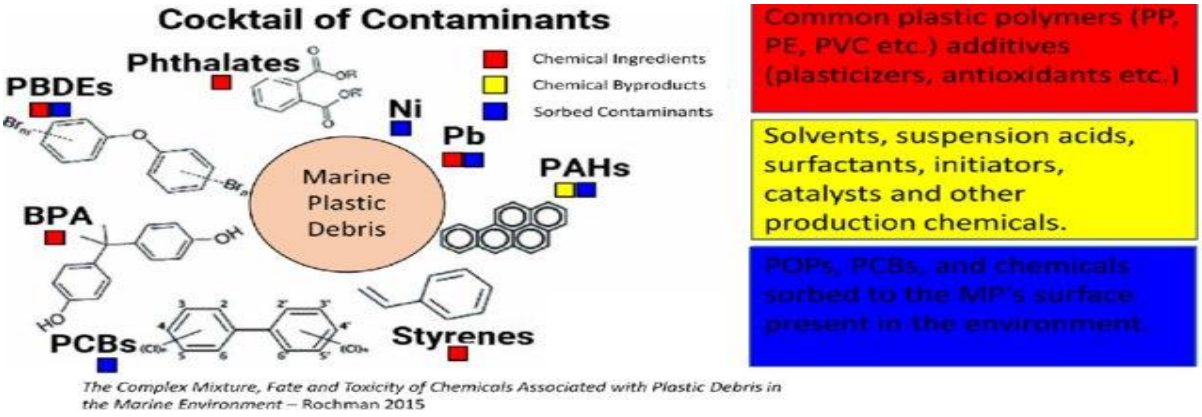


Figure 2 Micro plastic composition (Reimonn et al. 2019)

2.2.4 Classification of microplastic

Microplastics found in water or sediment can vary in size, shape, and color depending on the sampling strategies and processing of the sample (Cverenkárová et al. 2021). Microplastic is classified based on its physical and chemical characteristics, which can impact its distribution and impact which affect their aggregation, migration, and degradation. Physical characteristics, such as size, shape, density, color, and crystallinity, play a significant role in the potential harm caused by microplastics. For example, smaller microplastics have a higher chance of releasing additives and chemicals and attracting contaminants, while the shape of microplastics can impact their interaction with pollutants and microorganisms (Lozano et al. 2021). Microplastics exhibit a diverse range of shapes and are commonly categorized into two main groups - fibers and particles. Particles are further divided into subcategories such as spheres and pellets. Additionally, some studies have classified microplastics into other subcategories such as flake/films, foam, and chips (Priya et al. 2022) Figure 3. A review of 24 studies indicated that the most commonly found shapes of microplastics include fibers, pieces, films, microbeads, fragments, and foams. Films were found to be the most prevalent shape, followed by fibers and fragments. Fibers were primarily found in agricultural and farm soils, while fragments were concentrated in mulch and farming soils. In

wastewater, fibers made up the highest percentage of microplastics, followed by regular pieces (Sun et al. 2019; Dai et al. 2022). Additionally, the density and color of microplastics can provide valuable data on the type of polymer, level of pollution, and degree of weathering (Chen et al. 2021, Cverenkárová et al. 2021). Crystallinity, or the proportion of crystalline regions in a polymer, can impact the mechanical characteristics of microplastics and evolve over time due to polymer degradation or reordering. Understanding these characteristics can inform strategies to mitigate the harmful effects of microplastics on the environment and living organisms. Chemical characteristics, such as composition and surface groups, play a crucial role in determining the properties of microplastics. Furthermore, the physical properties of the polymers, including porosity, molecular size, and degree of degradation, can influence the rate of chemical release from microplastics (Chen et al. 2021)

Figure 3 shapes of micro plastic and their sources (Priya et al. 2022)

2.2.5 Type of polymers

The composition of polymers in micro plastics is crucial for identifying their sources of pollution. Microplastics comprise a heterogeneous group of polymer particles that can be classified into seven main categories: polyvinyl chlorides (PC), polyethenes (including low density [LDPE] and high density [HDPE]), polyamides (PA), polypropylenes (PP), polyurethanes (PU), polystyrenes (PS),

and polyethylene terephthalates (PET) (Hamidian et al. 2021). However, the European community recognizes over 130 different polymers as components of microplastics. The most commonly used plastic materials worldwide include PP (23%, used for packaging, food containers, and textiles), PE (17% LDPE, 15% HDPE, used for plastic bags, packaging, and microbeads), PS (7%, used for packaging), PET (7%, used for plastic bottles and synthetic fibers), and PA (1%, used for fibers such as nylon). Additionally, there are poly(methyl)methacrylate (PPMA, 1%, used for synthetic glass) and polycarbonates (1%, used for plastic bottles and synthetic glass) see Figure 4 (Habib, Thiemann, and Kendi 2020, Oliveira et al. 2020, Hardin 2021). The physicochemical and degradation characteristics of each polymer type affect the formation, distribution, and aggregation of MPs in environments.

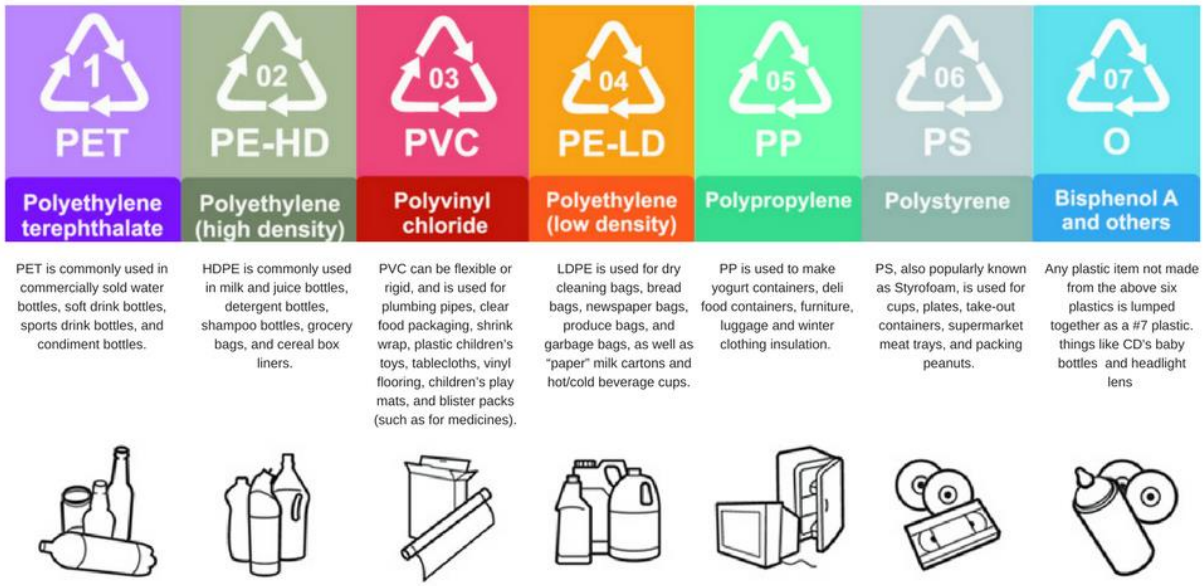


Figure 4 List of common plastics and their uses (Hardin 2021)

2.2.6 Microplastic presence in seawater systems

Microplastics are widely distributed in seawater and their regional distribution spans across the Atlantic, Midwest Pacific, Italy, and Tunisia in the Mediterranean, the Baltic Sea, the Gulf of Oman in the Arabian Sea, Indonesia Bali in the Indian Ocean, South China Sea, Korea Coasts, Nordic Sea in the Arctic Ocean, and Antarctic Ocean Figure 5 (Kye et al. 2023). The distribution and prevalence of microplastics are influenced by various anthropogenic and environmental factors. Human sources of microplastics include plastic waste from both land-based and marine industries, while environmental factors include geological characteristics, distance from land, and ocean

currents (Oliveira et al. 2020). Reports of plastic waste in marine systems date back to the 1960s, with most of it believed to come from inland sources that are transported by rivers (Acharya et al. 2021). Recent studies indicate that 44% of plastic waste is present in rivers, oceans, and along shorelines (Jiang et al., 2020; Rogers, Kara, 2022). Microplastic (MPs) fragments have been identified in various ecological sectors and pose a significant problem. Research studies indicate that MPs have been found from the highest peak to the depths of the ocean. The majority of MPs (80%) are sourced from land, while less than 20% are from water sources (Lamichhane et al. 2023).

Marine litter refers to any solid waste that ends up in the ocean, including materials from rivers, drains, and sewage. The improper disposal of plastic waste contributes significantly to the millions of tons of garbage that end up in oceans each year. In 2014, a study reported that there were at least 5.25 trillion individual plastic particles weighing approximately 244,000 metric tons floating on or near the surface of oceans. The Mediterranean Sea has been estimated to have 5-10% of the plastic pollution in the world's oceans (Castelvetro et al., 2021). Each year, billions of tons of waste end up in the ocean, with about 80% coming from land. Plastic waste is particularly problematic because it doesn't break down easily and can gather in certain areas due to waves and currents. Typically, microplastic concentrations are higher nearshore or in estuaries adjacent to land than in open sea regions (Kye et al. 2023). Physical forces like wind-driven currents, geostrophic circulation, and turbulent flows from tides or waves play a crucial role in the transport and dispersal of microplastics at various spatial scales. Additionally, ocean currents, winds, weather changes, and other environmental factors play a significant role in the transport, dispersion, and deposition of microplastics in marine environments.

Microplastics of different shapes and compositions have been detected in seawater due to the ocean's role as the ultimate receiver of water flow. The composition and shapes of microplastics in seawater vary depending on the regional characteristics, industries, and the surrounding environment. Studies have shown spatial associations between the types of microplastics found and human activities (Eerkes-Medrano, Thompson, and Aldridge 2015). The most common form of plastic waste in seawater is fragmented plastics, which are affected by ultraviolet light and mechanical forces from wind and waves. Pellet-type microplastics are present around Hong Kong

Island and have specific shapes influenced by various industries. Foam or bead-type plastics serve specific purposes such as floating mariculture facilities or exfoliating scrubs, while microplastics in film form are caused by fragmented plastic bags or vinyl used in agriculture. Fibers mainly originate from fishing nets and ropes or laundry washing. The prevalent plastic polymers identified in seawater are polyethylene (PE), polypropylene (PP), and polystyrene (PS), which are commonly used in marine industries and daily life (Kye et al. 2023).

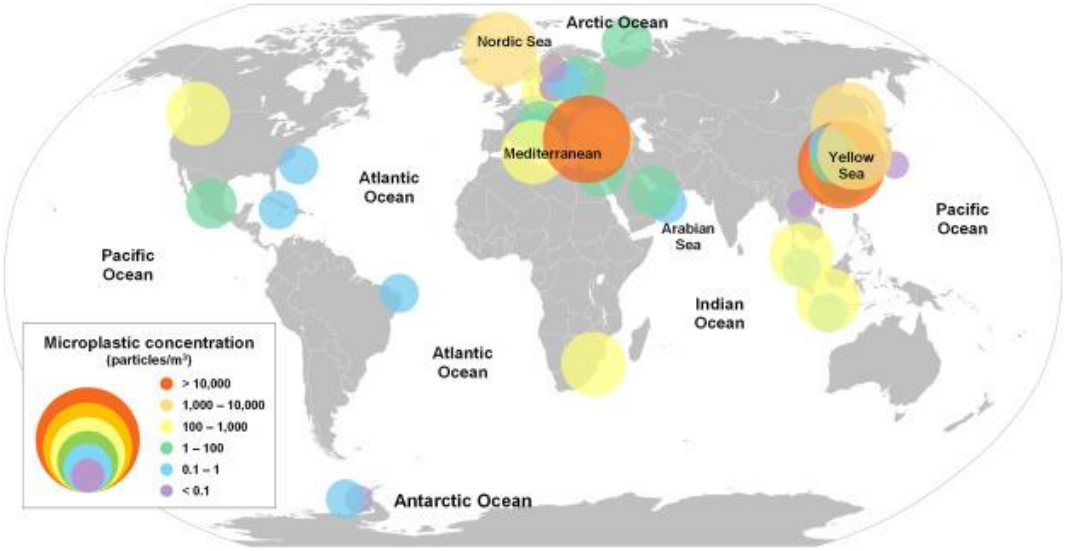


Figure 5 The map depicts the global distribution of microplastics in the oceans based on studies. (Kye et al. 2023)

2.2.7 Microplastic presence in freshwater systems

The presence and distribution of microplastics in freshwater systems are strongly influenced by the surrounding environment, with anthropogenic activities, hydrodynamic forces, and wastewater playing a significant role in the distribution of microplastics in freshwater bodies. Numerous studies have identified these factors as the primary drivers of microplastic pollution in freshwater systems (Kye et al. 2023). There are three potential ways in which microplastics enter freshwater systems, such as wastewater treatment discharge, agricultural runoff from land treated with sludge, and overflow of sewage water due to heavy rainfall (Sarijan et al. 2021). Recent studies have shown that microplastics are present in freshwater systems across different continents and are of various origins and compositions. Higher counts of pelagic microplastics were observed in Lake Erie due to its higher population density, while Lakes Huron and Superior had lower counts due to their lower population densities and larger sizes. However, even remote areas with low population

densities, such as Lake Hovsgol, had high microplastic densities (Eerkes-Medrano, Thompson, and Aldridge 2015). Freshwater ecosystems are known to be sensitive to their surroundings, resulting in the discovery of a substantial quantity of fiber-type PES and nylon microplastics in freshwater located close to urban regions. Moreover, freshwater has also been found to contain microplastics made up of polymers such as PE, PP, and PS, which are commonly employed.

2.2.8 Microplastic present in wastewater

Wastewater treatment plants (WWTPs) play a significant role in the transfer of microplastics from human activity to the natural environment. The concentration of microplastics in wastewater is subject to considerable variation due to study design, including sampling, pretreatment, and analytical techniques, as well as complex variables such as catchment areas, population served, nearby land use, and combined sewer systems, with different sources of wastewater contributing to the variability (Joana et al., 2018).

The majority of microplastics in WWTPs are derived from various sources, including home sewage, industrial wastewater, rainwater, and surface runoff from the land application of sewage-based fertilizer (Hanvey et al. 2017), with the primary and secondary sources leading to the yearly addition of hundreds of tons of plastic to the agricultural ecosystem from wastewater treatment plants in Europe and North America (Jiang et al., 2020). The presence of microplastics has been confirmed in sewage samples worldwide, with recent studies confirming the presence of microplastics in sewage samples from various countries worldwide, including Russia, Sweden, France, Finland, the United States, the United Kingdom, the Netherlands, Germany, Canada, Australia, Italy, Turkey, Denmark, Poland, China, and South Korea (U. Iyare, K. Ouki, and Bond 2020). Microplastics can also enter the freshwater when this wastewater is mixed with freshwater sources further, contributing to the production of microplastics in the water they transport and contaminating soil and groundwater through percolation. Moreover, since groundwater is often considered safe for drinking and is not treated, any microplastics present cannot be removed, increasing the risk of human consumption (Singh et al.2021, Tiffin et al. 2021).

Microplastics in wastewater vary widely in composition, shape, and concentration, and their presence in wastewater is a direct indication of human activities, and their abundance can be correlated with various urban-related watershed features and populations (Kye et al. 2023), with fiber-type microplastics being the most prevalent, followed by fragments and film types see Figure

6. The polymeric composition of microplastics in wastewater depends on the sampling location and environment (Hamidian et al. 2021), with PET, PP, and PE being commonly found (Kye et al. 2023). Wastewater treatment can eliminate a significant amount of microplastics; however, low concentrations that persist should not be disregarded due to the presence of other microplastic sources, including marine industrial waste and plastic litter. Microplastics can adsorb harmful substances, such as pharmaceuticals and pathogenic organisms, leading to further environmental contamination (Joana et al., 2018; Jiang et al., 2020). Therefore, an accurate analytical method for identifying and quantifying microplastics is crucial to understanding their prevalence in wastewater (Dyachenko et al., 2017).

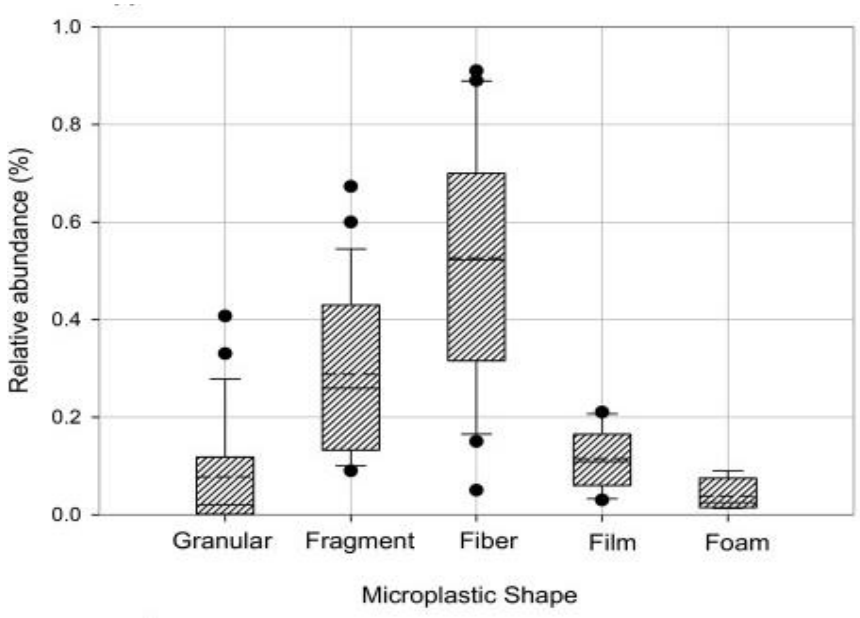


Figure 6 Shows the relative abundances of the various micro plastic forms found in WWTPs. (Sun et al. 2019)

2.3 Effects of microplastic

Microplastics pose a significant environmental and ecological threat due to their non-biodegradable nature and multiple pathways for harmful effects. Microplastics can be transported through surface runoff and wind, allowing them to move between different environmental compartments. Therefore, Microplastics are widely distributed in various environments, including oceans, freshwater ecosystems, and the atmosphere (Jiang et al. 2020, Zhang et al. 2021). As a result, microplastic waste has become the second-biggest environmental and ecological concern (Rogers, Kara, 2022). Their toxicity can be attributed to the polymer compounds used to manufacture them,

such as polystyrene, antibiotics, polycyclic aromatic hydrocarbons (PAHs), plasticizers, and potentially toxic elements (PTEs) (Sajjad et al. 2022). Additionally, MPs may contain heavy metals like cadmium, zinc, and lead, leading to detrimental effects (Lamichhane et al. 2023). These compounds can leak out of the plastic and accumulate in biological tissues, potentially endangering human health, agricultural safety, and soil organisms (Perez et al. 2022, Zhang and Chen 2020).

2.3.1 Effects of micro plastic pollution on water

The presence of microplastics (MPs) in the marine environment has become a significant scientific concern in recent years. Marine organisms, including fish, mussels, zooplankton, and sea birds, ingest MPs (Oliveira et al. 2020). The ingestion of microplastics by marine organisms is influenced by various factors such as their size, shape, color, and polymer type. Microplastics colored similar to food are easily mistaken for prey and ingested by fish. Microplastics in the form of fibers are commonly ingested by fish, while fragments are mainly found in bivalves such as oysters and mussels (Kye et al. 2023). Also, due to the presence of nutrients, microorganisms are drawn to plastic particles, and their biofilm development transforms the surface of microplastics, making them more easily ingestible by aquatic organisms (Cverenkárová et al. 2021). The small size and pointed ends of microplastics make them hazardous to organisms, which can cause a range of adverse effects, such as inflammation, lipid accumulation, and size-dependent toxicity (Sun et al. 2019). In 2018, the presence of microplastics in the tissues and digestive systems of over 114 aquatic species was reported, resulting in decreased food consumption, reduced energy for basic life activities, and neurological and reproductive damage (De Falco 2018).

2.3.2 Effects of microplastic pollution on soil

Plastic pollution has a significant impact on terrestrial ecosystems, with agricultural soil systems serving as major reservoirs for microplastics. These microplastics can enter soil through various sources, including compost and sewage sludge, landfills, air deposition, and irrigation with untreated water, flooding, littering, and road runoff (Chen et al. 2021, Lozano et al. 2021, Priya et al. 2022, Perez et al. 2022). Furthermore, the movement of microplastics in soils is affected by soil characteristics, such as ionic strength and cation type, as well as heterogeneity (Tian et al. 2022). Plastic waste, such as bags, films, and debris, can obstruct drainage pipes, leading to flooding, and has also been found in the stomachs of land birds and animals. MPs can move deep into the soil,

polluting underground water (Sajjad et al. 2022). The widespread disposal of plastic waste in landfills is a significant global environmental concern, particularly in agricultural soils, which are crucial for food production. Microplastics in these soils pose a danger to human health and food security and cannot be disregarded (Chen et al. 2021, Kärkkäinen and Sillanpää 2021). Microplastics are considered to pose a significant threat to terrestrial ecosystems, where soil may serve as a more extensive plastic reservoir than the oceans (He et al. 2018).

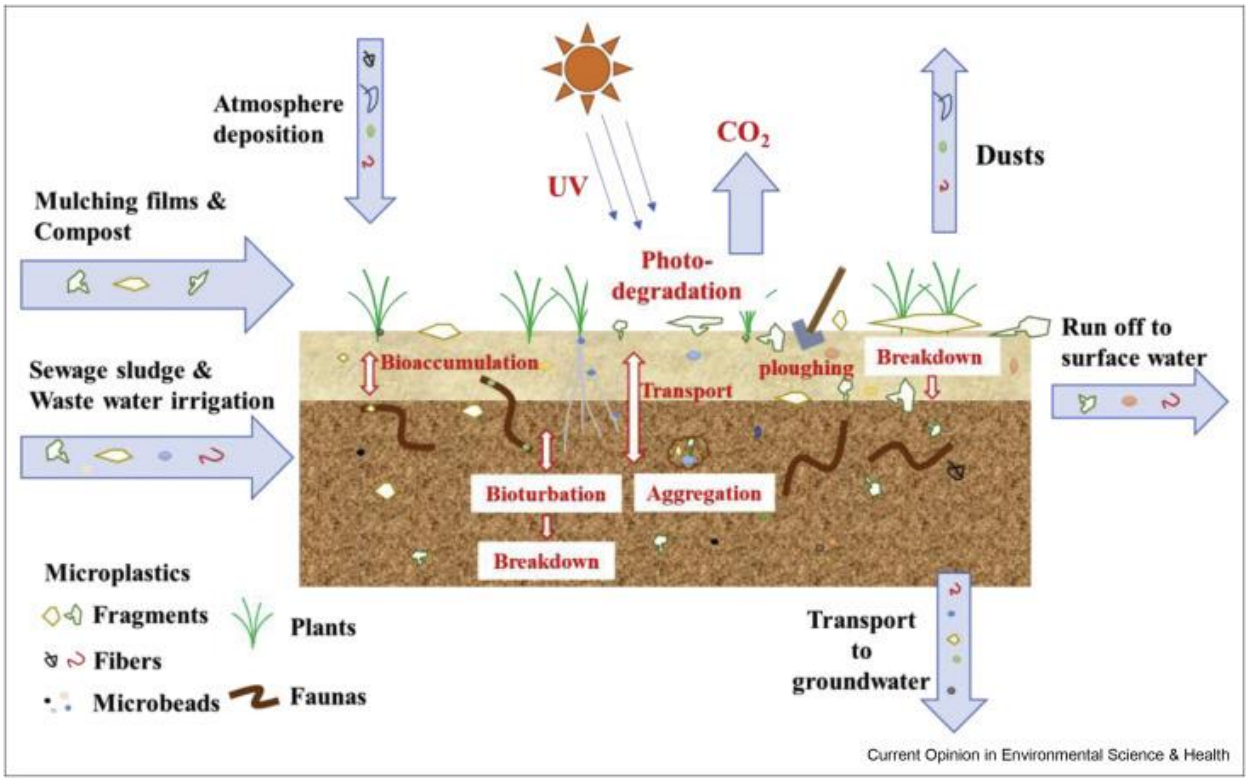


Figure 7 Sources, effects, and fate of microplastics in agricultural soil systems (Tian et al. 2022)

2.3.3 Effects of microplastic pollution on Human

MPs enter rivers, lakes, and oceans via domestic and commercial effluents and eventually accumulate in seafood and table salt, posing a severe risk to human health. Recent scientific research has demonstrated that microplastics (MPs) can enter the human body via ingestion, inhalation, and dermal contact. Smaller MP particles are particularly concerning as they can accumulate in organs such as the brain, liver, and kidney, thus posing a threat to human health (Silva et al. 2022). The utilization of Fourier Transform Infrared spectroscopy (FTIR) to examine human feces has provided indications of micro-sized plastic particles being excreted via the

gastrointestinal tract. Furthermore, FTIR techniques have detected plastic particles in human colectomy specimens, while Raman microspectroscopy has identified three polypropylene particles between 5 and 10 μm in human placental tissue(Leslie et al. 2022). It is important to note that children and infants are more susceptible to MP exposure than adults (Silva et al. 2022).

2.4 Microplastic analysis

Microplastics are a complex mixture of many types of polymers, which can have varying densities, sizes, and shapes, which makes it hard to identify all types of microplastics from complex environmental matrices using just one method. Therefore, scientists often use multiple methods. Each method has its own advantages and disadvantages, and the choice of methods can vary depending on the study. This highlights the importance of selecting appropriate sample collection and extraction techniques in order to accurately and effectively study microplastics (Shim et al. 2017, Rochman et.al 2017, Priya et al. 2022). The prompt discusses the increasing research focus on the rapid separation and characterization of primary and secondary microplastics (MPs) in aquatic and terrestrial environments. To identify and quantify MPs, various visual and analytical tools have been employed in many studies. Physical characterization through microscopy is often followed by chemical characterization through spectroscopy to confirm the plastic. Before identification, density separation, filtration, sieving, and visual sorting are necessary. These initial techniques allow for the identification of larger MP fragments' morphology, including their shape, size, and color (Lamichhane et al. 2023).

2.4.1 Analyzing process

Analyzing microplastics involves three key components: sampling, extraction, and quantification. Each step of this process is made difficult by the diversity of polymers in terms of type, size, color, and shape, as well as the lack of homogeneity within environmental samples (Hanvey et al. 2017, Park and Park 2021).

Sampling

Obtaining appropriate samples is critical for detecting microplastics in the environment, and the selection of sampling methods depends on the type of microplastics being examined, which can be present in water, sediment, soil, cosmetics, or living organisms. Several factors, such as the density, shape, characteristics, water velocity, and depth of the microplastics, may influence the sampling process (Koelmans et al. 2019).

Extraction

The second step in the microplastic analysis process is extraction, which involves physical separation and density separation, filtration techniques, and matrix removal. Physical separation is essential to isolate microplastics of different sizes, types, shapes, and colors from a complex sample matrix. On-site sieving is a commonly used physical separation method, but it is not effective for isolating small microplastics (<1 mm) (Hanvey et al. 2017). Density separation with filtering is the most widely used method for removing microplastics (1 mm) from sediment samples. Filtration techniques, including sieving and vacuum filtration, are frequently used in dry and wet sorting, respectively. To aid in the accurate and efficient isolation of microplastics from their matrix, matrix removal processes, such as chemical digestion, can be used to remove any organic materials that may interfere with the sample. Physical and chemical matrix removal methods have been shown to improve the effectiveness of filtering and measurement (Hanvey et al., 2017). Overall, extraction efficiency depends on factors such as particle size, shape, and polymer origin (Löder and Gerdts 2015).

Quantification and identification

Microplastics are counted to determine their regional and temporal distribution, accumulation rates, contaminants present in them, and their effects on aquatic life. Microplastic concentrations are typically measured in particles per square meter, cubic meter, or kilograms of dry sediment, and are counted under a microscope (Priya et al. 2022).

> **Manual counting**: In scientific terms, manual counting using an optical microscope (OM) is a widely employed method to identify and quantify microplastics in environmental samples (Jung et al. 2021). Visual identification of microplastics (MPs) is a widely used technique that employs either the naked eye or an optical microscope with 10-50 times magnification objectives. Image-analysis software may be used in conjunction with the microscope (Lamichhane et al. 2023). This technique involves visually sorting and counting microplastics based on their size, shape, color, and polymer type by magnifying images of sub-millimeter microplastics and analyzing specific surface textures and structural data. However, difficulties arise when attempting to identify small, shapeless, or colorless microplastics, leading to potential overestimation or underestimation of plastic content in samples (Löder and Gerdts 2015). Furthermore, there is a risk of mistaking non-plastic

particles as microplastics, making it challenging to obtain accurate results (Hanvey et al. 2017).

Polymer identification (FTIR; Raman; scanning electron microscope): Polymer identification methods such as Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR) including micro-FTIR and ATR-FTIR, and scanning electron microscopy (SEM) is used to classify microplastics from environmental samples and confirm that particles smaller than 1mm are plastics and identify the polymer used. These methods allow identifying the particles by their color, shape, morphology, chemical composition, and structure, which are important in identifying different types of environmental plastic contamination (Hanvey et al. 2017, Sun et al. 2019, Reimonn et al. 2019, Park and Park 2021).

- **Fourier transform infrared spectroscopy (FTIR)** is a widely used non-destructive method for identifying microplastics in water and sediment samples. FTIR offers several advantages, including the ability to obtain high-resolution data, scan large numbers of samples quickly, and at a relatively low cost. However, it may have limitations in accurately analyzing particles smaller than 10 micrometers. FTIR uses infrared radiation to create a spectrum that can identify the composition of microplastics by comparing it to a reference library. However, real-world samples may differ from the ideal samples present in these libraries, and therefore, it is necessary to create a reference library from various sources. Additionally, FTIR analysis is labor-intensive and requires the selection of microplastics using light microscopy before analysis. FPA-micro-FTIR imaging has been developed to make the process more efficient, but it has some limitations.
- **Raman spectroscopy** is another method used to identify microplastics, which uses light scattering to analyze a material's molecular vibrations and create a spectrum for identification. It offers a relatively high identification rate, but it is sensitive to fluorescence interference and requires proper sample purification before analysis. Custom protocols may also need to be developed.
- **Scanning electron microscopy (SEM)** provides detailed information on a particle's size, shape, and surface features by scanning its surface with a beam

of electrons. SEM can also be combined with other techniques to identify the elements in polymers and measure their concentration. SEM is particularly useful for identifying inorganic plastic additives.

Emerging techniques Pyrolysis gas chromatography/mass spectrometry (GC/MS)

In recent times, Pyrolysis gas chromatography/mass spectrometry (GC/MS) has emerged as a technique for identifying the polymers and additives present in microplastics and quantifying the organic compounds in the mixture (Hanvey et al. 2017). Pyrolysis is a technique that involves burning a sample in the absence of oxygen, typically utilized in thermogravimetric analysis. It provides a specific signature for certain polymers by studying the degradation of the polymer in relation to temperature. Despite its advantages, this technique is not widely used as compared to Fourier-transform infrared spectroscopy (FTIR) due to the limitations associated with further examination of the samples and the complexity of the data generated, which can be challenging to interpret (Lamichhane et al. 2023)

QA/QC In the analysis of microplastics in environmental samples, accurate determination of their presence and amount is a complex task due to potential contamination from various sources. Fibers, which can hover in the air, are especially prone to contamination and can complicate microplastic analysis (Löder and Gerdts 2015). To ensure reliable results and prevent overestimation or underestimation of plastic content, appropriate research methods and quality control measures must be employed. One such approach involves performing validation studies and using blank controls during analysis to check for contamination from sources such as air, equipment, or clothing of laboratory personnel (Joana C. Prata et al., 2021). Contamination can be minimized by cleaning equipment, using natural fabric lab coats, avoiding plastic materials, and covering samples with aluminum foil or Petri dishes to reduce airborne plastic contamination. Additionally, the recovery of microplastics during extraction methods should be evaluated to prevent sample loss. Despite the importance of these measures, many studies in the field have not effectively implemented them, as highlighted in a 2019 review of 50 publications (Sun et al., 2019).

2.4.2 Factors affecting microplastic analysis

The factors affecting the abundance and distribution of microplastics in water environments can be classified into two types: inherent properties of microplastics such as size, hydrophobicity, and specific gravity, and environmental factors such as industrial facilities near water systems, biological interactions, and meteorological phenomena see Figure 8. Exposure to UV rays, waves, and wind in the environment results in the degradation of plastic waste into smaller microplastics that can spread globally through atmospheric transport. Low-density microplastics can be deposited in the environment and resuspended by weather conditions, while human activities can also introduce plastic waste into the water system due to poor waste management practices. The variability of microplastics is influenced by both vertical and horizontal distribution, which is determined by specific gravity, biological interactions, and environmental factors. Weather conditions should be considered when studying microplastics in water environments, and long-term monitoring is necessary for accurate analysis. Mesh size variability and differences in sampling and pre-processing methods present challenges in the interpretation of microplastic analysis results. Establishing standardized protocols for microplastic sampling and pretreatment methods is a top priority, as different digestion reagents and density separation solutions can affect specific polymers in various ways. It is recommended to develop optimal protocols for each polymer analyzed to ensure accurate microplastic analysis results (Kye et al. 2023).

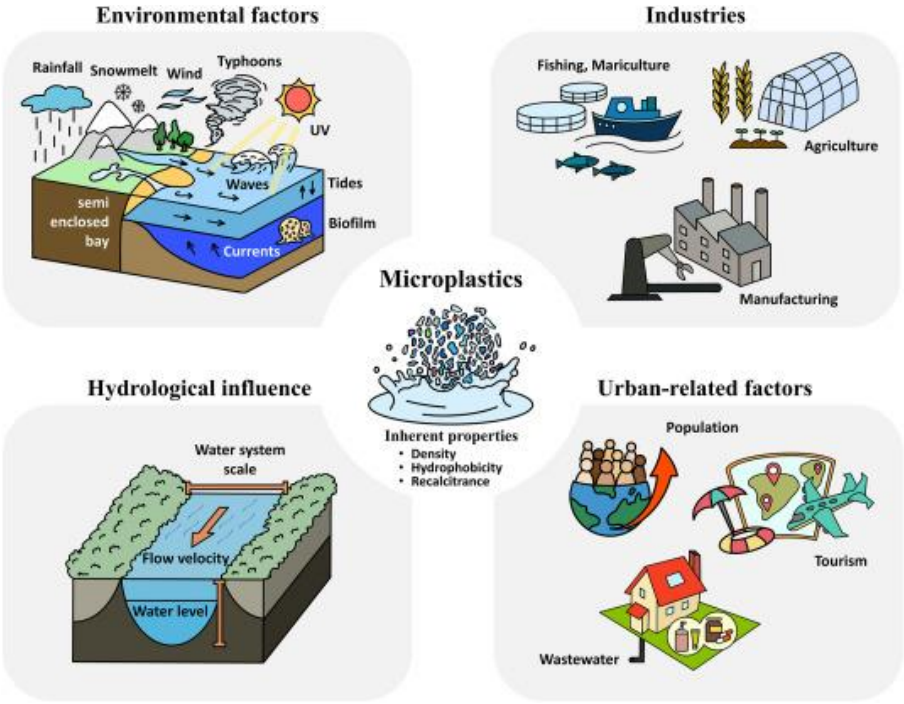


Figure 8 Factors affecting microplastic analysis (Kye et al. 2023).

3. Methods of the study

3.1 Experiment One: Assessing the resistance of microplastic polymers to various acidic and basic digestion treatments and investigating the effect of different digestion solutions on microplastic polymers.

3.1.1 Design of the Experiment

A systematic methodology comprising a series of controlled experiments is used for the scientific evaluation of the resistance of synthetic plastic polymers to various solutions, concentrations, and reaction temperatures. The objective of these experiments is to assess the performance of different types of polymers under varying conditions and determine their susceptibility to chemical degradation and thermal breakdown. To ensure reliability and consistency, the lab work is conducted under precisely controlled conditions with all variables accurately measured and recorded. The polymers are exposed to the test conditions for a predetermined duration, and their weight and microscopic images are evaluated for any changes. Throughout the experimental process, detailed notes are taken, documenting the testing conditions, outcomes, and any unanticipated findings.

For this investigation, experiments were performed utilizing acidic solutions (2M HCl, 2% H_2SO_4, and 10% H_3PO_4) and a basic solution (10M NaOH) to determine the effect of these solutions on the polymers. The digestion procedures were conducted at two distinct temperature levels (23°C or room temperature, and 60°C) and with varying reaction durations (24 hours for room temperature samples and 1 hour for 60°C samples). A total of 32 samples were prepared from four different types of polymers (PET/PETE, HD-PE, PE-LD, and PP), and the weight differences of the particles before and after treatment were used to assess their resistance. Furthermore, the extent of damage caused by the treatment was examined using a microscope to obtain a comprehensive evaluation of the behavior of synthetic plastic polymers under different test conditions.

3.1.2 Materials

- Plastic products of different shapes and polymers were selected by common plastic items as listed in Table 1. Polyethylene terephthalate (PET/PETE), high-density polyethylene (HD-PE), low-density polyethylene (PE-LD), and polypropylene (PP), with small sizes were prepared by the following steps. First, the polymers were cut in the laboratory into

small particles using disinfected scissors to produce small polymer-based MPs. second, the MPs were stored in a sealed bag as shows in Figure 9.

Table 1 plastic products and there polymer types

Polymer type	Sample type	ID
PET/PETE	Mineral water (spar)	1a
HD-PE	Yoghurt bottle (Jogobella)	2
PE-LD	Toilet paper package (Alouette,rossman)	4a
PP	Tea biscuit packaging (CBA piros)	5a

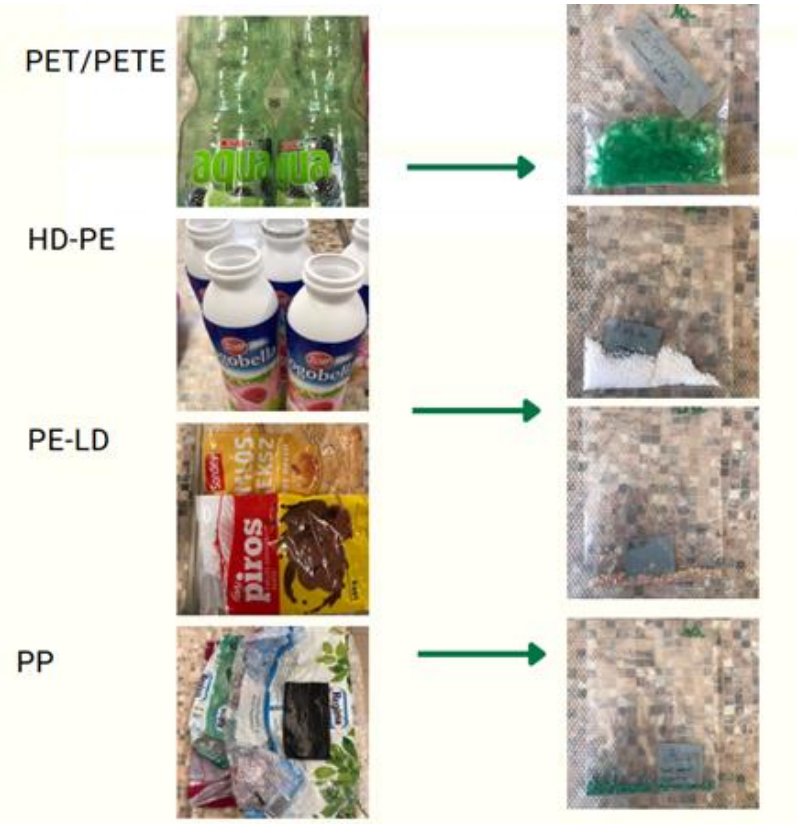


Figure 9 The plastic items pre- and post-cutting.

- Table 2 shows the Four organic matter digestion solutions were used and there concentration: hydrochloric acid (HCL) 2M, sulfuric acid (H_2SO_4) 2%, sodium hydroxide (NaOH) 10M, and phosphoric acid (H_3PO_4) 10%.

Table 2 the composition and concentration of the digestion solution.

Solution name	Concentration	ID
HCL	2M	A
H_2SO_4	2%	C
NAOH	10M	E
H_3PO_4	10%	G

- Aluminum foil.
- Microscope type (BTC BIM 312T)
- Analytical scale.
- Tube glass 12ml size.
- Filter paper type (MN619G) (slow filtration, phosphate-free filter paper, thickness: 0.17mm, filtration speed: 100s, basis weight: 75g/m^2)
- 1-1 Petri dish.
- Flask.
- Funnel.

3.1.3 The methodology and protocols established to investigate and analyze

In this study, the evaluation of the impact of different acidic and basic solutions on PET/PETE, HD-PE, PE-LD, and PP microplastics was carried out using four protocols listed in Table 3. Protocol 1 involved treating the microplastics with 5 mL of 2M HCl at both room temperature for 24 h and 60 °C for 1 hour. Similarly, Protocol 2 subjected the microplastics to 5 mL of 2% H_2SO_4 for 24 h at room temperature and 60°C for 1 hour. In addition, Protocol 3 and Protocol 4 subjected the microplastics to 5 mL of 10M NaOH and 5 mL of 10% H_3PO_4, respectively, under comparable conditions.

Table 3 the nature of the established protocols.

Protocol number	Solution	Polymer type	Temperature
1	HCL	PET/PETE HD-PE PE-LD PP	23 °C,60°C
2	H_2SO_4	PET/PETE HD-PE PE-LD PP	23 °C,60°C
3	NaOH	PET/PETE HD-PE PE-LD PP	23 °C,60°C
4	H_3PO_4	PET/PETE HD-PE PE-LD PP	23 °C,60°C

3.1.4 Experiment Procedure of Chemical Reactions of Microplastics

Figure 10 shows the steps of the filtration process. For this experiment, each cut polymer sample was weighed with an accuracy of 0.0001g (0.1mg), with approximately 0.1000g of PET/PETE and HD-PE, and 0.0100g of PE-LD and PP being weighed and transferred to sanitized 12ml separate glass test tubes (a). The tubes were then treated with 5 ml of the corresponding digestion solution, added slowly to ensure complete immersion of the sample material, and covered with aluminum foil to prevent air contamination (b). Exposure durations of 24 hours and 1 hour were employed for room temperature and 60°C temperature samples, respectively. After exposure, the samples were filtered, thoroughly rinsed with distilled water to remove any remaining acid and base agents (c), and dried in glass Petri dishes (d). The washed MP particles were dried overnight and stored in a desiccator before being weighed again at a later time with an accuracy of 0.01mg to determine the percentage change in weight. Microscopy images were used to document any variations in size, shape, or color in the polymers after treatment.

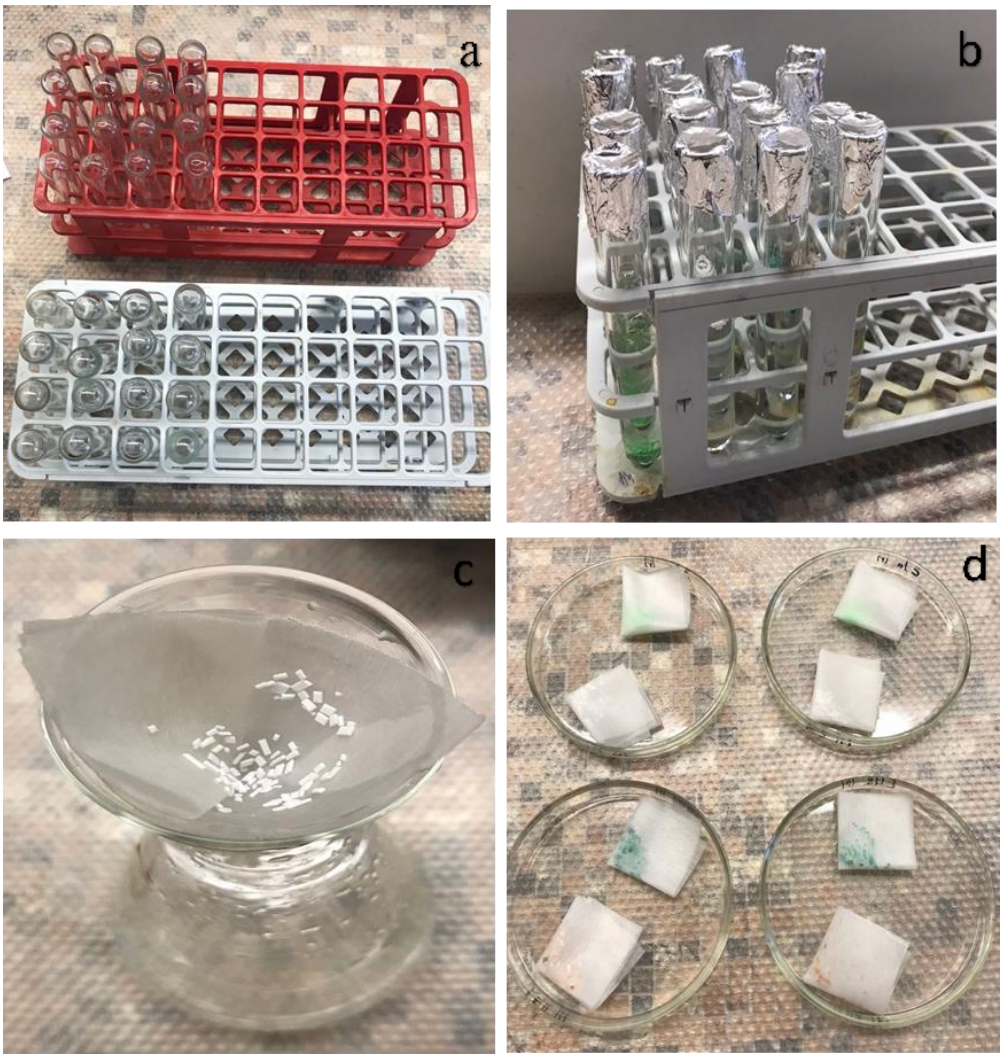


Figure 10 shows the steps of the filtration process (a, b, c and d)

3.1.5 Evaluation of digestion protocols impacts

In order to assess the influence of digestion protocols, criteria were established to evaluate the impact of the treatment on the microplastics, taking into account changes in mass, effects, and damage.

- Mass variation

To assess the mass variation of the polymer, the analytical scale was used to weigh the samples before and after the digestion protocols were carried out, with each protocol being tested twice as is shows in Table 4.

- Effects and damage aspects on the polymer.

The polymers were examined for damages resulting from the treatment using an electronic microscope of type BTC BIM312T at a magnification of 20x. The microscope was equipped with a camera and Troup view software to capture the images.

Table 4 The weight measurement variances prior to and post-treatment.

Solution ID	Polymer type ID	Temperature	MP original mass (g)	Filter paper + MP mass (g)	Filter paper weight (g)	MP mass after (g)
		Room temp 23 °C				
A	1a	r	0.0996	0.4737	0.3812	0.0925
A	2	r	0.0989	0.5081	0.4145	0.0936
A	4a	r	0.0123	0.3960	0.3834	0.0126
A	5a	r	0.0120	0.4058	0.3980	0.0078
C	1a	r	0.1000	0.4822	0.3848	0.0974
C	2	r	0.0996	0.5013	0.4036	0.0977
C	4a	r	0.0112	0.3934	0.3841	0.0093
C	5a	r	0.0101	0.4317	0.4291	0.0026
E	1a	r	0.0997	0.5003	0.4037	0.0966
E	2	r	0.0977	0.5232	0.4277	0.0955
E	4a	r	0.0116	0.4306	0.4239	0.0067
E	5a	r	0.0108	0.4121	0.4045	0.0076
G	1a	r	0.0971	0.5480	0.4537	0.0943
G	2	r	0.0983	0.5236	0.4270	0.0966
G	4a	r	0.0120	0.5168	0.5066	0.0102
G	5a	r	0.0122	0.4151	0.4057	0.0094
		60 °C				
A	1a	h	0.0951	0.4028	0.3078	0.0950
A	2	h	0.0971	0.4089	0.3120	0.0969
A	4a	h	0.0105	0.3157	0.3054	0.0103
A	5a	h	0.0113	0.3780	0.3684	0.0096
C	1a	h	0.0943	0.5032	0.4091	0.0941
C	2	h	0.0999	0.4931	0.3937	0.0994
C	4a	h	0.0142	0.3314	0.3168	0.0146
C	5a	h	0.0105	0.4367	0.4261	0.0106
E	1a	h	0.0943	0.5440	0.4502	0.0938
E	2	h	0.0987	0.4968	0.3981	0.0987
E	4a	h	0.0116	0.3636	0.3524	0.0112
E	5a	h	0.0114	0.3209	0.3104	0.0105
G	1a	h	0.0994	0.5545	0.4552	0.0993
G	2	h	0.0990	0.5239	0.4248	0.0991
G	4a	h	0.0109	0.3479	0.3375	0.0104
G	5a	h	0.0121	0.3882	0.3761	0.0121

3.1.6 Quality control

Several measures were implemented to mitigate contamination and guarantee the precision and accuracy of the experimental results. The glassware and equipment used were thoroughly cleaned and rinsed using a distilled water prior to usage. A cotton coat was worn during laboratory procedures to prevent synthetic clothing from contaminating the samples. The use of plastic materials was entirely avoided to further reduce the possibility of contamination. The samples were stored in glass tubes and covered with aluminum foils throughout the experiments to maintain their integrity. Blank samples were included in each batch of samples consisting of distilled water, solutions, and three sets of filter papers (original and dry) to confirm the absence of contamination during analysis. In the experiment, the absence of microplastics was observed in the blank samples.

3.2 Experiment Two: Quantifying the release of microplastics during washing and evaluating different filtration methods for the filtration process.

3.2.1 Design of the Experiment

The present study utilizes a systematic methodology, which involves a series of controlled experiments to evaluate the number and type of microplastics released from a domestic washing machine, and assess their removal efficiency after filtration using sand and filter paper. The primary objective of these experiments is to evaluate the release of microplastics on a real-life scale and analyze various mitigation techniques by testing the removal efficiency. To ensure precision, reliability, and consistency, the experiments are conducted under strictly controlled laboratory conditions, with accurate measurement and recording of all variables. The released fibers are counted and observed under a microscope using Troup view software, and detailed notes are taken throughout the experimental process to document the testing conditions, outcomes, and any unexpected observations.

In this study, five types of commercial garments (Cotton, Viscose, Elastane, Polyamide, and Polyester) are evaluated using a Zanussi ZWQ5102 washing machine at 30°C, using a synthetic wash program with a water requirement of 46 liters. The commercial powder detergent Tomi is used for the washing test, and all garments are washed together with a load of 2.5 kg. Two replicates of the wash water and rinsing water tests are performed, each filtered using either filter paper or a sand filter.

3.2.2 Materials

- Five different fabrics type was used were selected for the washing experiments: Cotton, viscose, elastane, polyamide, and polyester.
- The commercial powder laundry detergent used in the washing tests for the release is Tomi and the Coccolino Blue detergent.
- Washing machine type Zanussi ZWQ5102.
- Filter paper type MN619G (slow filtration, phosphate-free filter paper, thickness: 0.17mm, filtration speed: 100s, basis weight: 75g/m^2)
- Sand Filter , The parameters of the sand filter described in Table 5.
- Aluminum foil.
- Microscope type (BTC BIM 312T)
- Analytical scale.
- Petri dish.

Table 5 sand filter layer parameters.

Sand layer parameters	
Diameter (sand particle)	100-1000 µm
Thickness	3 cm
Filter layer's diameter	6 cm

3.2.3 The methodology and protocol established in the Quantifying the release of microplastics during washing and evaluating different filtration methods for the filtration process.

Table 6 shows the protocols used in this experiment. In this study, two protocols were implemented to quantify the number of microplastics released during a domestic washing process and to investigate the efficiency of the filtration procedure. In protocol one, the pre-wash water was filtered successively using filter paper and sand filter, with another sample filtered using filter paper only. Protocol two involved filtering the pre-rinsing water using filter paper and sand filter in sequence, with another sample filtered using filter paper alone.

Table 6 A compilation of utilized protocols and their corresponding identification codes.

ID	Experiment
1	Washing water + filter paper
2	Washing water + sand layer + filter paper
3	Rinsing water + filter paper
4	Rinsing water + sand layer + filter paper

3.2.4 Experiment Procedure of the washing process

- **Washing process**

  The synthetic textiles underwent laundering in a front-load washing machine model Zanussi ZWQ5102, using a quantity of 5 g of powder detergent (Tomi) with a concentrated softener detergent (Coccolino blue). The laundering process involved washing all textiles together under the "synthetic" program, utilizing the following settings: water temperature maintained at 30 °C, the total duration of 40 min, and water volume of 46 liters, with 23 liters for washing and 23 liters for rinsing. The washing water was obtained from a tap source. To evaluate microfiber release, the analytical procedure involved separately collecting 50-50 ml of post-wash and post-rinse water, directly from the washing machine's drainpipe.

- **Filtration process**

  The washing effluents generated during the pre-wash (a) and pre-rinsing (b) were collected separately and were subjected to filtration using MN619G filter paper and sand layer (c), as shown in the Figure 11 respectively. This was followed by filtration through MN619Ga filter paper. To prevent excess detergent from adhering to the filter surface and to capture any loose fibers, the filter was thoroughly rinsed with distilled water. Subsequently, the filter papers were transferred to 1-1 Petri dishes and dried for 1 hour at a temperature of 45-50°C in a drying oven. After cooling in an executor, the filter papers were weighed before

and after filtration to determine the number of microfibers released, which was normalized for the washing load.

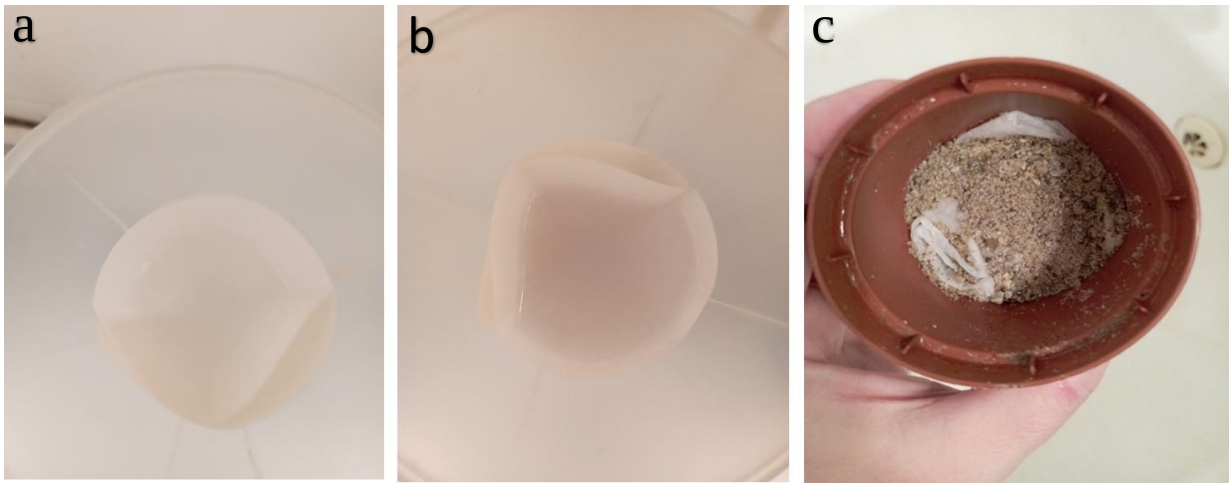


Figure 11 show the filtration process a) Filtering rinsing water, b) filtering washing water, and c) Sand filter layer

3.2.5 Quantification and observation of microplastics

The observations were conducted using two methods:

- Microplastic sizing and weight estimation involved measuring the mass of the filter papers before and after drying as shown in Table 7.

Table 7 Table of the filter paper weight before and after the filtration.

ID	Experiment	Original filter paper mass [g]	Filter paper mass after filtration [g]
1	washing water + filter paper	0.6607	0.6638
2	washing water + sand layer + filter paper	0.6148	0.6250
3	rinsing water + filter paper	0.6714	0.6738
4	rinsing water + sand layer + filter paper	0.6253	0.6330

- The counting method involved analyzing electronic microscope micrographs of the filter surfaces using ImageJ software to quantify the number and length of the microfibers released.

3.2.5 Quality control

To minimize sample contamination throughout the experiments and analyses, precautions were taken. A white lab coat and nitrile gloves were worn during all experimental procedures. To prevent fiber contamination from the laboratory air, the filters were kept in closed Petri dishes whenever possible, and were only exposed during drying, which occurred in an oven. To assess possible contamination, four blank samples were collected, including the original filter paper, water, a 10:1 water to washing powder (Tomi) mixture, and a 10:1 water to concentrate (Coccolino Blue) mixture. In the experiment, the absence of microplastics was observed in the blank samples.

4. Results and evaluation of results

4.1 The results obtained from experiment one: Assessing the resistance of microplastic polymers to various acidic and basic digestion treatments and investigating the effect of different digestion solutions on microplastic polymers.

The present investigation reveals that the degradation of microplastic polymers was markedly influenced by the digestion solutions utilized. The proportion of microplastic loss varied in a manner dependent on the type of polymer and digestion solution employed. Specifically, the samples subjected to testing at room temperature for 24 hours as shown in Table 8 exhibited a greater degree of harm as compared to those tested at 60°C for 1 hour as shown in Table 9.

Table 8 Table display microplastic loss% for samples subjected to treatment at room temperature (23°C).

Solution ID	Polymer type ID	Temperature	Microplastic loss %
		Room temp 23°C	
A	1a	r	0.71%
A	2	r	0.53%
A	4a	r	0.03%
A	5a	r	0.42%
C	1a	r	0.26%
C	2	r	0.19%
C	4a	r	0.19%
C	5a	r	0.75%
E	1a	r	0.31%
E	2	r	0.22%
E	4a	r	0.49%
E	5a	r	0.32%
G	1a	r	0.28%
G	2	r	0.17%
G	4a	r	0.18%
G	5a	r	0.28%

Table 9 Table display microplastic loss% for samples subjected to treatment at (60°C).

Solution ID	Polymer type ID	Temperature	Microplastic loss %
		60 °C	
A	1a	h	0.01%
A	2	h	0.02%
A	4a	h	0.02%
A	5a	h	0.17%
C	1a	h	0.02%
C	2	h	0.05%
C	4a	h	0.04%
C	5a	h	0.01%
E	1a	h	0.05%
E	2	h	0%
E	4a	h	0.04%
E	5a	h	0.09%
G	1a	h	0.01%
G	2	h	0.01%
G	4a	h	0.05%
G	5a	h	0%

4.1.1 Effects of Acids on Synthetic Polymers

The present study investigated the impact of acidic digestion solutions (HCl, H_2SO_4, and H_3PO_4) on various types of polymers at room temperature (23°C). The findings revealed that H_3PO_4 had a lesser impact on polymers than HCl and H_2SO_4, irrespective of the type of polymer and temperature as shown in Figure12. Among the synthetic polymers, PET/PETE exhibited a higher microplastic loss of 0.71% with the application of HCl at room temperature, while a slight decrease in weight was observed in HD-PE with a 0.53% microplastic loss and in PP with a 0.42% microplastic loss under the same concentration of HCl. However, the degree of degradation decreased with the increase in temperature up to 60°C, and the microplastic loss % for each polymer type (PET/PETE, HD-PE, PE-LD, and PP) was 0.01%, 0.02%, 0.02%, and 0.17%, respectively. H_2SO_4 caused a significant loss of microplastics for PP at room temperature with a 0.75% loss, while HD-PE and PE-LD showed an insignificant weight loss with a 0.19% loss for the same solution. At 60°C, the amount of microplastic loss % for all polymer types (PET/PETE, HD-PE, PE-LD, PP) was reduced, with corresponding loss % of 0.02, 0.05, 0.04, and 0.01, respectively. In contrast, H3PO4 had the lowest impact on all types of polymers, with only a 0.28% loss observed for each PET/PETE and PP, and a 0.18% loss for PE-LD, and the least impact observed for HD-PE with a 0.17% loss. At

60°C, there was a notable reduction in microplastic loss, with a loss percentage of 0% for PP polymer type, 0.01% for both PET/PETE and HD-PE, and nearly 0.05% for PE-LD.

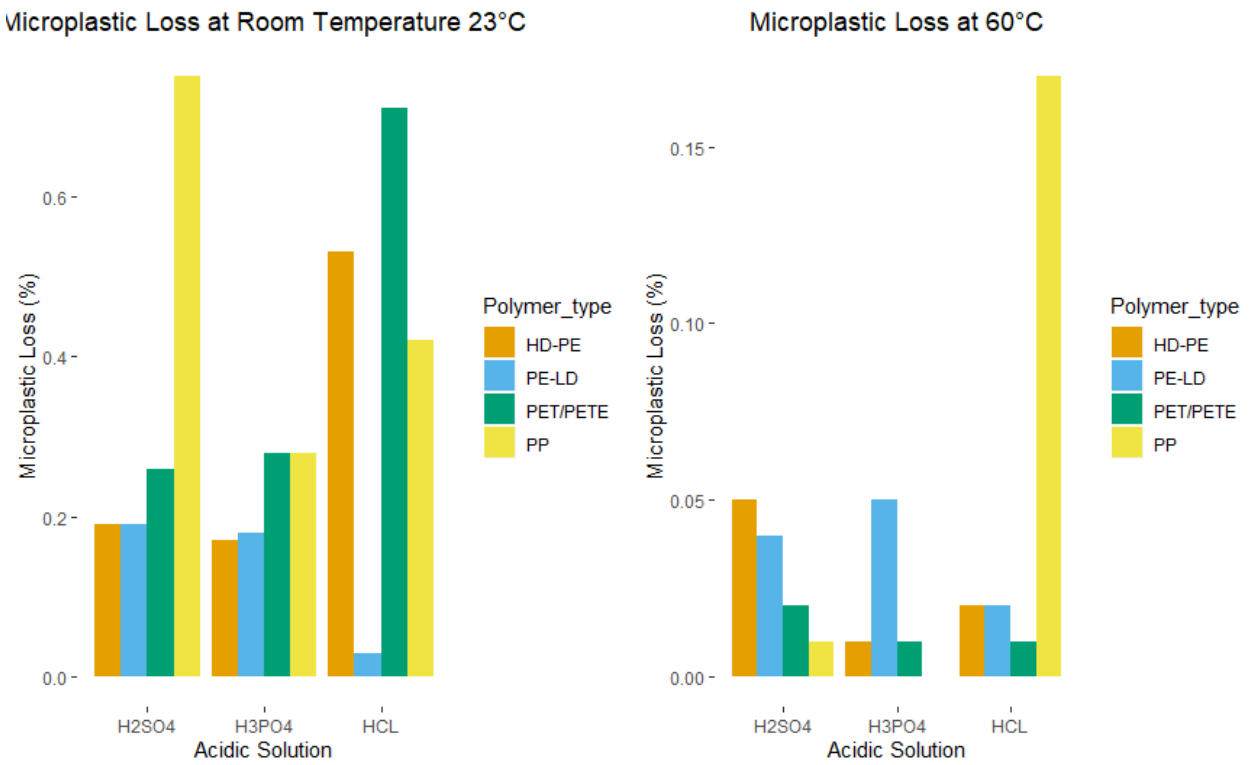


Figure 12 The plot depicts the percentage of microplastic loss for various types of polymers when exposed to acidic solutions at a 60°C and, temperature of 23°C.

4.1.2 Effects of the Base on Synthetic Polymers

Based on the findings presented in Figure13, it is apparent that NaOH-based protocols result in a negligible microplastic loss in most of the tested synthetic polymers, regardless of the temperature (room temperature and 60°C), with the exception of PE-LD. PE-LD demonstrated a comparatively higher loss percentage at room temperature (0.49%), though the difference was not statistically significant. PET/PETE and PP showed a slight weight loss of approximately 0.31% and 0.32%, respectively, under the NaOH protocol at room temperature. HD-PE experienced only a 0.22% microplastic loss when subjected to NaOH solution. At 60°C, the impact of the NaOH protocols on different polymer types were minimal, with microplastic loss % of 0.05% for PET/PETE, 0.04% for PE-LD, and 0.09% for PP. The polymer-type HD-PE showed no significant microplastic loss under these conditions. In conclusion, NaOH solutions have a relatively insignificant effect on the microplastic loss of most synthetic polymers at both room temperature and 60°C.

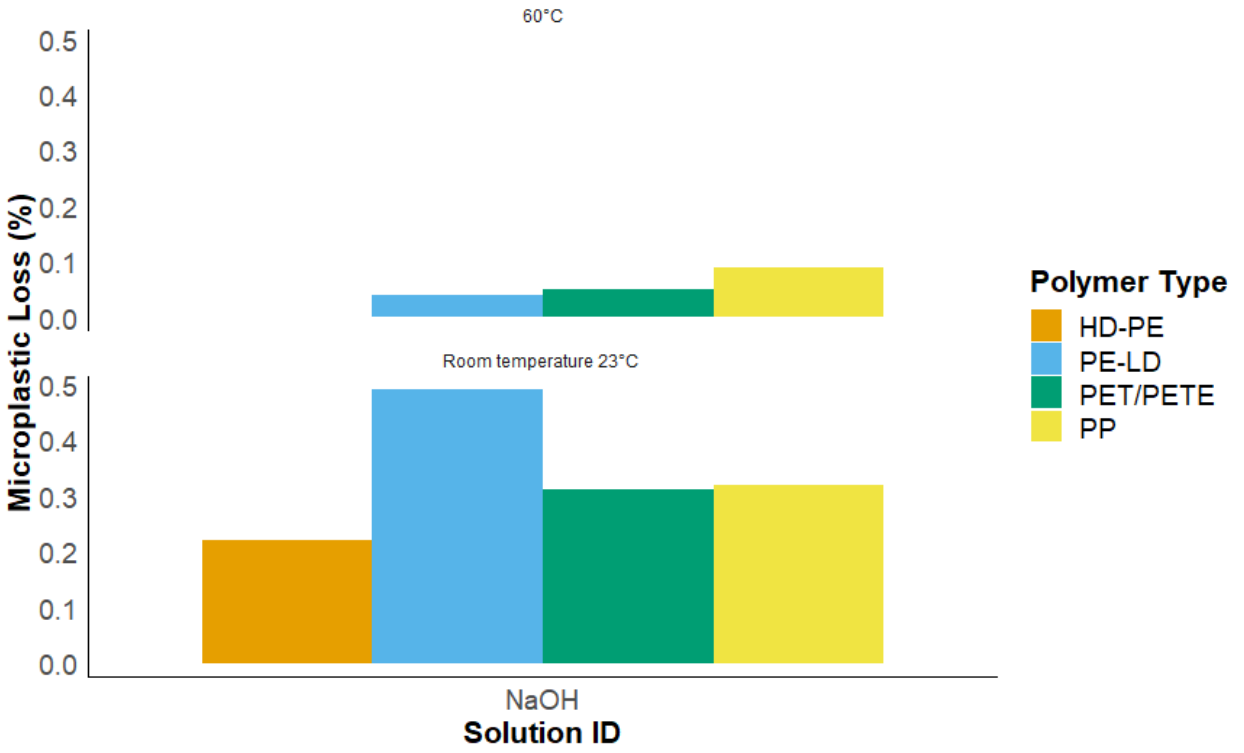


Figure 13 The plot depicts the percentage of microplastic loss for various types of polymers when exposed to NaOH base solutions at a 60°C and, temperature of 23°C.

4.1.3 Resistance of polymers

In this scientific study, the resistance of various polymers to different chemical solutions at different temperatures was investigated. PET polymer showed low resistance to HCl at 23°C, resulting in a high microplastic loss of 0.71%. However, PET/PETE polymer exhibited increased resistance to HCl at 60°C, resulting in a microplastic loss of only 0.01%. HD-PE microplastic particles demonstrated low resistance to HCl at room temperature, resulting in a microplastic loss of 0.53%, but high resistance to NaOH at 60°C with zero microplastic loss. LD-PE polymer showed higher resistance than other polymers, but NaOH at room temperature resulted in the least resistance with a loss of 0.49%. Testing LD-PE MP samples under 60°C showed less microplastic loss for all the solutions tested. PP polymer exhibited the lowest resistance when treated with H2SO4 solution at room temperature, with the highest microplastic loss among other polymers of 0.75%. Notably, the highest resistance was observed when treated with H3PO4 at 60°C, resulting in zero microplastic loss. Figure 14 depicts the highest resistance for polymers reacting with different solutions at room temperature, which was observed for PP and HD-PE. Conversely,

PET/PETE and LD-PE showed the highest resistance when treated under 60°C. Overall, HD-PE was the most resistant polymer under both tested temperatures.

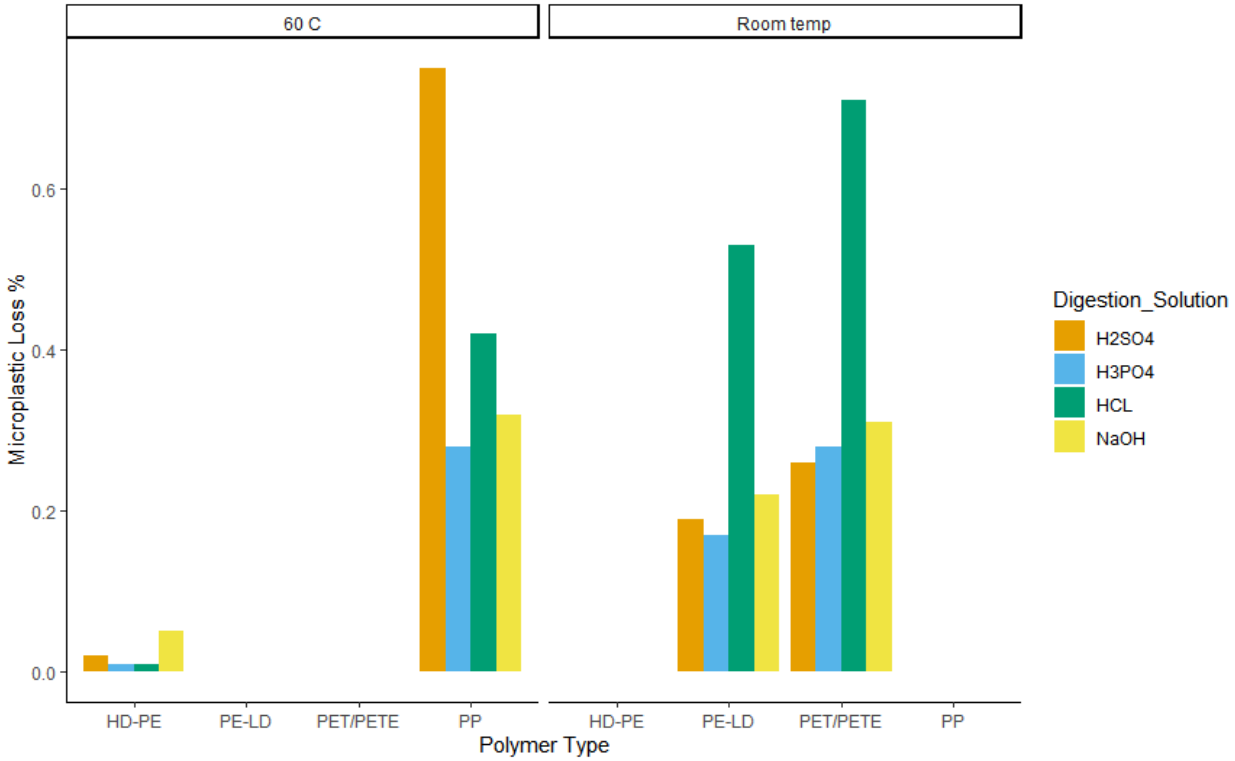


Figure 14 the plot depicts the most resistance polymers when treated with (H_2SO_4, H_3PO_4, HCL and NaOH) solutions at a 60°C and, temperature of 23°C.

4.1.4 Evaluating the Impact of Digestion Treatment on polymers particles

Employing a BTC BIM312T electronic microscope with Troup view software to observe the structural deteriorations caused by four different digestion treatments on the chemical stability of microplastic polymer samples by microscope images depict the surface of the samples that reacted with the acids and bases at room temperature 23°Cand at 60°C.

PET/PETE

Figure analysis revealed the occurrence of edge deformations in microplastic (MP) particles as shown in Figure 15, signifying chemical degradation between PET/PETE and HCl solution at room temperature Figure (a). However, the extent of damage was negligible, with almost no observable effects observed in samples treated at 60°C, as seen in Figure (b). Conversely, samples treated with H_2SO_4 at 60°C exhibited noticeable damage on the edge and surface, as depicted in Figure (c), whereas the effect on the

edge was more pronounced for samples treated at 23°C, as shown in Figure (d). NaOH treatment induced significant damage on the edge at 60°C, as seen in Figure (e), and at 23°C, as presented in Figure (f). On the other hand, H_3PO_4 solution treatment did not lead to any significant effects at 60 C temperatures, as demonstrated in Figure (g) and slightly effected the edges at the 23C samples Figure (h).

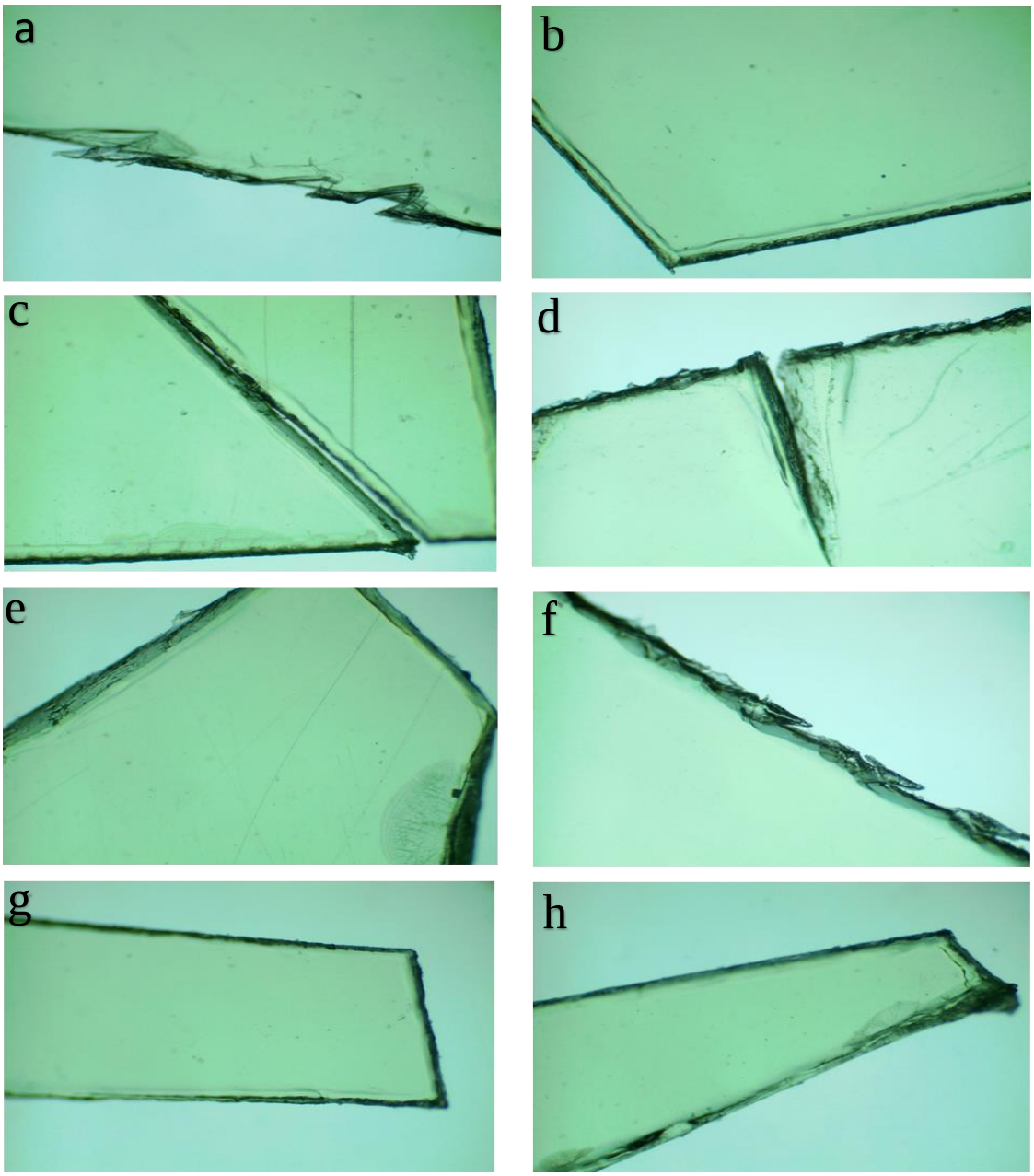


Figure 15 shows the effects of the different digestion solution on the PET/PETE.

HD-PE

The HD-PE microplastic polymers exhibit a compact and solid appearance, and their surfaces demonstrate no discernible effects when treated with various solutions as shown in Figure 16 . However, upon reaction with HCl, minor damages were observed primarily on the edges, but the effects were not significant for both 23°C and 60°C temperatures, as depicted in Figure (a) and Figure (b). Upon treatment with H_2SO_4, more pronounced damage was observed on the edges for samples treated at 23°C, as shown in Figure (c), whereas less damage was observed for samples treated at 60°C, as depicted in Figure (d). When subjected to NaOH treatment at 23°C, some damage was observed on the surface and edges of the sample, as illustrated in Figure (e), whereas no effects were observed for the 60°C sample, as shown in Figure (f). Additionally, no observable effects were found when reacting with H_3PO_4 at 60°C, as depicted in Figure (g), while minor damage was observed on the edges for samples subjected to treatment at room temperature, as shown in Figure (h).

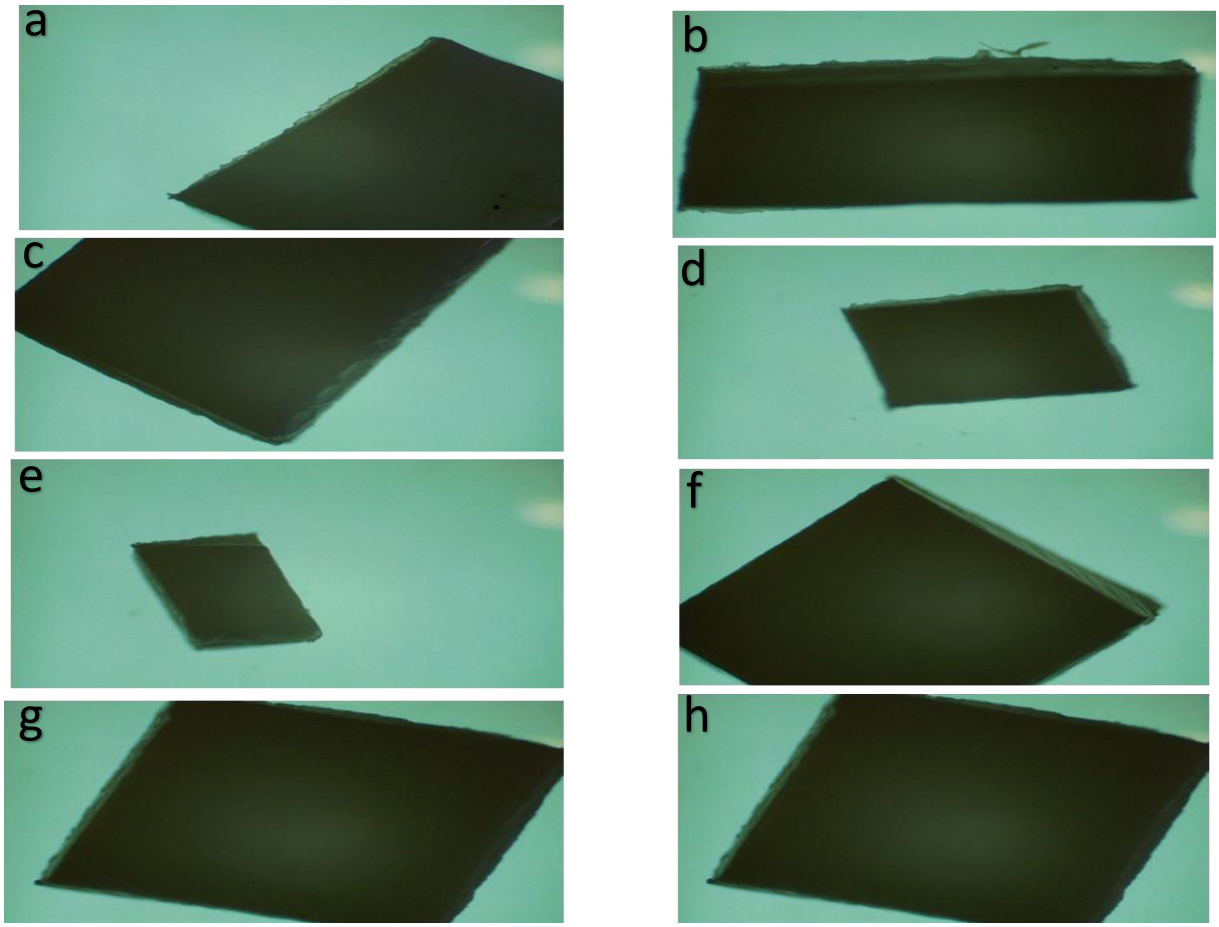


Figure 16 shows the effects of the different digestion solution on the HD-PE

PE-LD

Figure 17 shows the effects of different digestion solution on LD-PE. The LDPE MP samples were resistant to the treatment with HCL, and no effects were observed at both 23°C Figure (a) and 60°C, as seen in Figures (b). Additionally, there were no observed effects when treated with H_2SO_4 at both temperatures, as seen in Figures (c) for samples treated at 23°C and, Figure (d) for samples treated at 60°C. When treated with NaOH at 23°C, no effects were observed, as shown in Figure (e). However, the 60°C sample showed some surface damage with color changes, indicating that the treatment has effects on the polymers, as depicted in Figure (f). H_3PO_4 had surface effects on the sample treated at 23°C, as shown in Figure (g), while effects on the edges were observed for the 60°C samples, as seen in Figure (h).

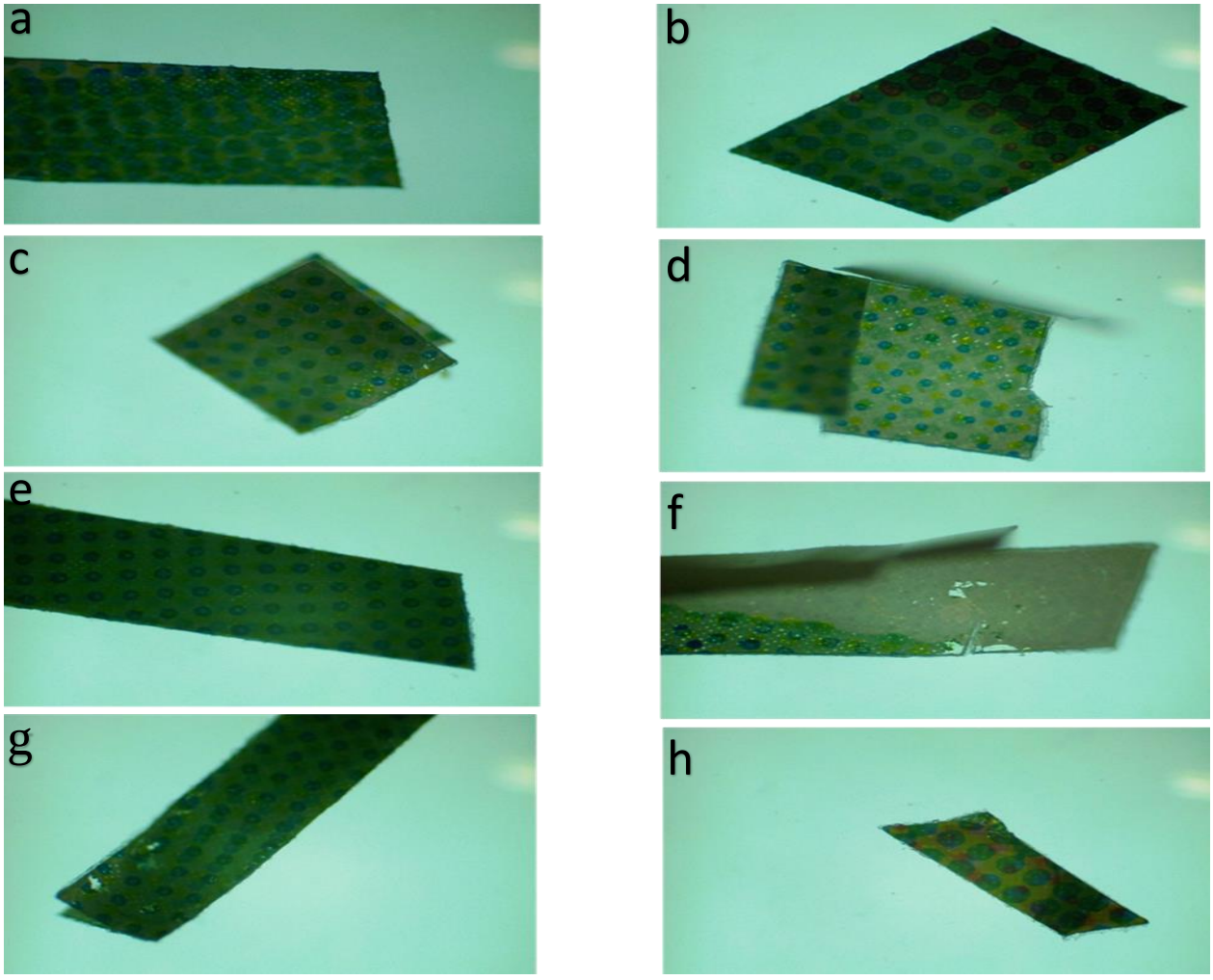


Figure 17 shows the effects of the different digestion solution on the PE-LD

PP

Figure 18 shows the effects of the different digestion solution on the PP. The PP polymer samples exhibited modifications and changes on their surface when subjected to HCl, H_2SO_4, NaOH, and H_3PO_4 at all temperatures and reaction durations. The PP MPs reacted with HCl at 23°C showed less damage on the surface, as seen in Figure (a), compared to the samples tested at 60°C, which exhibited clear damage on the surface with wide holes and changes in polymer color, as depicted in Figure (b). The reaction with H_2SO_4 at 23°C resulted in some surface effects, as shown in Figure (c), but as with HCl, the most observed damage was for the samples tested at 60°C, as seen in Figures (d), which included color vanishing. Similarly, the damage caused by NaOH at 23°C was observed on the surface, with slight color vanishing, as illustrated in Figure (e). However, the most significant damage was observed at 60°C, where more surface damage and changes in color were evident in Figure (f). H_3PO_4 at 23°C showed some effects on the surface, as seen in Figure (g), but more damage was observed after treatment at 60°C, where surface discoloration and the formation of huge holes were observed, as depicted in Figure (h).

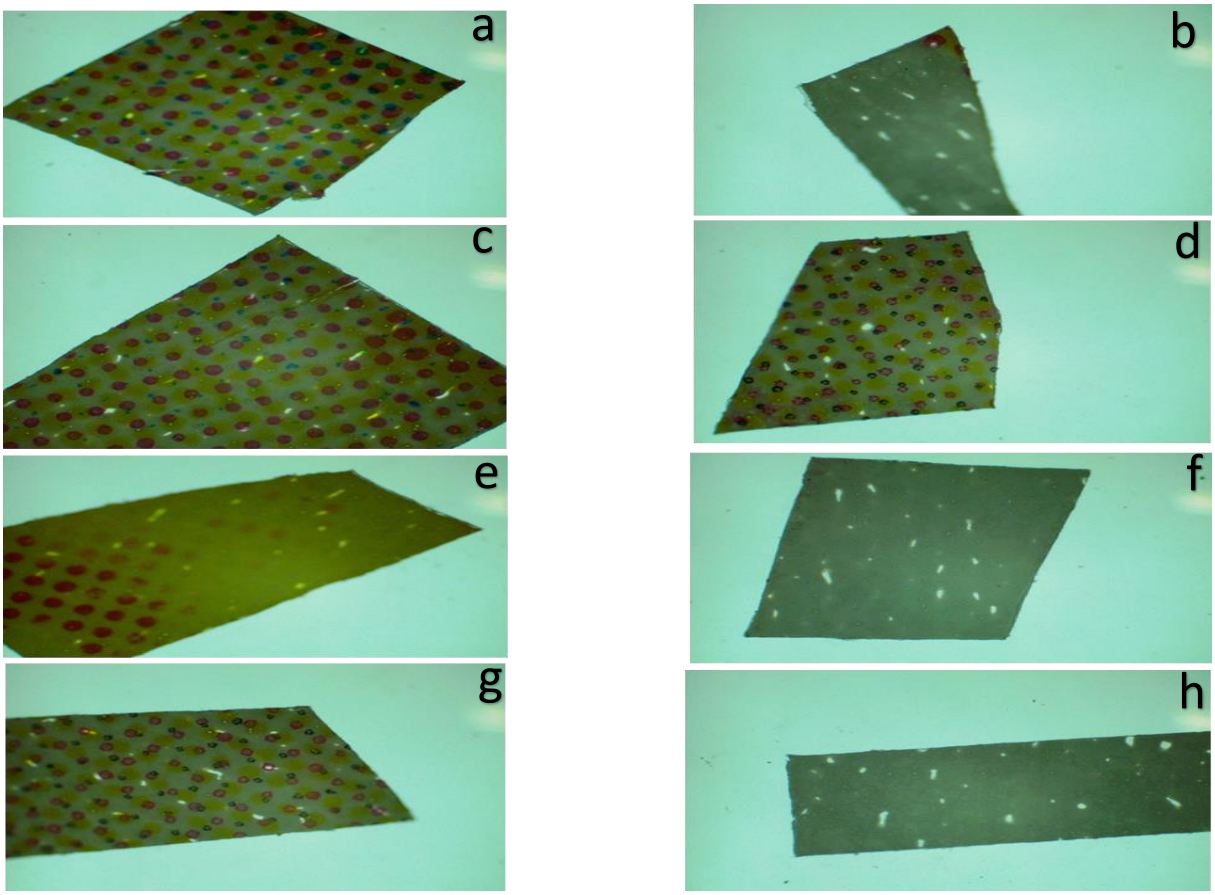


Figure 18 shows the effects of the different digestion solution on the PP

4.2 The result obtained from experiment Two: Quantifying the release of microplastics during washing and evaluating different filtration methods for the filtration process.

4.2.1 First phase of the experiment

In the preliminary stage, the objective was to evaluate the efficacy of different filtration techniques in eliminating effluent .The results indicate that the efficacy of different filtration methods in removing effluent from washing and rinsing water is significant, as demonstrated by the data presented in Table 10.

Table 10 the table presented displays the variations in weight observed in the filter paper following diverse filtration procedures.

ID	Experiment	original mass of the filter paper [g]	Mass after filtration [g]	Δm [g]
1	Washing water + filter paper	0.6607	0.6638	0.0031
2	Washing water + sand layer + filter paper	0.6148	0.6250	0.0102
3	Rinsing water + filter paper	0.6714	0.6738	0.0024
4	Rinsing water + sand layer + filter paper	0.6253	0.6330	0.0077

The presented Figure 19, illustrates the weight change in the filter paper after different filtration processes. The data revealed that the filtration of washing water with filter paper resulted in a higher effluent pass through the filter paper compared to the rinsing water sample, with a weight change (Δm) of 0.0031 g for the washing and 0.0024 g for the rinsing. Incorporating a sand layer in the filtration process led to a higher Δm, implying that the sand layer played a crucial role in retaining more particles on the filter paper. Specifically, the combination of filter paper and sand filter for washing water yielded a Δm of 0.0102 g, while for the rinsing water, it was 0.0077 g. These results suggest that the sand layer has a greater impact on the filtration of both washing and rinsing water, as depicted by the higher Δm values. Nevertheless, it is crucial to consider that the sand filter includes soil particles that contribute to the weight of the filter paper. Hence, the weight difference between the filter paper before and after filtration may not solely indicate the amount of

filtered washing or rinsing water. Additionally, other factors, such as the size and composition of the particles being filtered, could also influence the weight difference.

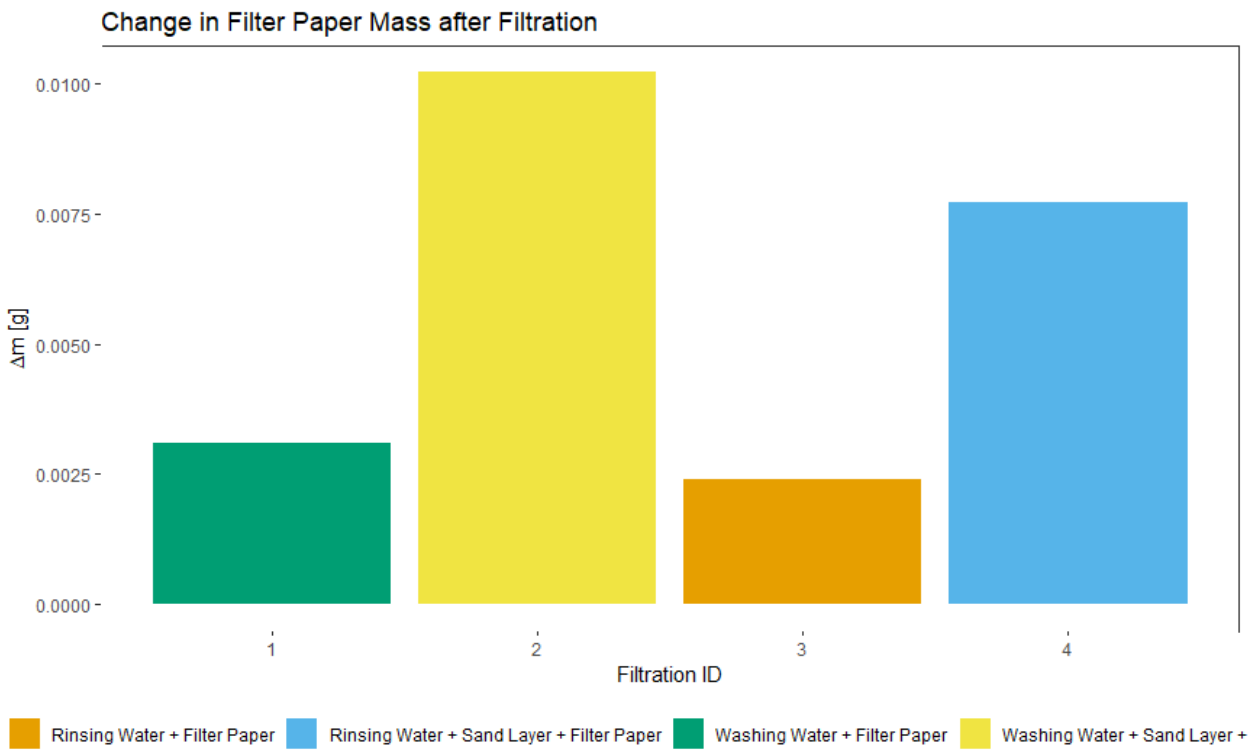


Figure 19 figure depicts the variation in filter paper weight resulting from the filtration of washing and rinsing water samples.

4.2.2 Second phase of the experiment

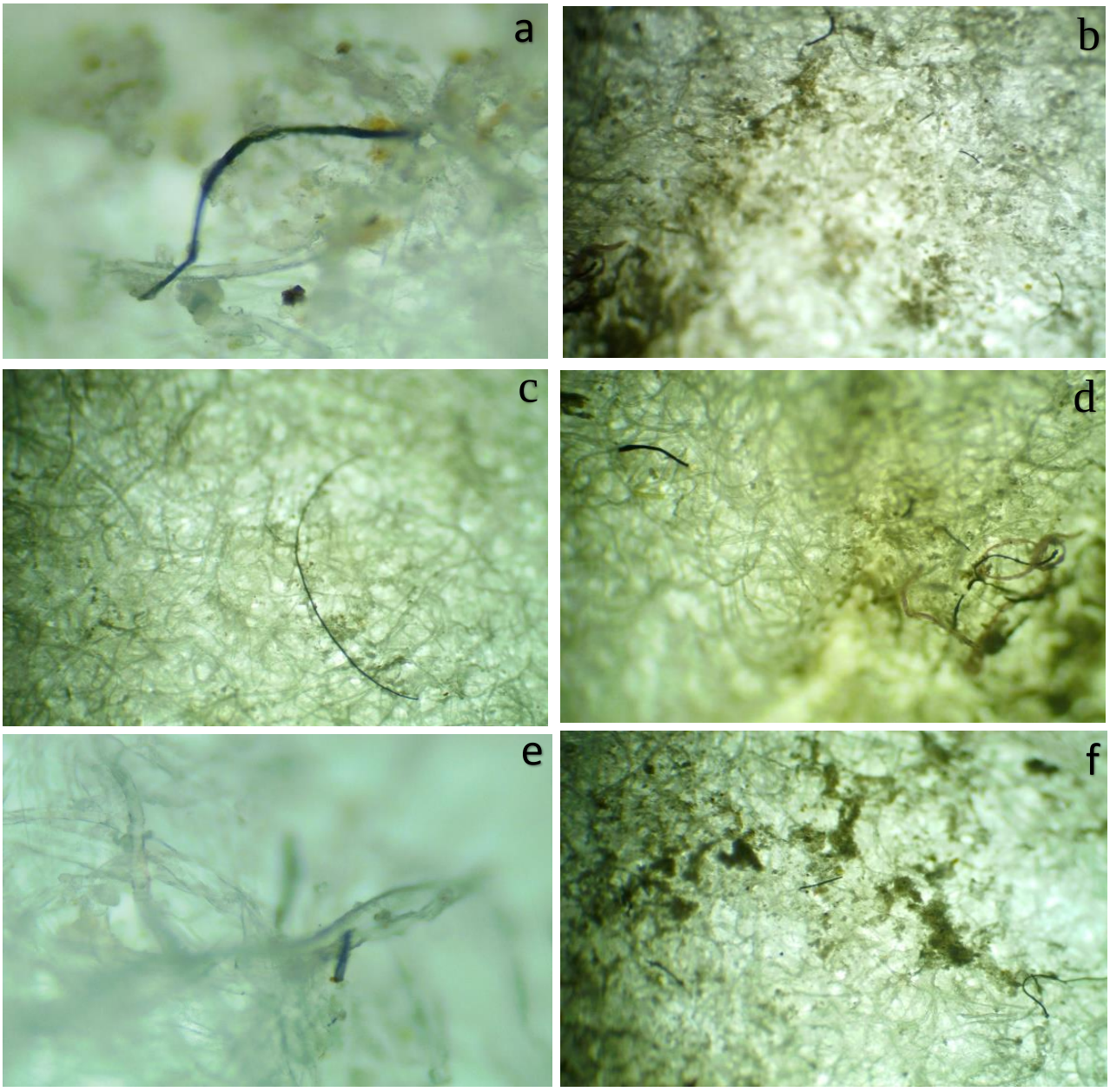
In the second phase of the experiment, an electronic microscope (BTC BIM312T) was utilized to investigate the filtered particles from the washing and rinsing water samples, with a particular focus on detecting any microplastic presence. The subsequent image analysis and particle counting were carried out one by one using the Troup view software. The result displayed in Table 11.

Table 11 Table displayed the number of particle observed using the microscope and their type and the color.

ID	Number of particle observed on microscope	Type	Color	Volume filtered (ml)
1	25	Fiber	Black	50
2	0	None	None	50
3	20	Fiber	Black	50
4	0	None	None	50

Samples filtered with filter paper only

Figure 20 display the fibers found in the pre-rensing and pre-washing water samples. Upon analyzing the filter paper following the filtration of 50 mL of the washing and 50 ml rinsing water samples, 25 particle as shown in figures (a, b, c,d,e and f) and 20 particle as shown in figures (g, h, i, j, k, and l) particles, respectively, were counted. The particles were identified as fibers with black-colored and were observed through an electronic microscope (BTC BIM312T). Furthermore, the average length of the fibers was determined using the Trop view software, revealing an average length ranging from 30-50 µm.



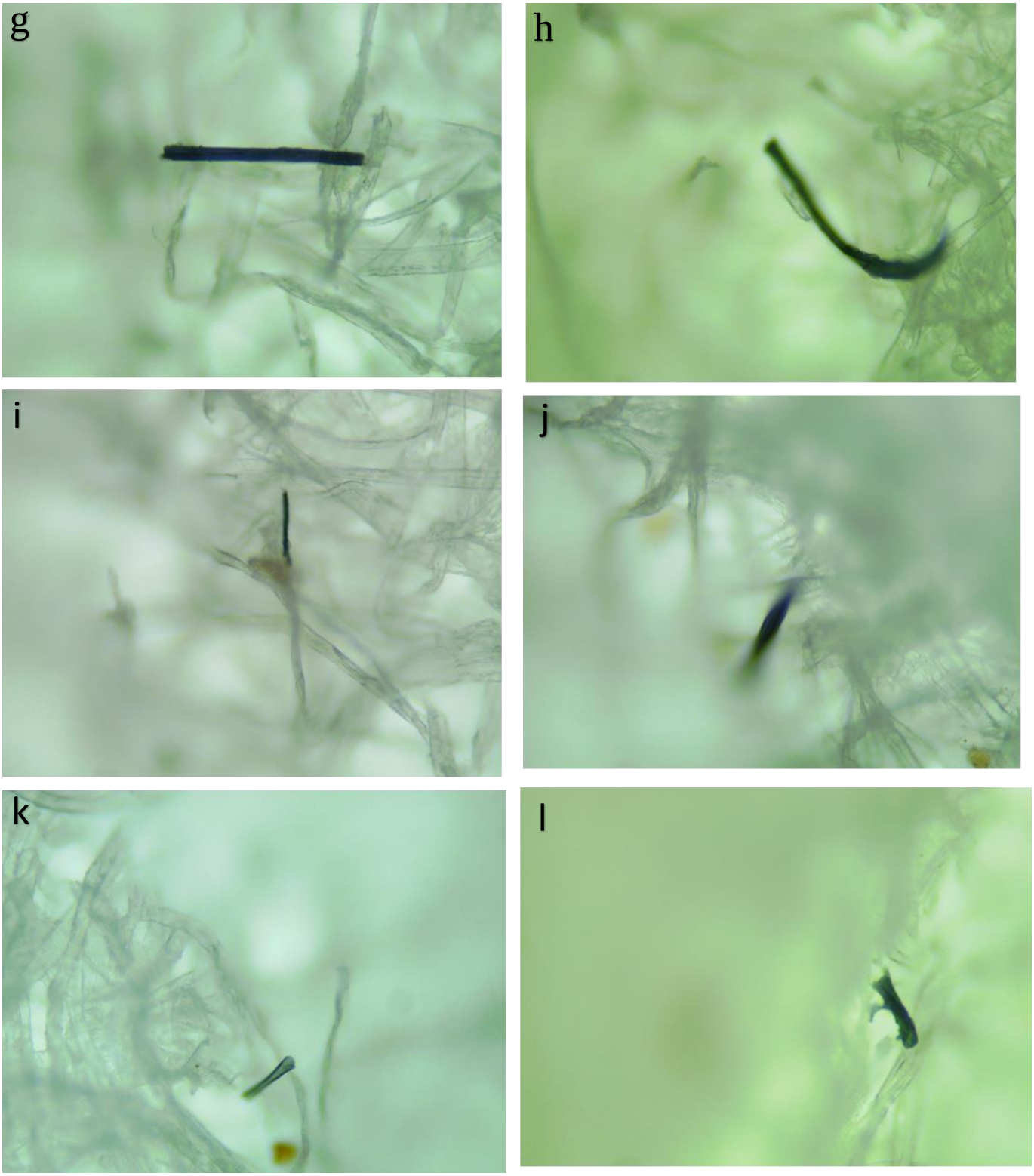


Figure 20 The fiber observed after filtering the washing sample is depicted in Figures (a, b, c, d, e, and f), the fiber observed after the filtering the rinsing samples is depicted in figures (g, h, i, j, k, and l)

Samples filtered with sand filter and filter paper

Figure 21 shows the Microscopic images for the samples filtered with sand filter. After passing 50 mL of washing and 50 mL of rinsing water samples through a sand layer and filter paper, respectively, no fibers were observed in either sample, as depicted in Figure (a and b) for washing samples and Figure (c and d)for rinsing water, respectively. Nevertheless, sand particles were visible on the filter paper, which could explain the observed weight differences in the first part of the experiment. The absence of fibers in the analyzed filter suggests that the sand filter is an efficient method for filtering samples and eliminating potential microplastic particles. Overall, the combination of a sand filter and filter paper shows promise as a method for microplastic filtration in water samples. These findings could have practical implications for future studies aiming to detect microplastics in environmental water samples.

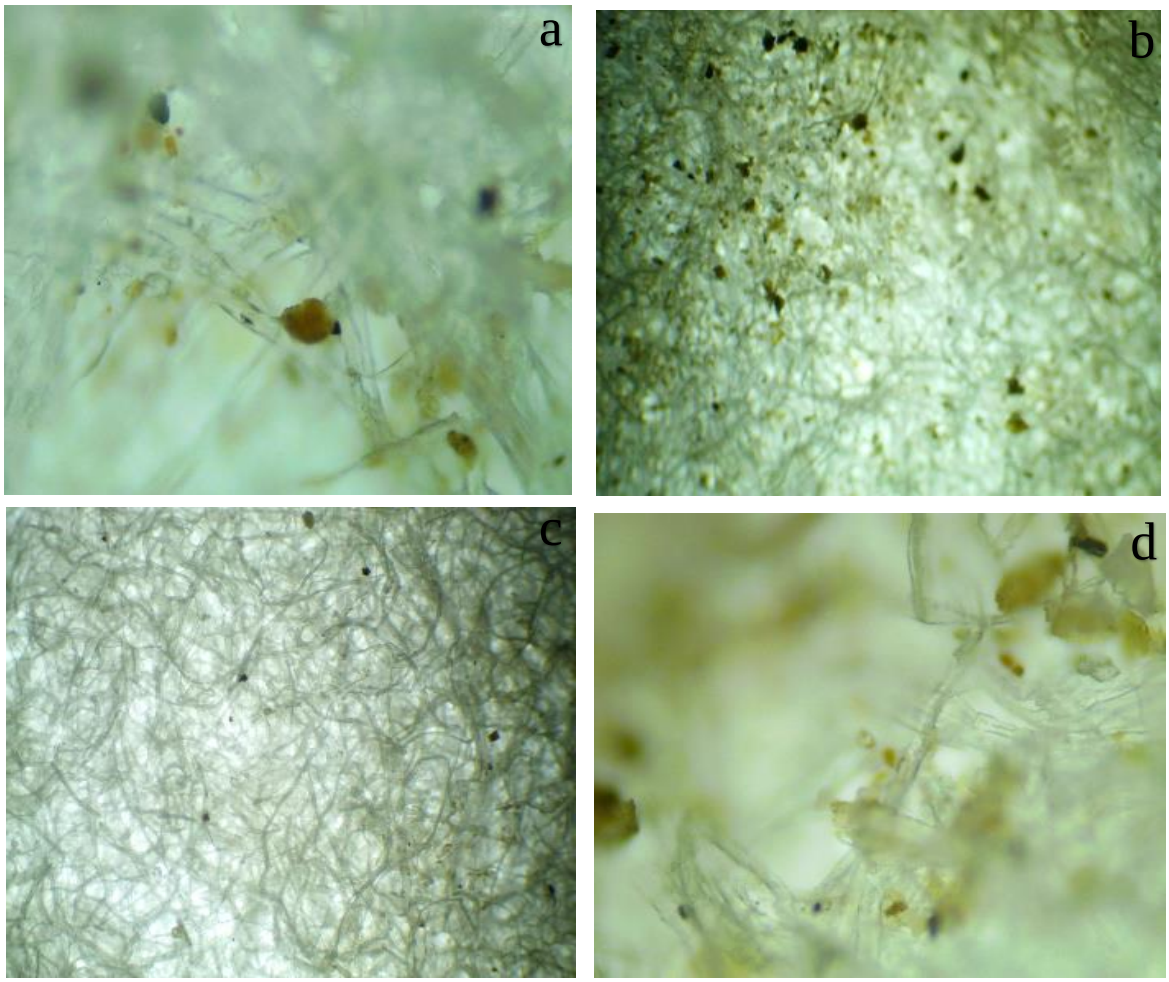


Figure 21 Figures (a and b) and Figures (c and d), present the sand particles captured on the filter paper of the washing and rinsing water samples, respectively.

Conclusions and recommendations

The first part of the study initially assessed the chemical resistance of microplastic polymers to acidic and alkaline agents to gain insight into their behavior in diverse chemical solutions. This is crucial for understanding the polymers' resistance to chemical degradation and their reaction to different solutions, aiding in identifying the microplastic origin and mitigating their environmental impact. Furthermore, the study aimed to determine optimal digestion solutions with the lowest impact on the polymer. The micro plastic Weight loss percentage results showed that PE-LD polymer had the highest resistance, while PP had the least resistance. Plus, the results indicate that the H_3PO_4 solution was highly effective for digestion with minimal microplastic loss, especially for samples tested at 60°C, where both PP and HD-PE demonstrated zero percent loss. However, microscopic analysis showed some surface effects on the polymers, particularly with PP, with observable changes in color in some cases. Therefore, while H_3PO_4 was the most effective solution for digestion, it may still affect the surface properties of the polymers, while the H_2SO_4 solution was the least affected solution for all polymers. These result indicate that reliance on microplastic loss measurement is insufficient, and microscopic examination is necessary to account for potential damages, and as the PP polymer had the least resistance in both tested part, suggesting its high presence in the environment as a microplastic. As the H_2SO_4 solution was the least affected solution for all polymers, suggesting its potential use as an essential part of the digestion protocol.

The second part of the study investigates the efficacy of a synthetic laundry program in reducing the release of microplastics during laundering and the amount of microplastic released at each step of the washing and rinsing process, plus, testing the effectiveness of a sand filter in capturing any released microplastics., The result revealed the presence of 25 fibers in washing and 20 fibers in rinsing water samples, with an average length of 30-50 µm and black color. However, the samples filtered through the sand filter showed no observable fibers, indicating the effectiveness of this filtration method. Results suggest that rinsing after washing may contribute to the release of fibers which shows that the use of a sand filter proved highly effective in filtering out microplastics, with over 99% of microplastics filtered out.

In conclusion, the first part shows that the use of organic matter digestion solutions not only significantly impacted microplastic loss but also may cause damage to synthetic polymers. Furthermore, relying solely on microplastic loss measurement is insufficient, and microscopic examination is necessary to account for any potential damages that may impact the particle

characteristics of the polymers and result in analytical errors when investigating the microplastic origin. PP exhibited the lowest resistance, indicating a high likelihood of presence in the environment compared to other tested polymers. The H_2SO_4 solution was the least affected solution for all polymers, suggesting its potential use as an essential part of the digestion protocol. The second part finding concludes that the rinsing process after washing may contribute to the release of fibers, and other factors such as particle size and composition could influence the weight difference between the filter paper before and after filtration also relying on weight is insufficient and microplastic examination is necessary also, the results highlight the potential of employing sand filter as a filtration method to mitigate the release of microplastics into the environment.

This investigation is significant because it offers insights into potential microplastic sources by identifying which polymers are disaggregated and which are more resistant and how each polymer interacts with different materials and can aid in the development of effective strategies to mitigate their impact. Therefore, examining the loss percentage of microplastics for different types of polymers exposed to various digestion solutions can be a crucial approach for identifying sources of microplastics and developing mitigation measures. Plus, these findings suggest the potential of employing this filtration method to mitigate the release of microplastics into the environment.

Standardizing techniques and practices for identifying and quantifying microplastics is imperative to comprehend their sources and environmental impact. However, variability among samples complicates the comparison between studies, resulting in inconclusive outcomes. Further research is necessary to fully understand the ecological implications of microplastics and develop effective strategies to mitigate their release and accumulation in the environment. With increasing per capita consumption of synthetic textiles, there is a need to investigate factors influencing microfiber release during laundering and develop effective mitigation approaches. Therefore, establishing standardized protocols and procedures for future studies is essential.

Summary

The production of plastic has become a significant part of human life, with annual global production reaching 380 million tons, resulting in 250 million tons of plastic waste. Environmental pollution caused by microplastics (MPs) is a growing concern worldwide, as it has largely anthropogenic origins, prompting research into the consequences of uncontrolled MPs in the environment. This scientific study focuses on examining the main sources of microplastic pollution, which are secondary microplastics arising from the degradation of larger plastic debris and washing machines.

To accurately detect microplastic polymer origin in environmental samples, pretreatment reagents are commonly used to extract them from interfering substances. However, the use of inappropriate pretreatment reagents can damage or underestimate microplastics. Therefore, the first part of this research aims to evaluate the effectiveness of commonly used digestion solutions in extracting microplastics (MPs) with the least effects on microplastic polymers to evaluate the efficiency of commonly employed digestion solutions in extracting microplastics while limiting the effects on polymer particles. Additionally, the study examines the resistance of various commonly used polymer types, namely PET/PETE, HD-PE, PE-LD, and PP, under different experimental conditions for various types of solutions, namely sulfuric acid, hydrochloric acid, phosphoric acid, and sodium hydroxide, to evaluate their resistance. The second part of this study focuses on investigating the role of the washing processes of synthetic textiles on microplastic release and the effectiveness of sand filters in screening out microplastics.

The study found that HD-PE demonstrated the best resistance to acid and base digestion solutions and the least effect on particles, while PP showed the opposite, indicating that the PP polymer may present more potential to exist in the environment as a microplastic. The proposed digestion protocol, H_2SO_4, allows for the investigation of microplastic origins with minimal damage to the polymers, while the use of H_3PO_4 is not supported, as it shows effects on polymers that may alter the polymer particles and cause errors in identifying the origin of the polymers. In the second part of the study, washing and rinsing synthetic fabrics released microfibers ranging from 25 to 20 fibers from the washed fabric, indicating the release of microfibers during the washing process. However, when the sample was filtered with a sand filter, no fibers were found, indicating the effectiveness of the sand filter in capturing possible microplastic from the washing samples

The primary objective of this scientific investigation is to identify polymer types that exhibit reduced resistance to digestion, thereby enabling crucial information to be obtained on the sources of microplastics in the environment and assisting in the development of effective strategies to minimize their impact. Furthermore, the data collected from this research could be utilized to create measures that restrict the discharge of microplastics into the ecosystem, with particular emphasis on safeguarding water bodies, which are significantly impacted by this type of pollution and are vital for human health and well-being. In general, this research establishes the basis for the preparation and pre-treatment of microplastics and the assessment of their contamination and associated risks in the environment. By providing insights into the sources, impacts, and fate of microplastics, this study informs the development of prospective solutions despite uncertainties surrounding the origins and interactions of microplastics with diverse materials.

The present scientific investigation provides recommendations for mitigating the release of microplastics into the environment, based on the research findings. Specifically, monitoring the utilization of washing machines and synthetic textiles, and implementing sand filters to trap microfibers and microplastics are recommended. The study suggests the utilization of HD-PE and PET/PETE polymers instead of PP, as they demonstrate superior resistance to digestive solutions and are less prone to become microplastic. Adequate pretreatment reagents for extracting microplastics are also emphasized, with the use of the H_2SO_4 digestion protocol recommended for investigating microplastic origins with minimal damage to the polymers. Additionally, relying solely on weight differences may not yield accurate results, leading to potential errors and overestimation of the findings, and it is important to incorporate microplastic analysis into the research.

Abstract of Thesis

Investigation of micro polymer origin

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Department of Water Management and Climate Adaption/ Institute of Environmental Sciences
Primary thesis advisor: András Sebők, Assistant researcher, Institute of Environmental Sciences/ Department of Soil Science

Microplastics have become a pervasive environmental concern, and their origin and their impact on the ecosystem are not fully understood. This study aimed to investigate the micro polymer origin by evaluating the chemical resistance of microplastic polymers to different agents and determine optimal digestion solutions with the lowest impact on the polymer. The study also investigated the effects of clothes washing process in the release of microplastic into the environment.

The purpose of this thesis is to examine the behavior of microplastic polymers in various chemical solutions in order to identify their origin and reduce their environmental impact. The study found that relying solely on microplastic loss measurement is insufficient, and microscopic examination is necessary to account for any potential damages that may impact the particle characteristics of the polymers and result in analytical errors when investigating the microplastic origin. The study found that PP is most likely to be present in the environment compared to other polymers, and the H_2SO_4 solution is the least affected solution for all polymers, suggesting its potential use as an essential part of the digestion protocol.

Additionally, the study evaluated the effectiveness of a synthetic laundry program in reducing microplastic release during laundering and tested the use of a sand filter in capturing any released microplastics. The results suggest that rinsing after washing may contribute to the release of fibers, and relying solely on weight to measure microplastic filtration is insufficient. The use of a sand filter was highly effective, filtering out over 99% of microplastics. The findings suggest the potential of employing this filtration method to mitigate the release of microplastics into the environment.

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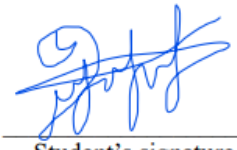

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