Occurrence and Removal of Microplastics in Activated Sludge Treatment Systems: A Case Study of a Wastewater Treatment Plant in Thailand

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Abstract. *Domestic wastewater treatment plants involved as a point source of microplastics (MPs) distribution to the environment. The conventional wastewater treatment systems were not designed for MPs removal. Due to immense wastewater effluent discharge, the rate of MPs discharge is possibly high and thus needs to be evaluated. Therefore, a conventional activated sludge wastewater treatment plant at Mahidol University Salaya Campus was selected to investigate the occurrence of MPs and their removal efficiency. The influent and effluent samples were collected and filtered with 100 µm filter bags by using a pump system. The samples were treated with Fenton's reagent, and sodium iodide was used to remove interferences in the environmental matrix. The MPs were manually picked and grouped by size and shape using a stereomicroscope followed by determining the chemical composition using Attenuated Total Reflection-Fourier Transform Infrared ATR-FTIR spectroscopy. The overall concentration of MPs was found to be 0.40 MPs/L in the influent and 0.05 MPs/L in the effluent sample. The treatment plant showed 86.5% efficiency in MP removal. The predominantly detected shapes of MPs were fibers with 86 % and fragments with 85 % removal rate. The detected MPs with the size range of 600-1100 µm were in high abundance in the effluent sample. Additionally, the concentration of larger-sized MPs was significantly lower in the effluent with good removal efficiency. The chemical composition of the detected types of MP fragments revealed PP, PMMA, cellophane, and PET fibers in the effluent. Interestingly, high-density polymers such as PVC, blend PC/PE, and PTFE/P microbead were not detected in the effluent. The results indicate that the small MPs are still released with the effluents and a few types of MPs could be retained in the sludge. Therefore, the understanding of how MPs are released along with effluent wastewater and their composition may help in determining the potential sources of MPs in incoming wastewater to treatment plants.*

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1. Introduction

Microplastics (MPs) are defined as small plastic particles less than 5 mm in size. MPs are categorized into two types—primary and secondary. Primary MPs such as pellets and microbeads are mainly used as raw materials for numerous industrial applications and intentionally added for direct use in consumer products. Secondary MPs result from the degradation of larger plastics due to weathering or mechanical effects [1]. The occurrence of MPs in natural environments has increased due to the rapid growth in synthetic plastic production, increase in application, improper disposal, and mismanagement of plastic waste [2]. There are different sources of MP pollution in urban areas depending on a variety of activities such as degradation of large plastics; agricultural activities (greenhouse films, organic fertilizers); and discharge from wastewater treatment plants receiving influents containing personal care products, paints, laundry detergents, and releases from the tearing of tires, bitumen, and road marking paints, etc.[3]. Domestic wastewater treatment plants (WWTPs) are especially important point sources of MP contamination in the aquatic and soil environment due to the high-volume discharge of effluent wastewater and the possibility of some

MPs being accumulated in the sludge. Although MP removal in WWTPs is high, it is not possible to remove all the MPs as the treatment plants are not designed for that purpose. Previous studies have reported the occurrence of MPs in the range of 0.28 particles L^{-1} to 3.14×10^4 particles L^{-1} in the influents, and 0.01 particles L^{-1} to 2.97×10^2 particles L^{-1} in the effluents from WWTPs. The total load of MP discharge was still found to be considerably high within the range of 5.00×10^5 to 1.39×10^{10} particles per day due to the huge discharge of effluents into the environment [4]. During the wastewater treatment process, some MPs may also be left behind, which get accumulated in the sludge. MPs can thus enter agricultural lands when treated sewage sludge is used as an organic fertilizer. Public awareness of environmental concerns has increased in recent years due to the alarming data on the occurrence of MPs in the environment [3]. MP contamination in freshwater and tap water, and its effect on aquatic organisms has been studied, and the impact of this contamination in Thailand cannot be denied. However, the knowledge of MPs in WWTPs is still limited. Therefore, this study aims to investigate the occurrence and removal of MPs in activated sludge systems, adopted in the urban areas of Thailand. A WWTP at Mahidol University, Salaya campus, Thailand was selected as a case study. The results provide relevant information on the occurrence of MPs in terms of the characteristics and their removal depending on the size, shape, and chemical composition.

2. Methodology

2.1 Study Area

Wastewater samples were collected from a WWTP located at Mahidol University, Salaya Campus, Thailand, that employs an extended aeration-activated sludge system. The treatment plant receives wastewater from a separate sewer system. The WWTP consists of an equalization tank, a grit chamber, two aeration tanks, two sedimentation tanks, and sludge dewatering units. With a treatment capacity of 3,000 m^3/day, it can serve a population of approximately 20,000. The main sources of the wastewater inflow (000 m^3/day) include student dormitories, multi-purpose buildings, cafeterias, and other facilities [5].

2.2 Sample Collection

The sample collection was conducted on 17 November 2020 during the peak time of wastewater inflow between 9:00 to 11:00 am. The influent sample was collected from the outlet of the automatic fine screen and the effluent sample from the outlet of the secondary sedimentation pond. The sample collection was conducted for 30 min at each sampling point by pumping with a submersible pump placed one meter below the water surface. Approximately 750 L sample volume was collected. Subsequently, the collected wastewater samples were filtered with 100 µm pore size filter bags on-site. The bags with filtered samples were then transferred to glass bottles (1L) and transported to the laboratory for further analysis.

2.3 Extraction of MPs

The main purpose of this step was to remove and digest the non-plastic materials that might interfere with the physical and chemical identification steps. The collected bags with filtered samples were washed with deionized (DI) water before being transferred samples into beakers. The obtained samples were again filtered through a 100 µm filter sheet to reduce the volume of cleaning water. The samples were then transferred to beakers by spraying a small amount of DI water (approximately 200-220 mL of final sample volume) for the removal of organic matter removal step. The wet peroxide oxidation (WPO) method was used to remove the organic matter in the samples by using Fenton's reagent in a 1:2 ratio (50 mL of 0.05 M $FeSO_4.7H_2O$ was added first and followed by 100 mL of 30% H_2O_2). The removal of organic matter was conducted at 50°C for 1 h. The filtered samples were then kept overnight for density separation with a 5.3 M sodium iodide (NaI) density solution ($1.52 g/cm^3$). The lighter, floating materials were filtered with Whatman GF/C filter papers (1.2 µm pore size) by using a vacuum filtration unit. The filter papers were subsequently dried at 40°C in the oven for 24 h.

2.4 Quantification and Identification of MPs

The extracted MP particles and fibers on the filter papers were observed. It is to be noted that it was difficult to pick up MP particles and fibers due to the small particle size for chemical identification. It was therefore necessary to only consider the MPs (subsamples) that could be pick up easily from each filter paper using tweezers. These MPs were measured for size and recorded for the shape (based on commonly reported morphology e.g., fibers, fragments, films/flake, and spheres/microbeads). The colors of the MPs were determined with visual sorting under stereomicroscope (Motic Asia, Hong kong) coupled with Miotic Images Plus 3.0The collected suspected MPs, equivalent to approximately 50% of total particles, were chemically identified by (Thermo Nicolet 6700, ATR-FTIR), with diamond crystal and 32 scans. The obtained spectra from FTIR were compared with standard spectra libraries including the Hummel polymer sample library and the HR Nicolet sample library.

2.5 Evaluation of Removal Efficiency of MPs

The occurrence of MPs in wastewater samples was recorded according to the size, shape, color, and chemical composition of the MPs. The MP removal efficiency (RE%) of the WWTP was calculated using the concentrations of MPs counted from the influent (CMPs, infl.) sample and the concentrations of MPs counted from

the effluent (CMPs, effl.) sample by following the equation given below (Eq.1) [6]. The reporting unit used was the number of MPs per liter (MPs/L).

$$\text{Removal Efficiency, RE (\%)} = \frac{(\text{CMPs,infl.}) - (\text{CMPs,effl.}) \times 100\%}{(\text{CMPs,infl})} \quad \text{Eq. (1)}$$

3. Results and Discussion

3.1 Occurrence of MPs and their Removal

The concentration of MPs in the treatment plant was found to be 0.40 MPs/L and 0.05 MPs/L in the influent and effluent samples, respectively, with 86.5% removal efficiency. Considering the limitation in the collection of small MP particles and fibers, 296 MPs from a total of 431 suspected MPs from the influent sample, and 40 MPs from a total of 65 suspected MPs from the effluent sample were chemically identified by FTIR-ATR. The unreported/suspected MPs were observed on the Whatman GF/C filter papers under a stereomicroscope. However, these MPs were not counted and confirmed as MPs due to the difficulty in their collection for the FTIR identification step and due to the unavailability of appropriate chemical identification equipment facilities. However, the results of microplastics concentrations from this study were revealed to be lower estimation, the effluents from WWTPs are still a potential source of MP pollution to the aquatic environment.

3.2 Shape Distribution and the Respective Removal of MPs

The MPs were categorized as fragments, fibers, films/flakes, and spheres/microbeads, and this categorization is presented in Fig. 1. Table 1 presents the occurrence of the MPs in all samples according to their shapes. The shapes were determined to be fragments, fibers, sphere/microbeads, and films. Fragments and fibers were found to be the two dominant shapes, similar to the shape distribution pattern of MPs in a previous study [7]. The removal efficiency for each shape was found to be 100% (sphere/microbeads), 86% (fibers), 85% (fragments), and 75% (films). The removal efficiency was considered good for each shape. Interestingly, the spherical/microbead-shaped MPs were not detected in the effluent sample. These MPs may settle quickly because of their shape settling behavior [8]. Previous studies have also revealed that microbeads could greatly decrease in size when incubated in wastewater. It is therefore easy for microbeads to pass through coarse as well as fine screens [9,10]. The non-detection may therefore be attributed to the microbeads settling down in the sludge or being broken down into smaller MPs. In addition, several black fibers (as synthetic fibers) were also observed. However, the MP fibers retained on the filter papers could not be confirmed as fibers. Therefore, the presented results for MPs shape occurrence and its removal for this WWTP were only based on the MPs that could be identified by FTIR. The main reason was that the fragment MPs shapes could pick up easily however, the small MPs fibers are difficult to pick up for chemical identification. The identification of the shapes is an important factor in determining possible emission sources for MPs. For example, fragment, film/flakes are mainly released from the fragmentation of plastics used as packaging for food, non-food, and other non-packaging purposes [11]. Fiber MPs, on the other hand, are primarily released from laundering activities while spheres/ microbeads are derived from the use of personal care products.

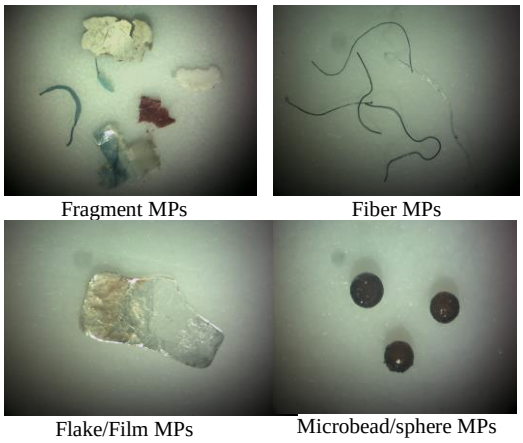


Fig. 1 Categorization of MPs by shape

MPs shape	Number of MPs in influent	Number of MPs in effluent	Removal efficiency (%)
Fragments	184	27	85
Fibers	78	11	86
Sphere/microbeads	26	0	100
Flakes/films	8	2	75

Table 1 Occurrence and removal of MPs by shape

3.3 Occurrence and Removal of MPs by Size

Size measurement is vital to estimate the common size range of the MPs that are released from wastewater treatment plants and to evaluate the removal efficiency of target MPs. The size range and removal efficiency of each MP are categorized and presented in Table 2. The MPs of the size range 600-1100 µm were found in highest abundance in the effluent, followed by MPs of 1600-2100 µm size, 100-600 µm, and 1100-1600 µm. The concentration of the larger-sized MPs was significantly lower in the effluent sample. It may be assumed that the larger-size MPs were efficiently removed by the WWTP. No clear trend for removal efficiency with respect to size range was observed. MPs larger than 2100 µm

demonstrated good removal efficiency while MPs of the size range 1600-2100 µm demonstrated the lowest removal efficiency. As per previous studies [12], MPs of the size range 800-1600 µm (fragments and fibers) were the most detected forms of MPs in aquatic organisms collected from fields. Therefore, the high abundance MPs of sizes 100-2100 µm from the effluent discharge of this plant need to be prioritized to reduce emission.

MPs size (µm)	Number of MPs in influent	Number of MPs in effluent	Removal efficiency (%)
100 - 600	124	8	94
600 - 1100	67	12	82
1100 - 1600	27	6	78
1600 - 2100	26	9	65
2100 - 2600	18	2	89
2600 - 3100	11	1	91
3100 - 3600	7	1	86
3600 - 4100	7	1	86
4100 - 4600	9	1	89

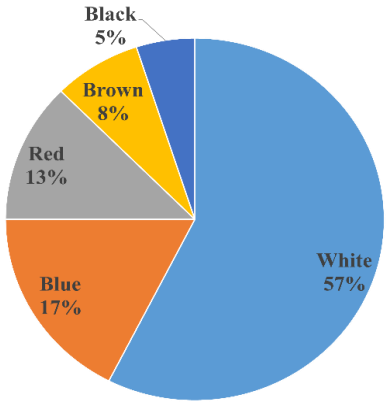
Table 2 Occurrence and removal of MPs by size

3.4 Distribution of MPs by Color and their Potential Impacts

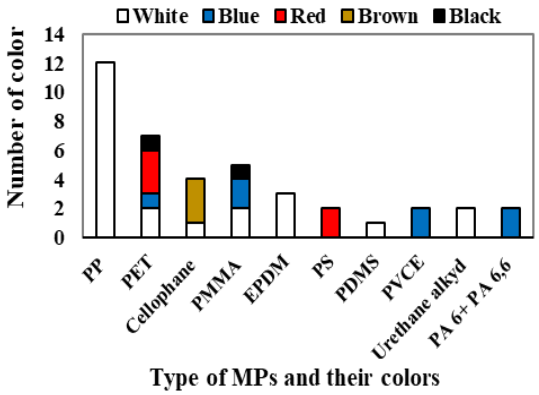
The color distribution and chemical composition of each type of MPs in the effluent sample showed in Fig. 2 (a) and (b). The abundance of MPs according to the colors was found to be white (57%), blue (18%), red (12%), brown (8%), and black (5%). MPs of the most abundant color (white) were composed of PP, PET, cellophane, PMMA, EPDM, and urethane alkyd resin. Interestingly, previous studies assumed that the distribution of MPs colors was not obviously influenced on the MPs removal rate in WWTPs and suggested that the color diversity may not cause a difference in the buoyancy, sedimentation, biofouling, and fragmentation of wastewater-based MPs [13]. In this study, opaque colored microbeads were found in the influent sample. As opaque and white colors MPs are similar to planktons in water bodies, it could be mistaken as a primary food source for feeding fish. Thus, the color of the MPs could possibly highly affect aquatic organisms due to the fish species mistaking the MPs as food [14,10]. The identification of colors could potentially support the determination of potential sources of MPs**.**

3.5 Chemical Composition of MPs

In this study, various chemical composition of MPs was observed and the detected number of MPs in both influent and effluent samples are showed in Fig.3. The different types of polymers in the MPs were identified as polyvinyl chloride (PVC), polyethylene terephthalate (PET), polystyrene (PS), polyethylene (PE), polypropylene (PP), polymethyl methacrylate (PMMA), cellophane, polytetrafluoroethylene: propene (PTFE/P), polyethylene: propylene: diene (EPDM), polyamide 6+polyamide 6,6 (PA6+PA6,6), a blend of polycarbonic acid, carbonate, polyethylene mix (blend PC/PE), urethane alkyd (linseed oil-rich), and polyvinyl chloride ethylene (PVCE). Moreover, a few numbers of other polymer types were also detected in the WWTP i.e., alkyd resin, polyvinyl stearate (PVS), polyester resin (PES), polydimethylsiloxane (PDMS), polycarbonate resin (PC), polyurethane (PU), and diglyceryl ether of bisphenol a mixture (BADGE).



(a)



(b)

Fig. 2 (a) Proportion of MP colors and (b) Observed types of MP colors and their chemical composition in effluent sample.

PVC, PET, PS, PE, PP, PMMA, and PTFE/P were the most common types of MPs that were observed in the influent sample with the total number of occurrences detected as 55, 41, 40, 25, 23, 21 and 20, respectively. These types of MPs were frequently detected in domestic wastewater samples. According to the observed shapes, these MPs could possibly be released from the abrasion of household plastics, food packaging materials, the use of personal care products, and laundering clothes. PVC, the most abundant MP, could have been accidentally released from the abrasion of the water pipeline network facility in the WWTP sampled in the study. Furthermore, the highly abundant MP types such as PP, PET, and PMMA were detected in the numbers of 9, 7 and 5 particles, respectively, in the effluent sample. The observed result of this study was found to be similar to a previous abundance

study in a WWTP in Thailand [15]. Moreover, some types of MPs were also observed in small amounts in both samples as shown in Fig3It is to be noted that the MPs such as PVC, PTFE/P and blend of polycarbonic acid, carbonate, polyethylene mix (blend PC/PE) were not detected in the effluent sample. This may be related to their high densities: PVC (1.16 to 1.58 g/cm^3), PTFE (2.1-2.3 g/cm^3), and blend PC/PE (1.09-1.20 g/cm^3) [16]. Those reported as low-density polymers float in the water surface while high-density polymers settle down rapidly, as observed in a previous study [1]. It may be assumed that the high-density MPs were left behind in the sludge. The density of specific polymers is an important factor that could potentially affect the removal of MPs in WWTPs.

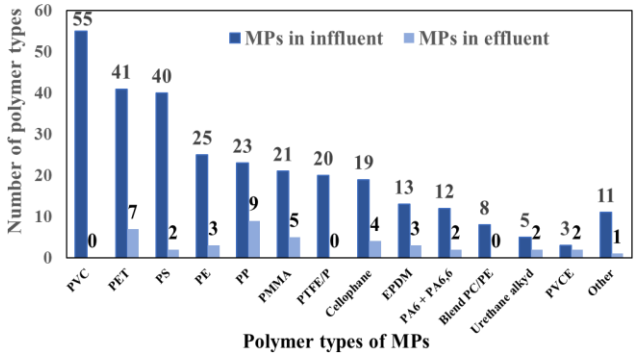


Fig. 3 Type of MPs in the Influent and Effluent Samples

4. Conclusion

Based on the results of this study, the WWTP with an extended activated sludge system demonstrated good efficiency (86.5%) for the removal of MPs. The MPs of sizes ranging between 600-1100 µm were still present in high abundance in the effluent, which is released into water bodies. The predominant shape of MPs present in the effluent was found to be fragmented, and fragment MPs were composed of PP, PMMA, cellophane, PE, PS, and PET. These types of polymers are mainly used for packaging materials. Although the wastewater collection system of this plant has a separate sewer system, retained plastic wastes such as water bottles, cups, straws, and packaging plastics wastes were also observed in the wastewater before automatic fine screen during on-site sampling. Therefore, the high abundance of fragment MPs with specific chemical compositions may be attributed to the fragmentation of larger plastics during the treatment process from the accidental release of plastic waste in the plant. Moreover, the synthetic MP fibers such as PET, PP, and PA6+PA6,6, and microbeads such as PTFE/P, PS were observed as primary MPs. This study indicates the potential sources of microplastic particles and fibers in wastewater and how they are released with the effluents to the environment. The study also discusses the possibility of the MPs being left behind in sludge.

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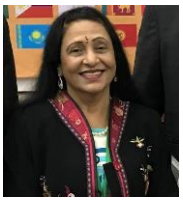

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