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Preliminary study on the occurrence of microplastics in a local sewage treatment plant

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Abstract. Recent studies have found that sewage treatment plants are potential source of microplastics pollution to its receiving freshwaters, which potentially affect freshwater ecosystems. This study aimed to investigate the occurrence of microplastics in sewage treatment plant, i.e., outlet of a primary clarifier and within an activated sludge system. A digestion method (oxidation digestion using Fenton's reagent coupled with wet peroxide oxidation technique) followed by microplastics extraction were attempted. Microplastics were detected, through instrumental characterizations, in all sewage samples in this study with microplastics found in the primary clarifier included polyamide, polytetrafluoroethylene and polyethylene terephthalate, while microplastics in the activated sludge system were polyamide, polystyrene and polyvinyl chloride. Results indicated that the sewage treatment plant successfully reduced microplastics from the primary clarifier to the activated sludge system by retaining bigger sizes of microplastics during early treatment stages of the system.

1. Introduction

Microplastics are tiny fragments and particles that have a length lesser than 5 milimeters (< 5 mm) and may not be visible to our naked eye. According to Bricker et al. [1], microplastics occurrence were resulted from sources such as microbeads in personal care and cosmetic products, pellets used in the manufacturing of feedstock or resin, scrubbers used in abrasive cleaning agents, and plastic powders used for moulding. Microplastics is one of the main ingredients in personal care and cosmetic products, such as in toothpastes, shampoos, cosmetics, and shaving materials, for the purpose of emulsion stability and skin conditioning [2,3]. Meanwhile, secondary microplastics are formed from the degradation of plastics of various sources. Mechanical disintegration, shield off and breakdown of larger plastic fragments from used products such as textiles, paint, or rubber are examples of secondary microplastics [4,5]. Regardless of its sources, these microplastics can be classified into various groups depending on their forms: threads, bits, microbeads, and sheets. Microplastics are chemically polymers, since they consist of polyethylene (PE), polypropylene (PP), polyamide (PA), polyvinylene (PE), polypropylene (PP), polystyrene (PS), Chloride (PVC), and Polyethylene terephthalate (PET) [6].

Microplastics pollution became a major environmental issue just a few years earlier, when the environmental impacts of this form of pollution were documented [7,8,9]. The toxicological problem with these non-biodegradable microplastics is that their particles or fragments will not be decomposed by dissolution and will flow down a drain, getting deposited in the water bodies where it will be more accumulated through biomagnification and bioaccumulation [10]. In the aquatic environment, microplastics are highly flexible (plasticised) and carried to several areas owing to their lighter weight and elasticity [11]. When ingested by aquatic organisms, it can be detriment. Besides, the level of toxicity and physiological consequences of microplastic contaminants on different animals, as well as the danger to human health from the consumption of contaminated water and food, are not well studied [12].

Despite of its adverse impacts, there has not yet been any federal action or legislation explicitly aimed at minimizing the possibility of potential impacts from microplastics on the aquatic ecosystem. Because of the rapid development of the global plastics industry and the amount of plastic being produced, ASEAN have agreed that microplastics will be prohibited in personal care goods by year 2020. During the same period, Asian countries, such as Japan and China, have also begun to rotate their usage of TT500 [13]. However, there has never been any laws and legislations controlling the flow of microplastics through the aquatic environment in Malaysia.

Sewage treatment plants have been identified as the main contributor for the emission of microplastics into the aquatic environment [8,14]. According to Akarsu et al. [15], microplastics typically spread into the domestic atmosphere and these chemicals may escape from treatment facilities, reaching any water bodies. Carr et al. [16] and Peters and Bratton [17] reported detection of significant amounts of multi-coloured microplastics from the water samples obtained from shorelines that have been processed at sewage treatment facilities. Meanwhile, Kang et al. [18] reported that microplastics is difficult and costly to be removed from a sewage treatment plant as it requires huge volume of chemicals and sophisticated equipment to remove it. Therefore, this study was formulated with the aim of investigating the occurrence of microplastics in several stages of a sewage treatment plant, through the specified sewage sampling, sample digestion, and microplastics extraction approaches.

2. Methodology

2.1. Sample collection

Grab samples of sewage were collected from a sewage treatment facility, located in Kuala Lumpur, Malaysia. For a typical sewage treatment plant, processes involved are as follows (* indicates location of sample collection for the present study): **Screening and pumping → Grit removal → Primary settling* → Aeration / activated sludge* → Secondary settling → Filtration → Disinfection**. Two sampling locations were selected, *i.e.*, outlet of a primary clarifier (primary settling) and from an activated sludge system. On-site measuring of three water quality parameters were conducted: pH, dissolved oxygen (DO), and temperature. After that, water samples were collected and stored in the chiller at 4^{o}C.

2.2. Digestion method

Sewage samples need to be digested prior to microplastics extraction, in order to remove any organic matters that may be present. Oxidation digestion required the use of 30% hydrogen peroxide (H_2O_2) with ferrous iron (iron (II) sulfate, $FeSO_4$), which was then known as the Fenton's reagent.

For preparation of ferrous sulphate solution, concentrated sulfuric acid, distilled water and ferrous sulphate powder were used. 0.05M Fe (II) solution and 3 mL of concentrated sulfuric acid were added into 500 mL of water. Then, 7.5 g of ferrous sulphate powder was added into the mixture of concentrated

sulfuric acid with distilled water. The Fenton's reagent was obtained after the solution was well-mixed by using a stirrer.

Next, wet peroxide oxidation technique was employed in accordance with description by Masura et al. [19]. Ratio between the Fenton's reagent with 30 % of hydrogen peroxide was adjusted to 1:2. 250 mL of sewage was poured into 500 mL of beaker. 40 mL of 0.05 M ferrous sulphate solution and 80 mL of 30 % hydrogen peroxide were added to 250 mL of sewage. The mixture was then heated by using a laboratory hotplate at 70 °C with magnetic stirring for 30 minutes until the reaction complete. To ensure the organic matters were completely removed, magnetic stirrer was used instead of glass rod. If the reaction was incomplete, an additional amount of 30 % H_2O_2 were added until all organic matters disappeared.

2.3. Microplastics extraction

Upon completion of the digestion, microplastics were extracted from each sewage sample by using the vacuum filtration method. After the filtration was completed, the pump was turned off and funnel was removed. Next, the filter paper (Grade GF/C Glass Fibre Filter Paper, Diameter 47 mm; Whatman; United Kingdom) will be removed onto petri dishes by using forceps and it was covered with aluminium foil. Filter papers were dried at room temperature for 48 hours before further analysis.

2.4. Microplastics characterization

Physical and chemical characteristics of microplastics were examined by using two types of equipment, *i.e*., digital microscope (Olympus DSX510 Digital Microscope; Olympus; Japan) and Fourier-transform Infrared Spectroscopy (FTIR) (Spotlight 200i FTIR Microscope System; PerkinElmer, Inc; USA). To visualize the microplastics in a digital microscope, 5X magnifications were used. FTIR was used to identify the polymer types of microplastics particles found in the sewage samples. A FTIR spectra could provide information on the chemical functional groups of polymers and may be used to identify chemical changes caused by microbial activity, whether they were caused by consumption or synthesis.

3. Results and discussion

Microplastics was detected in all four sewage samples (two from the primary clarifier and another two from the activated sludge). **Figure 1** to **Figure 4** below are digital microscopy images indicating various shapes and sizes of the microplastics found. In general, the shapes and sizes were found to be various and differ for every different samples. The figures also shown different colours of microplastics, such as orange, yellow, blue, red, white, and green.

For microplastics (fibrous shape) found from the primary clarifier, their range of size were in between 151 to 2,040 µm. Microplastics in fragmented shape were found to be in the size range of 70 to 1,845 µm. Also, pelleted and granular shape microplastics in size range of 46 to 145 µm and 10 to 139 µm, respectively were noticed. Finally, microplastics of film-shaped were observed to be in size ranging from 143 to 580 µm. On the other hand, microplastics of various shapes and sizes were also detected from the activated sludge. Fibrous shape microplastics obtained were in the range of 67 to 1,024 µm. Microplastics in fragmented shape were found ranging from 113 to 990 µm. As for the pelleted and granular shape microplastics, the obtained size were in the range of 67 to 69 µm and 83 to 92 µm, respectively. Lastly, film-shaped microplastics were found in the range of 360 to 536 µm. Noticed that all microplastics of similar shape went through size reduction, moving from primary clarifier to activated sludge. This suggests that the activated sludge process has successfully reduced the size of the microplastics. Similar observations were obtained in the literatures whereby the size of the microplastics could be reduced when they went through different stages of a sewage treatment system [8,15].

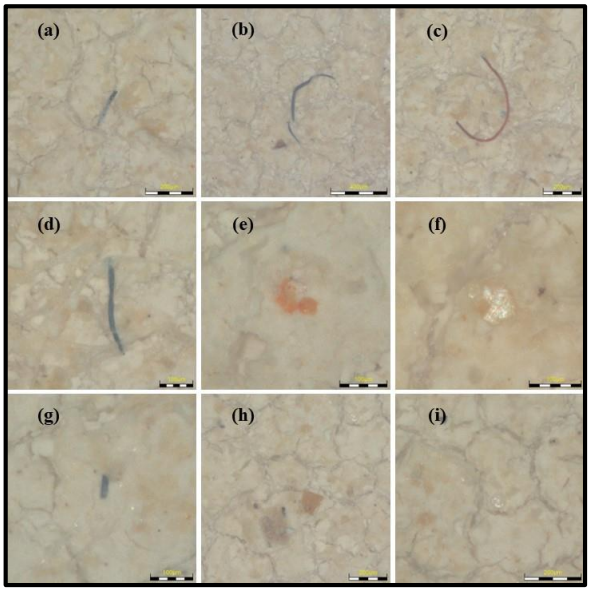

Figure 1(a) to 1(i). Microplastics obtained from sewage samples collected from outlet of the primary clarifier (**Point 1**; 5 x magnifications; Scale bars from (a) to (i): 200 µm, 400 µm, 200 µm, 100 µm, 100 µm, 100 µm, 100 µm, 200 µm, 200 µm)

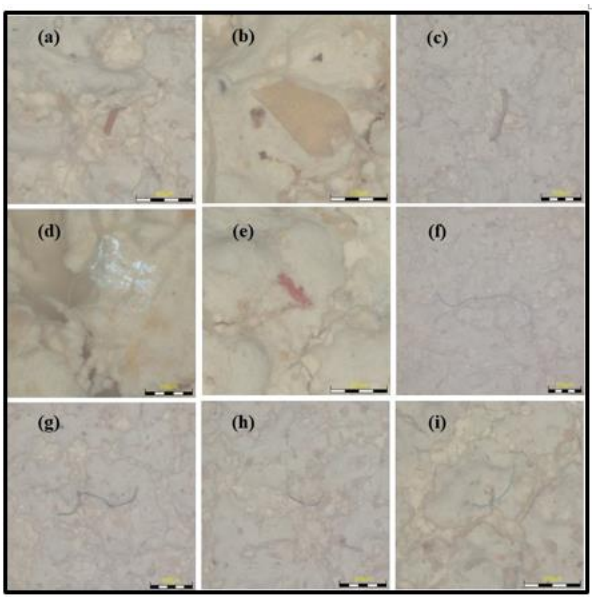

Figure 2(a) to 2(i). Microplastics obtained from sewage samples collected from outlet of the primary clarifier (**Point 2**; 5 x magnifications; Scale bars from (a) to (i): 400 µm, 200 µm, 500 µm, 100 µm, 200 µm, 500 µm, 500 µm, 500 µm, 400 µm)

There are numerous literatures which documented size, shapes, and colours of microplastics. According to Cheung and Fok [20], all the facial scrubs used in the experiment included colourless plastic microbeads, and some of the facial scrubs also contained coloured microbeads in their formulations. Most of the facial scrubs had more than one sort of microbeads in terms of colours. The common colours of microbeads includes light blue, blue, and white. Another study by Praveena et al. [21] reported microplastics of green and colourless were found. On the other hand, Cheung and Fok [20] discovered

that the particle sizes of microbeads ranged from 24 µm (the detection limit) to roughly 800 µm (the maximum size). The mean sizes of the microbeads are between 85 µm and 182 µm, with outliers on the upper end of the size range being unusual. This reflects the homogenous manufacturing of this specific type of primary microplastic, and the mean sizes are generally near to the medians.

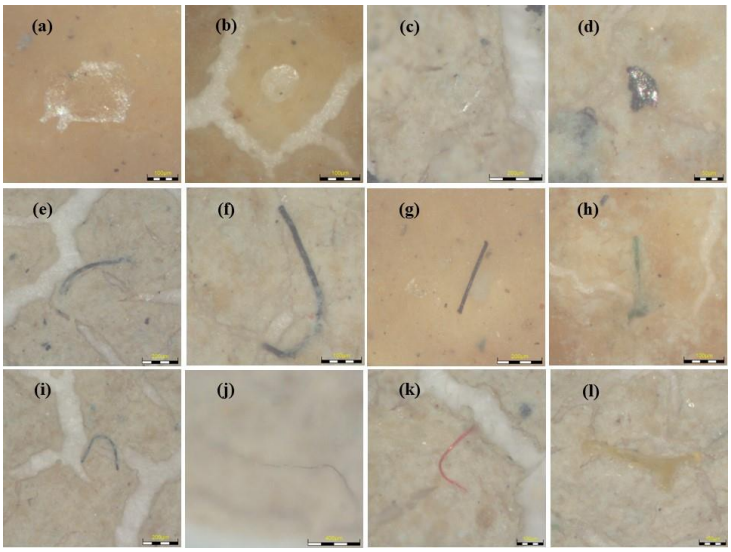

Figure 3(a) to 3(l). Microplastics obtained from sewage samples collected from the activated sludge system (**Point 1**; 5 x magnifications; Scale bars from (a) to (l): 100 µm, 100 µm, 200 µm, 50 µm, 200 µm, 100 µm, 200 µm, 100 µm, 200 µm, 400 µm, 100 µm, 100 µm)

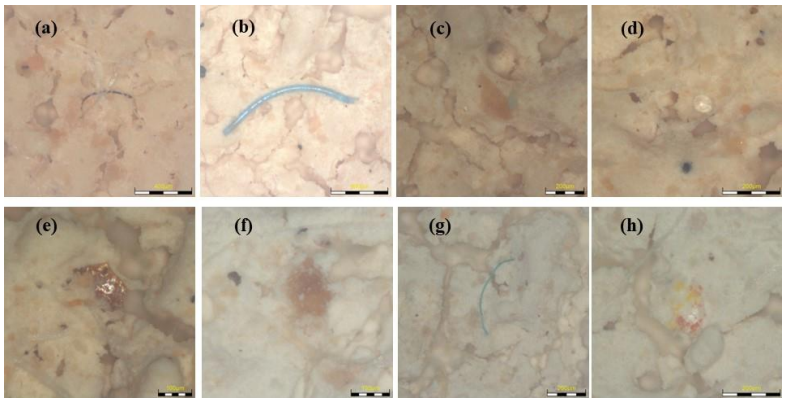

Figure 4(a) to 4(h). Microplastics obtained from sewage samples collected from the activated sludge system (**Point 2**; 5 x magnifications; Scale bars from (a) to (h): 400 µm, 400 µm, 200 µm, 200 µm, 100 µm, 100 µm, 200 µm, 200 µm)

The next characterization approach focused on the chemical characteristics of the microplastics. **Figure 5** portrayed spectrum of microplastics obtained from outlet of the primary clarifier while **Figure 6** showed spectrum of microplastics obtained from scanning microplastics obtained from the activated sludge system. Spectrum indicating microplastics extracted from point 1 of the primary clarifier has an absorption band of 3,000-3,600 cm^{-1} at the first peak. It has a strong and broad vibration which corresponded to the hydroxyl group (O-H). The transmittance percentage of the hydroxyl group was obtained at 84.79 %. The presence of the hydroxyl group could be due to the used of hydrogen peroxide

when conducting the sample digestion procedure as 30% hydrogen peroxide was added. For the second peak obtained for sample extracted from point 1 of primary clarifier, the absorption bands were noticed at 1,500-1,700 cm^{-1} and it has strong vibration which corresponded to the amide group, as the amine group were bonded to the carbonyl group (C=O). Based on the literature reported by Robertson [22], the obtained amide group constituted as one of the functional groups of the polyamide microplastics, whereby this type of thermo-polymer was used for manufacturing of food packaging. The transmittance percentage of the amide group was obtained at 80.45%, slightly differs from the first peak. Next, absorption bands of the last peak were obtained at 1,100-850 cm^{-1}, suggested that the sample has C-F stretching and strong vibration known as the fluoro compound. Fluorine and carbon were covalently bonded in certain chemical configurations of polytetrafluoroethylene [23].

As for spectrum obtained through scanning the second sample from the primary clarifier, the absorption bands of the first peak were obtained at 2,900-3,500 cm^{-1}, suggesting that it has strong and broad vibration which could be corresponded to the hydroxyl group (O-H). The hydroxyl group's transmittance percentage was measured at 76.60 %. Similar to sample from point 1, presence of the hydroxyl group was possibly due to the previous digestion method. For the second peak seen, the absorption bands were found at 2,100-2,200 cm^{-1}. It has significant and strong vibrations that are associated with the thiocyanate (S−C≡N). The transmittance was obtained at high percentage which was at 97.69 %. Also, the C-H bending with weak vibration was obtained as the third peak, proposing that the functional group of that peak was aromatic compound. Aromatic compound and C=C stretching work as functional group of polyethylene terephthalate (PET). At 1,600-1,700 cm^{-1} of absorption bands, C=C stretch with medium vibration was obtained. The alkene group is the most common functional group found in microplastics.

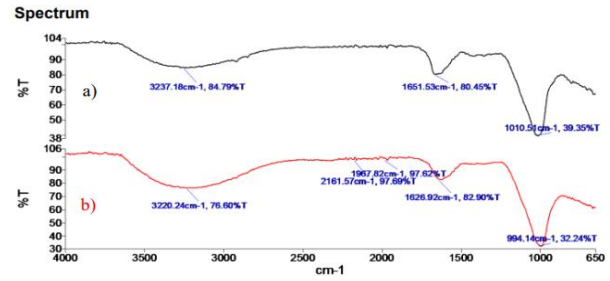

Figure 5. A spectrum obtained through extracted microplastics from the primary clarifier: (a) **Point 1**, (b) **Point 2**

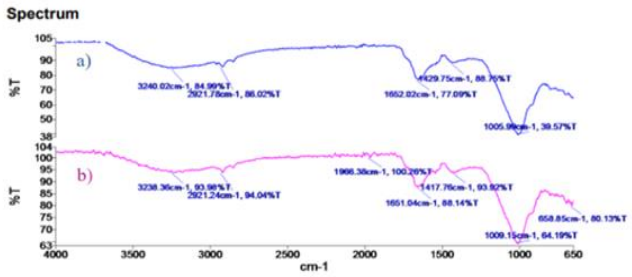

Figure 6. A spectrum obtained through extracted microplastics from the activated sludge system: (a) **Point 1**, (b) **Point 2**

In **Figure 6**, the first peak acquired from both sampling points of the activated sludge system was at 3,000-3,600 cm^{-1}, which has strong and broad vibration that corresponded to the hydroxyl-group (O-H).

Next, the absorption bands for the second peak of both sampling points were found at 2,800-2,950 cm^{-1}, due to its significant C-H stretch and medium vibration, which could be corresponded to the alkane group. The transmittance for second peak of point 1 sample was 86.02 % while point 2 sample was 94.04 %, respectively. The absorption found at the third peak of point 1 sample was obtained at 1,500-1,700 cm^{-1}, which could be corresponded to the amide group as it occurred in strong vibration, suggesting that the type of microplastics obtained was the polyamide. Also, the absorption bands of the fourth peak for point 1 sample was obtained at 1,400-1,450 cm^{-1}, showing carboxylic acid was the functional group as O-H bend and has medium vibration. Since the final peak (point 1 sample) exhibited the C-F stretching and strong vibration, fluoro compound was corresponded as the functional group, with absorption bands in the range of 1,100-850 cm^{-1} (polytetrafluoroethylene). As for point 2 sample, also obtained from the activated sludge system, the third peak was obtained in the range of 1,950-2,000 cm^{-1}, corresponding to the aromatic compound with weak vibration. This obtained functional group suggested that the type of microplastics was the polystyrene. Finally, the absorption of the last peak was obtained at 650-680 cm^{-1}. It was interpreted as the halo compound due to the C-Cl stretching and its strong vibration. Chlorine exists as one of the functional groups of the polyvinyl chloride.

4. Conclusion

Oxidation digestion using Fenton's reagent coupled with wet peroxide oxidation technique for pre-treatment of sewage sample prior to microplastics extraction have successfully acquired microplastics from a sewage treatment plant, i.e., discharge from the primary clarifier and within the activated sludge system. Physical characterization was conducted to evaluate the colour, shapes, and sizes of microplastics obtained, which revealed a reduction in the size of this emerging pollutants during the treatment process. Various shapes of microplastics were found, such as fibrous-shaped, film-like shaped, granular and fragmented shapes, in addition to those in pelleted appearances. Chemical characterization of the extracted microplastics through FTIR revealed different types of polymers presence, with polyethylene, polyvinyl chloride, polytetrafluoroethylene, polystyrene, polyester, and polyamides constituted the majority of the discovered microplastics.

Acknowledgments

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