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EVALUATION OF MICROPLASTICS IN SEWAGE SLUDGE FROM INDUSTRIAL WASTEWATER TREATMENT ACTIVITIES

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Abstract. The preliminary screening of microplastics (MPs) in sewage sludge from centralized industrial wastewater treatment plants (WWTPs) in Da Nang city of Viet Nam was investigated in this study. The MPs samples were collected in the sewage sludge from two industrial wastewater treatment plants namely Hoa Cam (HC) and Hoa Khanh (HK). The obtained results indicated that the concentrations of microplastics in the sewage sludge of HC and HK were 1,164 and 3,745 particles/kg dry weight, respectively. The microplastic sizes varied from 1.6 to 5,000 µm, of which the size in the range of 1.6 - 100 µm was dominant at HC (42.5 %) and HK (51.8 %). The shape of microplastics was mainly in the form of fragments and fibers. The MPs colour was also observed and the results showed that black, grey and yellow were the main colours of MPs. In addition, MPs chemical composition was determined and the results indicated that polyethylene terephthalate (PET) and polyethylene (PE) were dominant with 36.7 % and 25.2 % for HK and 23.5 % and 25.8 % for HC, respectively. This investigation provides preliminary evidence of MPs in sewage sludge from industrial wastewater treatment activities in Viet Nam, which will be of greater interest in future studies.

Keywords: industrial wastewater, microplastics (MPs), preliminary screening, sewage sludge, chemical composition.

Classification numbers: 3.2.1, 3.3.2.

1. INTRODUCTION

The release of microplastics into nature poses a threat to aquatic and terrestrial ecosystems. Their entry into the food chain endangers human health as well. This problem needs further investigation into the fate of microplastics (MPs) in the environment as the call of the World Health Organization (WHO, 2019) [1]. Wastewater treatment plants (WWTPs) transfer microplastics to the ocean, with a rate of 25 %, from other sources such as road runoff (66 %), wind transfer (7 %), and activities at sea (2 %) [2]. A statistical analysis showed that WWTPs worldwide are introducing 8 trillion microparticles into our aquatic ecosystems every day [3]. Many researches indicated that WWTPs play an important role in releasing MPs from municipal and industrial effluent runoffs into the environment [4–8]. Other studies also found an increase in the concentration of MPs downstream flows from wastewater treatment plants [9–11]. Comparison of plastic fibers detected in coastal sediments and fibers collected from the effluent of WWTPs showed that a significant portion of the microfibrils was found to be associated with the effluent of WWTPs [12]. MPs were detected in all wastewater treatment processes of WWTPs [13]. The differences in MPs concentration can be related to various complex factors, such as sources of wastewater (from industry or municipal, etc.), population served, economy, and human activities. The review article showed that the MPs concentration in industrial WWTPs is higher than that in the municipal with mean values of 5.23×10^3 and 1.27×10^2 particles/L [13]. It is a fact that the technologies in WWTPs are not specifically designed to remove MPs, and MPs have not been concerned in WWTPs, thus, the removal efficiency is different among different technologies. MPs still exist in WWTPs with complexity and diversity in composition and chemical characteristics. When wastewater passes through a treatment plant, most of the MPs are retained in the sewage sludge by settlement process [14–16]. Sewage sludge can be defined as the final solid component produced as a byproduct of wastewater treatment. MPs are considered as an emerging pollutant detected in sewage sludge in recent environmental challenges. Ngo *et al*. [17] showed that some treatment processes can remove MPs by trapping them in the sludge. Mason *et al*. [18] and Murphy *et al*. [19] also found that most of the MPs in wastewater treatment stations are retained in the sludge. Based on the total amount of the sludge, it is estimated that approximately 4.6×10^8 plastic particles are discharged daily from a 10,000 m^3/day WWTP in Finland and the average amount of MPs in the sludge entering the environment reaches about 1.56×10^{14} particles per year [20, 21]. It is noteworthy that MPs can carry pathogens, chemicals pollutants, and potentially invasive species affecting the aquatic ecosystem, food chains, and human beings [22, 23]. Therefore, the occurrence and the fate of MPs have been investigated in previous researches [4, 5, 7, 10 - 12]. The investigated characteristics included concentration, shape, size, colours, and chemical compositions of MPs such as polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC), poly-amide (PA, nylon), polyethylene terephthalate (PET), polystyrene (PS), polycarbonate (PC) in marine water, wastewater, sediment, and sludge waste.

This work aims to investigate the distribution of MPs in sewage sludge from two WWTPs (HC and HK) in Da Nang city, Viet Nam. The characteristics of microplastics are identified and the chemical composition of MPs is determined, which will contribute to the research of microplastics in Viet Nam in the future.

2. MATERIALS AND METHODS

2.1. Sampling sites

Sewage sludge samples were collected from industrial wastewater treatment plants (WWTPs) in Da Nang City, Viet Nam, including Hoa Cam WWTP (HC) and Hoa Khanh WWTP (HK) (Figure 1).

Both plants apply the secondary wastewater treatment method, and the daily treatment capacities are up to 2.000 (HC) and 5.000 (HK) m^3/day. Raw littles and suspended solids in wastewater are retained by grit chamber and primary settling, after that the wastewater is treated by A2O technology (anaerobic - anoxic - oxic) and SBR (sequencing batch reactors) technology at HC and HK WWTPs, respectively. Finally, the wastewater is degraded at the lagoon and disinfected before being discharged into the environment.

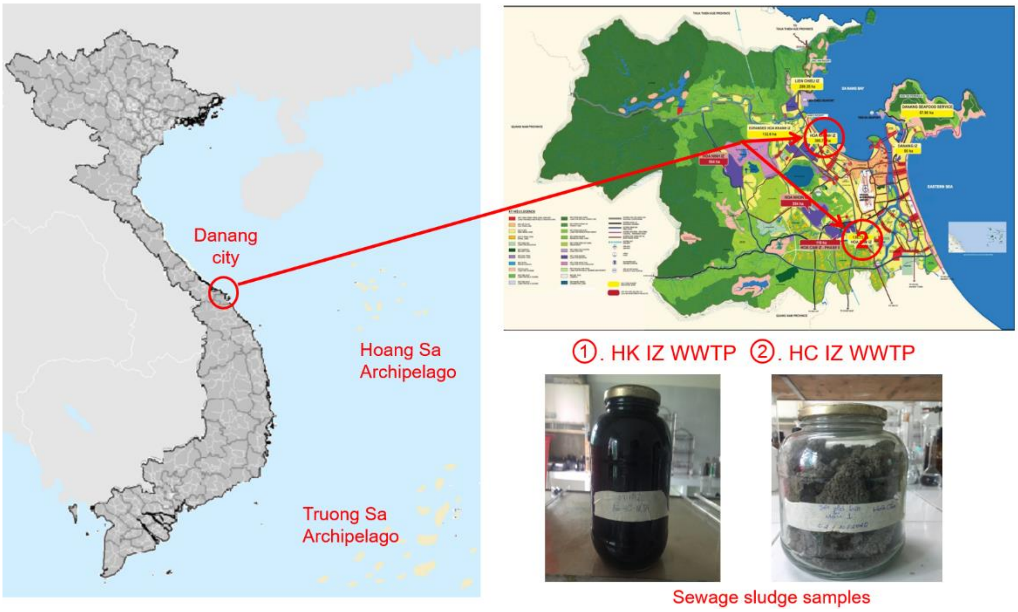


Figure 1. Sewage sludge sampling sites.

2.2. Sampling methods

Methods used to collect and preserve sludge samples are based on TCVN 6663 - 13:2015 - guidance on the sampling of sludge and TCVN 6663 - 15:2004 - guidance on preservation and handling of sludge and sediment samples. At HC WWTP, sludge is collected at drying beds by a metal spoon (composited sample from 5 random locations). For sludge samples of HK WWTP, stainless steel buckets are used to take samples from biological tanks. All samples are contained in 1000 mL glass bottles. After collection, these samples are stored in an insulated container containing HT-Icepack's gel ice packs at a temperature of 5 ^{o}C and transferred to the laboratory for further storage at 4 ^{o}C. Samples are homogenized prior to compositing the samples in triplicate for microplastic analysis.

2.3. Sample extraction and analytical method

Sludge samples were taken from the refrigerator, allowed to cool naturally, and analyzed for microplastics at the laboratory of Danang Environmental Technology Center, belonging to the Institute of Environmental Technology, Vietnam Academy of Science and Technology (VAST). Because polymer mixtures of MPs vary in shape, size and color, the analytical method must identify them all. The microplastic analysis procedure is presented in Figure 2 with MPs size varying from 1.6 to 5,000 μm. This procedure is based on the method of analyzing microplastics in the marine environment of the NOAA Marine Debris Program [24] and inherits the results of a grassroots scientific research project selected by the Institute of Environmental Technology in 2019 [25].

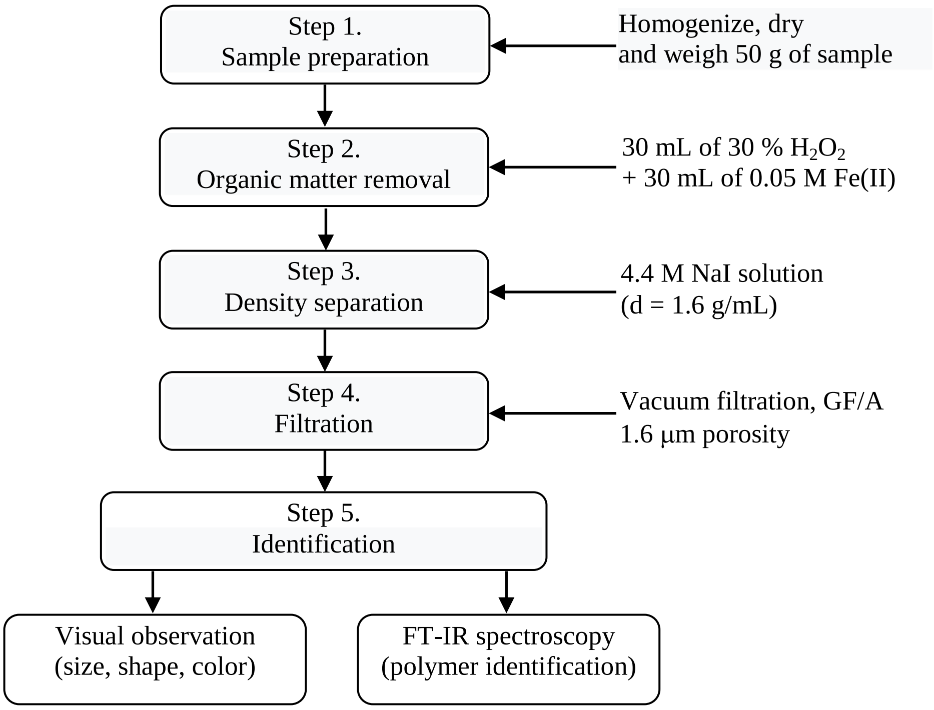


Figure 2. Microplastic analysis procedure in sewage sludge samples.

In step 1, sewage sludge samples were homogenized and dried at 50 - 60 ^oC for 48 - 72 hours in a drying oven (Yamato DX402, Japan). After that, the extraction of microplastics was performed on subsamples of 50 g of dry sludge. For the treatment of natural organic compounds, 30 mL of 30 % hydrogen peroxide (H_2O_2, Merck, Germany) and 30 mL of 0.05 M Fe(II) solution ($FeSO_4.7H_2O$, Merck, Germany) were added to the dried sludge sample, and the mixture obtained was kept at 40 ^oC for 48 hours. This reaction, called Fenton reaction, obtained the highest efficiency in organic matter removal without affecting the extraction of MPs polymers [24, 26]. Then, a density separation step was carried out using a 4.4 M sodium iodide solution with a density of 1.6 g/mL (NaI, Merck, Germany) and all polymers floating on the surface of the mixed solution were separated. Then, the MPs polymers were filtered through a GF/A Glass Microfiber filter with a pore size of 1.6 μm (Whatman, Germany) using a diaphragm vacuum pump (Chemical Resistant Vacuum Pump N 840 FT.18, D-79112 Freiburg, KNF NEUBERGER, Germany). Filtered samples of MPs were used for visual observation (size, shape, and colour) by stereomicroscopy and polymer identification by FTIR spectroscopy.

Microplastic concentration, shape, size, and colour were determined using a Stemi 508 stereo microscope of Carl Zeiss, Germany, with a maximum magnification of 50X by controlling the objective lens and the sharpness of the microscope. Particles were manually sorted from the filtered paper using fine-tip tweezers under a stereo microscope for chemical composition analysis [21]. After visual assessment, the chemical composition of MPs in the sewage sludge samples of two WWTPs was determined by Fourier transform infrared spectroscopy FTIR - 6800, Jasco company, Japan in the mode of attenuated total reflection (ATR) with wavenumbers from 600 to 4000 cm^{-1}, a resolution of 8 cm^{-1}, and a scan number of

16 - 32 times. The obtained infrared spectra peaks of the functional groups from each sample were compared with the relevant standard spectra of common polymers in the spectrum library (Jung *et al*. 2018) [27]. Finally, the obtained MPs for each sample of the sewage sludge were determined after collecting and screening by frequency (cm^{-1}) from the spectral peaks of the MPs samples.

3. RESULTS AND DISCUSSION

3.1. Microplastic concentration

The obtained results indicated that the concentration of microplastics in the sewage sludge of HC and HK was 1,164 and 3,745 particles/kg dry weight, respectively. These results were compared with the research of Liu *et al*. [13] on MPs concentration in sewage sludge of different wastewater treatment processes (columns a, b, c, d, and e) in Figure 3.

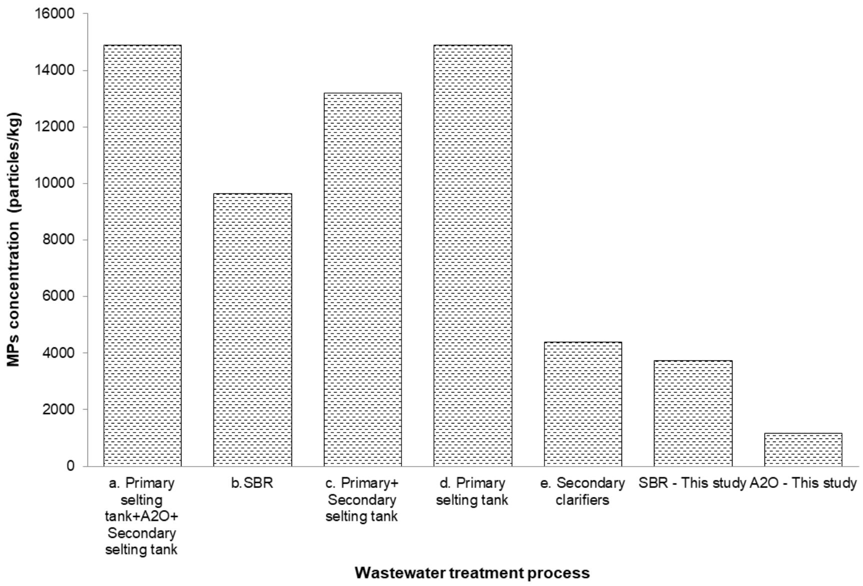


Figure 3. MPs concentrations in sewage sludge of different wastewater treatment processes.

Most of MPs from wastewater were retained in the sludge [13, 16, 17], and subsequently, the MPs concentration in the sludge was much higher than that in the wastewater [7, 13]. Depending on the wastewater treatment technology, the concentrations of MPs in the sludge were different and are shown in Figure 3. The abundance of MPs in the sludge from the primary treatment was higher than that in the secondary process [13]. A research by Sun *et al*. showed that heavy microplastics or microplastics trapped in solid flocs will settle during sanding and gravity separation, while light floating microplastics can only be removed during grease filtration or skimming on the surface of the primary clarifiers [7]. Fibers microplastics are also more easily removed than other shapes during pretreatment because they are more easily entrained in the flocculation particles and separated from the sediment [7, 13]. Thus, the removal efficiency of the treatment is closely related to the characteristics of wastewater and microplastic composition of polymers. Laboratory experimentation in Slovenia indicated that on average 52 % of microbeads were captured by activated sludge, while larger MPs particles were less well retained [28, 29].

The MPs concentrations of HK, where wastewater is treated by SBR technology, and HC, where A2O technology is applied, were the smallest values in comparison with the MPs concentrations in sludge in other studies (Figure 3). In this research, MPs concentration of HC was higher than that of HK. It was explained that different treatment technology resulted in different MPs concentrations in sewage sludge of different wastewater treatment processes. This sewage sludge is often disposed of by landfilling, which can cause microplastics to enter soil and groundwater via leachate, as mentioned in the study by Liu *et al.* [13]. In addition, in the world, sludge is also used in many applications, such as agricultural purposes, soil composting, incineration, etc. Because of the inevitable accumulation of microplastics during wastewater treatment, they have continued to cause environmental impacts on soil, water, and airborne environment [13]. In Norway, 5×10^{11} MP particles were found in the soil where the sewage sludge was applied to agricultural land [30]. Therefore, the study of microplastics concentration in sewage sludge is the basis for appropriate management solutions for wastewater treatment technology.

3.2. Shape, size and color of microplastics

Shape is an important feature for microplastics classification and it affects removal efficiency in treatment plants [31, 32]. There are up to nine shapes of MPs at WWTPs in the world: fiber, fragment, film, pellet, foam, particle, ellipse, line, and flake [13]. Fibers and fragments were the most widely detected MPs in wastewater with a frequency of 91.32 and 65.43 %, respectively [13]. The results obtained from our study were completely consistent with this finding, fiber and fragment MPs were dominant in both HC and HK WWTPs, with a ratio of 67.02 and 19.16 % in HC; 82.08 and 12.08 % in HK, respectively. Other shapes of MPs in sewage sludge in HC and HK WWTPs including pellet, film, and foam were 7.70, 2.85, 3.27 % and 4.12, 1.20, 0.98 %, respectively (Figure 4).

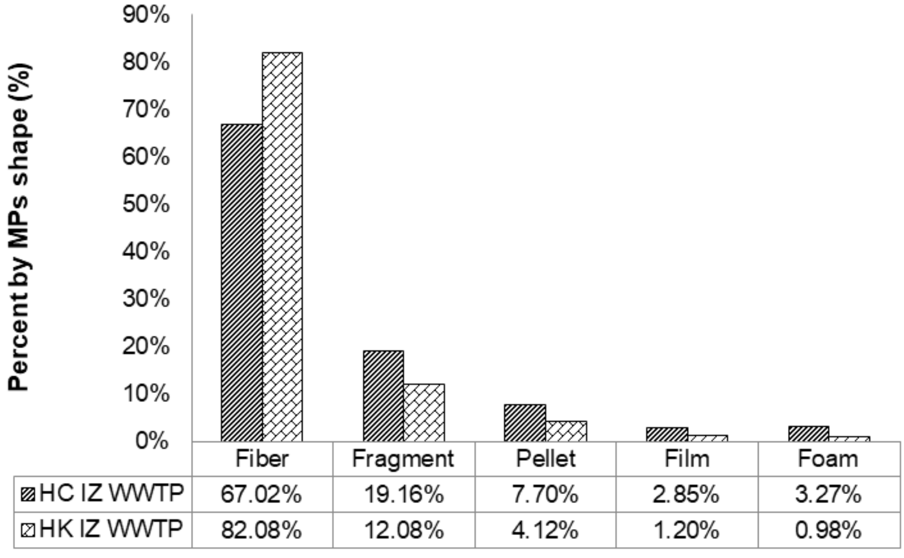
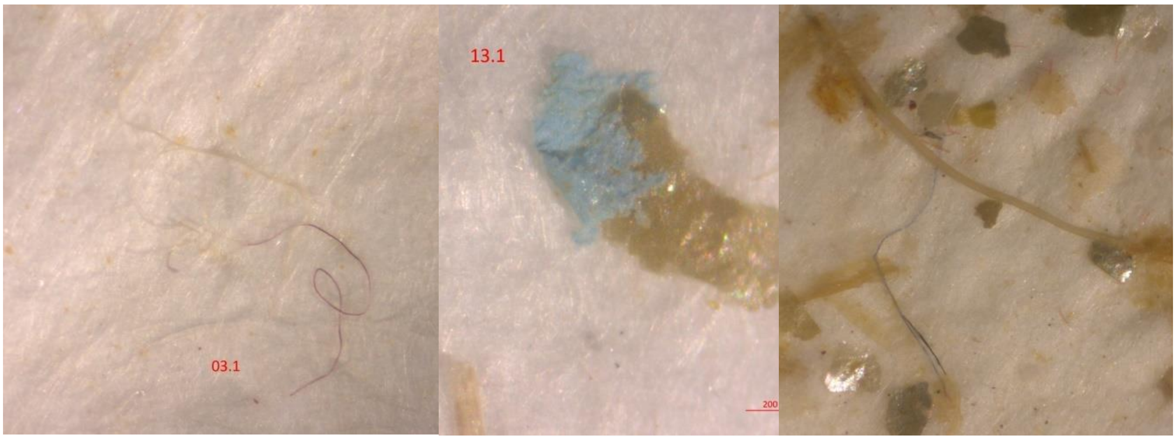


Figure 4. Percentage of MPs shapes of sewage sludge samples (%).

According to the actual situation, there are many factories and companies producing garments, textiles as well as civil plastics for production, daily life, and children's toys leading to more and more plastic waste. During production and processing, a small part of plastics can be lost, scattered, then MPs were collected into the wastewater treatment system and removed, after which they remain in the sludge. That is why MPs are present in sewage sludge at WWTPs.

Images of the MPs shapes are presented in Figure 5 with fiber and fragment shapes of MPs found in HC and HK.



Fiber Fragment Fiber and fragment

Figure 5. Images of MPs shapes.

Similar to the shape factor, the size of MPs is also an important factor affecting their performance and transformation in WWTPs [13, 33]. In the sewage sludge samples, most of the MPs sizes varied from 1.6 to 100 μm, reaching 42.5 and 51.8 % at HC and HK WWTPs, respectively. The size of MPs from 100 to 500 μm was presented with 37.7 % of HC and 27.6 % of HK in sewage sludge. MPs size in the range of 500 to 5000 μm is the lowest, with 19.9 % of HC and 20.7 % of HK. With the smallest size of MPs, the highest ratio of MPs was identified. The results are shown in Figure 6.

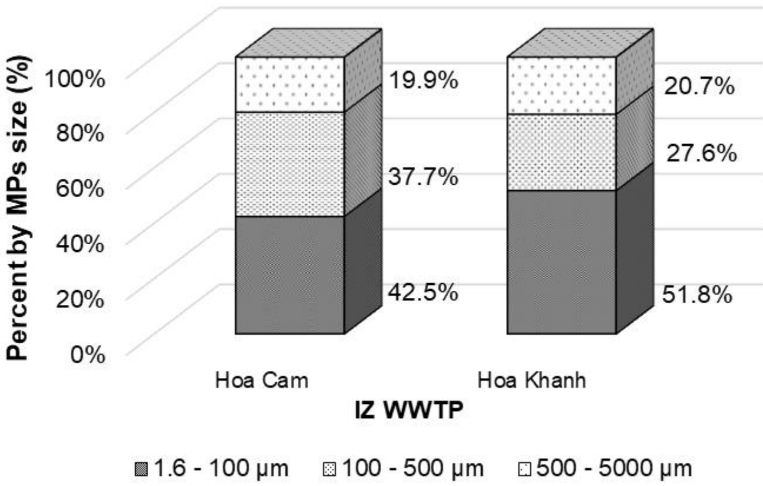


Figure 6. Percentage of MPs sizes of sewage sludge samples (%).

MPs with size less than 0.5 mm were easily trapped in the activated sludge of a bioreactor system by bacteria, while MPs in the size range of 0.5 to 5 mm were easily separated by primary settling [13]. Hence, in this research, the ratio of MPs size in the range of 500 - 5000 μm is the smallest due to the wastewater treatment technology applied which may remove MPs with big size out of the wastewater system and thus in the sewage sludge it was the smallest of the data obtained from HC and HK.

Besides the characteristics of shape and size, the color of MPs is one of the most important parameters of physical characterization of MPs, which can be useful to identify potential sources of plastics as well as potential contamination during sample preparation [34]. Regarding the color of MPs in sewage sludge, dominant black (38.9 and 62.2 %) and yellow colors (39.24 and 34.20 %) at HC and HK WWTPs were presented, respectively. The remaining color scales were grey in both HC and HK (21.59 and 3.60 %), and red in HC WWTP (0.27%) as shown in Figure 7. These colors of microplastics were also detected in sludge samples from a wastewater treatment plant in China from the researches by Li *et al*. [21] and Ren *et al*. [34].

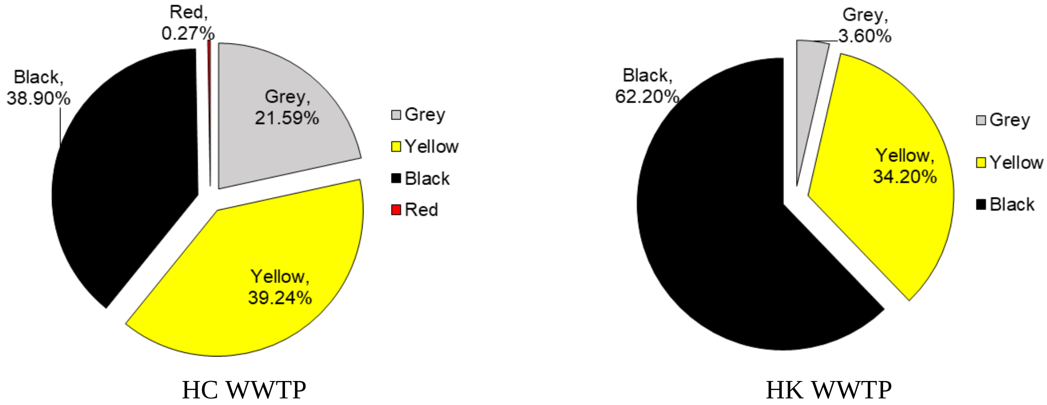


Figure 7. Percentage of MPs colors of sewage sludge samples (%).

3.3. Chemical composition of microplastics

The results of determining MPs polymers in sewage sludge samples from HC and HK WWTPs are presented in Figure 8.

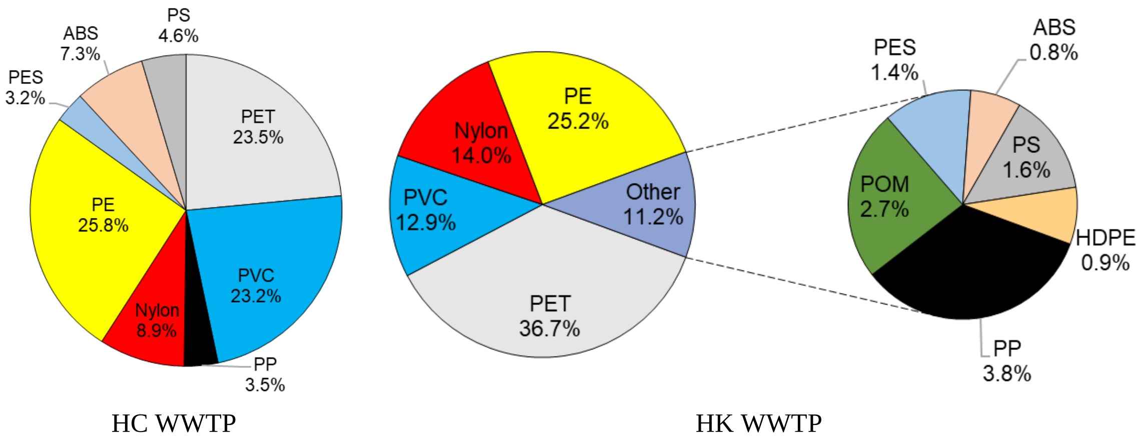


Figure 8. Percentage of MPs polymers in sewage sludge samples (%).

In particular, MPs such as polyethylene terephthalate (PET) and polyethylene (PE) were mainly found with 23.5 and 25.8 % at HC WWTP; 36.7 and 25.2 % at HK WWTP in the sewage sludge samples. In the HC WWTP, the MPs composition was in the following order: PE > PET > PVC (polyvinyl chloride) > Nylon (polyamide) > ABS (Acrylonitrile butadiene styrene) > PS (Polystyrene) > PP (Polypropylene) > PES (Polyester), with PE being the highest (25.8 %). In the HK WWTP, the order of MPs composition was as follows: PET > PE > Nylon > PVC > others, where PET reached 36.7 % in total. Other compounds were analyzed and the obtained results indicated the presence of PES, ABS, HDPE (High density polyethylene), POM (Polyoximethylene), PS and PP in a total of 11.2 %. The data is shown in Figure 8.

The MPs polymer composition at HK WWTP was more diverse because of the diverse presence of industries around the WWTP (up to 50 % of the treated wastewater is industrial production wastewater), while at HC WWTP, domestic wastewater of officials and employees was treated, thus limiting the composition of MPs. Similarly, the study on MPs in the sludge from 8 WWTPs in Norway also showed that the most common polymeric compositions were found to be PE (30.5 %) and PET (26.7 %) [30].

Hence, PE, PP, and PS mainly originated from plastic products, including food packaging bags, plastic bottles, and plastic cutlery [20, 35]. PA, PET and PES microplastics are mainly derived from textiles and general clothing compounds, thus, they are the main sources of household microplastics [7, 36]. Furthermore, mechanical grinding of plastic tire products and the textile industry, etc. were also identified as important sources of microplastic components of PE, PP, PS, and PES [37, 38]. Therefore, the chemical composition of MPs was evaluated in this research.

4. CONCLUSIONS

As a preliminary research on the distribution of MPs in sewage sludge in Viet Nam, this study showed that the concentration of microplastics in the sewage sludge of HC and HK WWTPs was 1,164 and 3,745 particles/kg dry weight, respectively. The characteristics of MPs in sewage sludge samples were identified, such as: the dominant size was 1.6 - 100 µm, the crucial shapes were in the form of fragments and fibers, the main colors were black, grey and yellow. The chemical composition with various polymers of microplastics in sewage sludge of HC and HK WWTPs was evaluated by stereomicroscopy and Fourier transform infrared spectroscopy (FTIR), in which polyethylene terephthalate (PET) and polyethylene (PE) were dominant with 36.7 % and 25.2 % (HK), and 23.5 % and 25.8 % (HC), respectively. This research captured interesting data as it was the first investigation of MPs in sewage sludge in Viet Nam and contributed to MPs research in the development strategy of future MPs management solutions in Viet Nam.

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CRediT authorship contribution statement. Author 1: Methodology, Investigation, Funding acquisition. Authors 2 - 5: Formal analysis, Data systematization, Research outline. Author 1 and Author 6: Manuscript completing.

Declaration of competing interest. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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