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Article

Size and Shape Distribution of Microplastics in PET Recycled Wastewater and Their Removal Behavior during the Coagulation–Flocculation Process

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A B S T R A C T

Plastic Recycling Facilities (PRFs), although intended to reduce plastic pollution, can act as potential sources of microplastic release due to mechanical processing activities. This study aims to investigate the size and shape distribution of microplastics in polyethylene terephthalate (PET) recycling wastewater and to evaluate their removal behavior through coagulation–flocculation processes. Wastewater samples were collected from several treatment stages of the wastewater treatment system at a PET recycling facility. The results indicate that microplastics are predominantly fragment-shaped, with medium-sized particles representing the most abundant fraction. Coagulation–flocculation was shown to effectively remove microplastics, particularly fragment-shaped and medium-sized particles, compared to film and fiber forms. Removal efficiency was influenced by microplastic size and shape characteristics. These findings suggest that coagulation–flocculation has strong potential as an effective method for controlling microplastic release from PET recycling wastewater and provides a basis for developing more targeted wastewater treatment strategies.

1. Introduction

Global plastic production exceeded 460 million metric tons in 2023, intensifying the need for effective recycling strategies to support circular economy goals and reduce environmental pollution (PlasticsEurope, 2024). Mechanical recycling of post-consumer plastics, particularly polyethylene terephthalate (PET), plays a critical role in decreasing virgin plastic demand and diverting waste from landfills and aquatic environments (Ragaert et al., 2017). However, recent studies indicate that plastic recycling facilities may unintentionally act as point sources of microplastic contamination (Suzuki et al., 2022; Brown et al., 2023).

During mechanical recycling, plastics undergo processes such as washing, shredding, and extrusion, which promote abrasion and fragmentation, leading to the generation of microplastic particles (Guo et al., 2022). Microplastic concentrations in wastewater from recycling facilities have been reported to be significantly higher than those in municipal wastewater influents, reaching up to 1.12×10^8 particles m^{-3} (Brown et al., 2023; Sun et al., 2019). These findings highlight the importance of understanding microplastic release from recycling operations.

The environmental behavior and removability of microplastics are strongly influenced by their physical characteristics, including particle size and morphology (Koelmans et al., 2022). Smaller particles tend to remain suspended and disperse more readily, while larger particles are more likely to settle in sediments (Kooi et al., 2018). In addition, particle shape affects biological interactions and treatment performance, with coagulation–flocculation processes exhibiting size- and shape-dependent removal efficiencies (Rajala et al., 2020). Nevertheless, systematic evaluations of how specific microplastic characteristics influence removal in industrial wastewater systems remain limited.

Although microplastics in natural waters and domestic wastewater have been extensively studied (Leslie et al., 2017; Sun et al., 2019), research on wastewater from plastic recycling facilities is still limited. Microplastics from mechanical recycling processes have distinct size and morphological characteristics, which affect treatment efficiency. Understanding these characteristics is crucial for optimizing the coagulation–flocculation process, particularly regarding coagulant selection, operational conditions, and the removal behavior of medium-sized particles (100–1000 μm) and the effect of morphology on treatment performance (Suzuki et al., 2022; Zhou et al., 2021; Khan et al., 2023; Tang et al., 2022).

2. Literature Review

2.1 Occurrence, Fate, and Impacts of Microplastics in Wastewater Treatment Plants (WWTPs

In general, wastewater treatment is carried out through several main stages, including preliminary, primary, secondary, and tertiary treatment. During the preliminary and primary stages, large materials are removed through screening, grit chambers, and sedimentation processes. Secondary treatment typically employs the activated sludge process in aeration tanks followed by secondary sedimentation to reduce organic matter loads. Tertiary treatment is subsequently applied for disinfection using chlorine, ozone, or ultraviolet irradiation. In addition to producing treated effluent, these processes also generate sludge, which is further treated through thickening, stabilization, and dewatering prior to reuse or safe disposal (Pal, 2017; Enfrin et al., 2019).

Despite the multiple treatment stages applied, numerous studies have shown that conventional wastewater treatment plants (WWTPs) are not fully effective in removing microplastics. Although substantial reductions occur during primary, secondary, and advanced treatment stages, microplastics are still detected in raw wastewater, final effluents, and sludge. Reported microplastic removal efficiencies in WWTPs range widely from 40% to 99.9%, with primary and secondary treatments contributing most significantly to removal, while advanced technologies such as membrane bioreactors demonstrate higher but more variable performance. Nevertheless, due to the large volumes of wastewater treated, considerable quantities of microplastics continue to be discharged into aquatic environments on a daily basis (Ziajahromi et al., 2017; Blair et al., 2019; Talvitie et al., 2017; Carr et al., 2016).

These findings indicate that, while WWTPs play a critical role in mitigating microplastic pollution, treatment performance remains inconsistent and influenced by multiple factors.

In this context, a better understanding of microplastic characteristics, particularly particle size and morphology, is essential, as these properties strongly affect transport behavior and removal efficiency during treatment processes

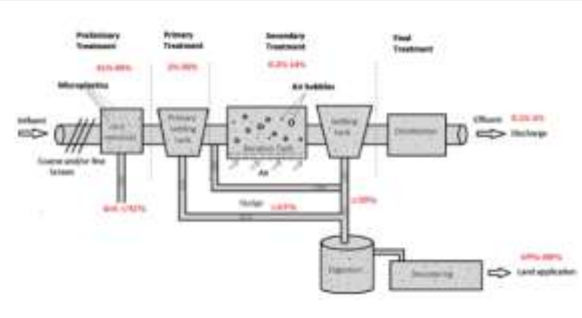

Figure 1. General Representation of a Wastewater Treatment Plant (Turan et al, 2021)

2.2 Microplastic Removal Using Coagulation Technology

Among the various treatment technologies, coagulation–flocculation is one of the most widely applied processes for microplastic removal from wastewater. Iron-, aluminum-, and polymer-based coagulants, including alum, $FeCl_3 \cdot 6H_2O$, polyaluminum chloride (PAC), polyacrylamide (PAM), $Fe_2(SO_4)_3 \cdot 9H_2O$, and $Al_2(SO_4)_3 \cdot 18H_2O$, have been commonly used for this purpose (Rajala et al., 2020; Lu et al., 2021; Zhou et al., 2021; Prokopova et al., 2021; Xue et al., 2021). The coagulation process functions by destabilizing suspended microplastics through adsorption, charge neutralization, and floc formation, thereby enabling their removal via sedimentation. Although different coagulants exhibit varying removal efficiencies, the fundamental mechanisms are generally similar and are strongly influenced by the hydrolyzed coagulant species formed during the hydrolysis process (Zhou et al., 2021).

The main mechanisms involved in microplastic removal during coagulation–flocculation include: (1) adsorption bridging, in which coagulants attach to microplastic surfaces and link particles together to form larger flocs (Lu et al., 2021; Xu et al., 2021); (2) sweeping, whereby microplastics are entrapped within settling coagulant flocs (Zhang et al., 2021); (3) charge neutralization, which reduces electrostatic repulsion on microplastic surfaces and accelerates aggregation, with PAC and $FeCl_3$ reported to achieve zeta potentials close to zero under optimal coagulation conditions (Zhou et al., 2021); (4) hydrophobic interactions between hydrophobic microplastics and coagulant flocs, which may be enhanced by the presence of hydrophobic organic matter and promote aggregate formation (Khan et al., 2023); and (5) aggregation, in which initially small and dispersed microplastic particles combine into larger flocs that can be more readily removed through sedimentation or filtration.

Based on the reviewed literature, it is evident that wastewater treatment plants play a crucial role in reducing microplastic loads; however, their removal performance remains highly variable and strongly dependent on treatment technology. Although coagulation–flocculation has been widely applied and shown promising potential for microplastic removal, existing studies largely focus on overall removal efficiency or coagulant performance, with limited attention given to the influence of microplastic physical characteristics. Systematic evaluations of how particle size distribution and morphological characteristics affect microplastic removal behavior during coagulation–flocculation remain scarce, particularly for industrial wastewater generated from plastic recycling facilities. Therefore, this study aims to investigate the size and shape distribution of microplastics in PET recycling wastewater and to assess how these physical characteristics influence microplastic removal behavior during coagulation–flocculation treatment, in order to strengthen the understanding of treatment performance and support the development of more effective microplastic control strategies.

3. Research Methodology

Sampling in this study was conducted using a modified protocol based on the National Oceanic and Atmospheric Administration (NOAA) method. Samples were obtained from the coagulation-flocculation sedimentation unit and subsequently separated into a liquid fraction (supernatant) and a solid fraction (sediment). The sediment fraction was dried by heating at 150°C for 24 hours, then subjected to an oxidative treatment using the Wet Peroxide Oxidation (WPO) method.

The dried sediment was suspended in 250 mL of distilled water, followed by approximately 30 mL of a 30% hydrogen peroxide (H_2O_2) solution to degrade organic matter, with the volume adjusted according to the sample's organic content. The mixture was allowed to react for approximately 5 minutes and then heated at 75°C for 30 minutes.

Density separation was performed by adding sodium chloride (NaCl) at a ratio of 6 g per 20 mL solution, followed by stirring using a hot stirrer at 300 rpm and a temperature of 75°C for approximately 1 hour. The addition of NaCl increased the solution density to approximately 1.15 g mL^{-1}, thus facilitating flotation and separation of microplastic particles (Masura et al., 2015). The resulting suspension was then filtered using a hydrophobic polytetrafluoroethylene (PTFE) membrane with a pore size of 0.22 µm and a diameter of 47 mm. The filter membrane was dried overnight before being observed using a stereomicroscope (Olympus SZ2-ILST) at 6.7× to 35× magnification, equipped with an OptiLab camera.

Based on preliminary research, the coagulation–flocculation process in the treatment unit was carried out at a pH range of 6–8 with a predetermined optimum coagulant dosage to achieve maximum microplastic removal. Sampling was carried out at three main points in the wastewater treatment system, namely (1) the equalization tank, (2) the buffer tank, and (3) the clarifier tank.

4. Results and Discussion

4.1. Size distribution of microplastics

The size distribution of microplastics was analyzed to identify dominant particle fractions in PET recycling wastewater and to evaluate their relevance to microplastic behavior during treatment processes. Microplastic particles were classified into five size ranges, namely 1–250 µm, 251–500 µm, 501–750 µm, 751–1000 µm, and 1001–5000 µm. This classification provides insight into the size characteristics of microplastics generated during mechanical recycling operations and serves as a basis for assessing the performance of coagulation–flocculation across different particle size fractions.

As shown in Fig. 2, the 251–500 µm size fraction represents the most dominant category, accounting for approximately 40% of the total microplastic particles. This is followed by the smallest fraction (1–250 µm), which contributes nearly 30%, while particles in the 501–750 µm range account for about 20%. Larger microplastics (>750 µm) occur at much lower proportions, with the 751–1000 µm and 1001–5000 µm fractions contributing approximately 7% and less than 5%, respectively.

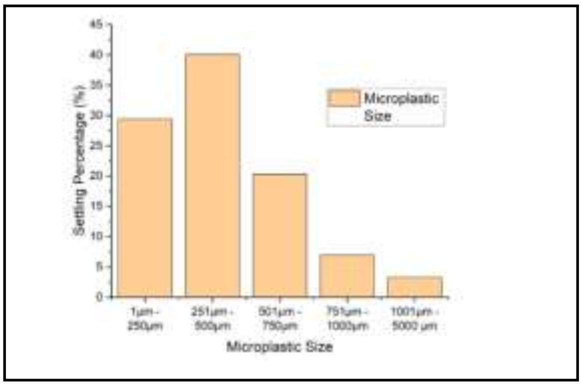

Figure 2. The classification of microplastic sizes

The predominance of medium-sized microplastics (251–500 µm) indicates that mechanical processes such as washing and shredding in PET recycling facilities play a key role in generating fragmentation products within this size range. From a treatment perspective, this size distribution is particularly relevant, as medium-sized particles exhibit a balance between surface area and settling potential. Compared to smaller particles (<250 µm), which tend to remain suspended and resist aggregation, medium-sized microplastics are more readily destabilized and incorporated into flocs during coagulation–flocculation. Conversely, very large particles (>750 µm), although more prone to settling, are present in relatively small proportions and therefore contribute less to overall removal performance. These findings suggest that the dominance of the 251–500 µm fraction may partly explain the effectiveness of coagulation–flocculation observed in PET recycling wastewater treatment.

4.2. Morphological types of microplastics

Microplastic particles identified in the samples were classified into three main morphological types, namely fragments, films, and fibers. Fragment-shaped microplastics were the dominant morphology, accounting for approximately 76% of the total particles, followed by films (21%) and fibers (1.3%). The predominance of fragments is consistent with previous studies conducted in plastic recycling and industrial processing environments, where mechanical abrasion, washing, and shredding promote the fragmentation of rigid plastic materials such as PET bottles (Guo et al., 2022; Suzuki et al., 2022; Brown et al., 2023). Similar fragment-dominated distributions have also been reported in recycling wastewater and sludge, indicating that industrial plastic processing is a major source of fragment-type microplastics.

In contrast, studies conducted in municipal wastewater treatment plants frequently report a higher proportion of fiber-shaped microplastics, often attributed to textile laundering and domestic wastewater inputs (Talvitie et al., 2017; Ziajahromi et al., 2017). The very low abundance of fibers observed in this study therefore represents a notable difference, which can be explained by the industrial origin of the wastewater and the absence of significant textile-related sources. Additionally, differences in pre-treatment processes and sampling locations may further influence the observed morphological distribution.

Microplastic morphology strongly influences removal behavior during coagulation–flocculation. Fragment-shaped particles, due to their irregular surfaces and higher rigidity, provide more effective contact points for adsorption and aggregation, thereby facilitating incorporation into coagulated flocs. Similar observations have been reported by Rajala et al. (2020), who found higher removal efficiencies for fragment-like microplastics compared to fibers during coagulation-based treatment. In contrast, film-shaped microplastics, which are relatively flat and flexible, tend to exhibit lower collision efficiency and weaker attachment to flocs, resulting in less effective removal. Fiber-shaped microplastics, although present in low proportions in this study, are widely reported to exhibit poor settleability due to their high aspect ratio and buoyant behavior, making them more resistant to sedimentation-based removal processes (Talvitie et al., 2017).

Overall, the dominance of fragment-shaped microplastics in PET recycling wastewater suggests favorable conditions for coagulation–flocculation treatment compared to wastewater dominated by fibers or films. The observed similarities and differences relative to previous studies highlight the importance of wastewater source, industrial processing methods, and treatment configuration in determining microplastic morphology and, consequently, removal performance.

4.3. Limitations and Implications for Industrial Wastewater Management

Despite providing valuable insights into the size and morphological characteristics of microplastics in PET recycling wastewater, this study has several limitations. First, the evaluation of removal behavior is primarily based on particle size and morphology distributions, while direct quantification of removal efficiency for each size and shape category was not performed. Second, polymer-specific identification was not included, which may further influence microplastic behavior during coagulation–flocculation. Additionally, the study was conducted under specific operational conditions (pH 6–8 and optimum coagulant dosage), and variations in wastewater composition or treatment conditions may affect the generalizability of the results.

Nevertheless, the findings have important implications for industrial wastewater management. The dominance of medium-sized, fragment-shaped microplastics suggests that coagulation–flocculation is a promising treatment option for PET recycling wastewater, particularly when process conditions are optimized to promote aggregation and settling. Understanding the relationship between microplastic physical characteristics and removal behavior can support the design of more targeted treatment strategies, such as adjusting coagulant type and dosage to enhance the removal of smaller or more flexible particles. Ultimately, this approach can contribute to reducing microplastic discharge from plastic recycling facilities and improving environmental protection.

5. Conclusion

Microplastics in PET recycling wastewater are predominantly medium-sized particles (251–500 µm) with fragment-shaped morphologies, confirming that mechanical recycling processes are the primary source of their generation. Beyond characterizing their occurrence, these findings demonstrate that the dominance of medium-sized, rigid fragments provides favorable conditions for removal through coagulation–flocculation, as such particles exhibit higher aggregation and settling potential compared to smaller or more flexible microplastics.

From a practical perspective, this study highlights the suitability of coagulation–flocculation as an effective treatment strategy for controlling microplastic discharge from PET recycling facilities, particularly when process conditions are optimized. The low proportion of fiber-shaped microplastics further suggests that treatment performance in industrial recycling wastewater may differ significantly from municipal wastewater, where fiber-dominated microplastics often reduce removal efficiency. Consequently, understanding the relationship between microplastic physical characteristics and removal behavior can support the development of more targeted and efficient wastewater treatment designs, contributing to improved environmental protection and more sustainable plastic recycling operations.

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