Analysis and Characterization of Microplastic from Personal Care Products and Surface Water in Bangi, Selangor

(Analisis dan Pencirian Mikroplastik daripada Produk Penjagaan Diri dan Air Permukaan di Bangi, Selangor)

Nur Hapizah Suardy, Nurfaizah Abu Tahrim & Suria Ramli*

ABSTRACT

Primary microplastics which include microbeads are added into cosmetics and personal care products as scrubbing agent and mostly used because of their uniformity and effective exfoliating properties. However due to their small size, microplastic cannot be filtered by the water treatment plant and flow into our waterways posing a great threat to the aquatic life. Therefore, this study aims to detect the presence of microplastics in personal care products and surface water. Microplastic from personal care samples were extracted using warm water at 60 ⁰C and then characterized using ATR - FTIR to determine the polymer composition. Based on the spectrum, the extracted microplastics were confirmed as polyethylene with significance peaks at 2800 - 2900 cm^{-1} (C-H stretching) and medium band at 700 cm^{-1} which indicates the presence of ethylene networks. The extracted microplastics were also identified as polystyrene with the important peaks appear at 3300 and 1600 cm^{-1} with additional weak peak at 1400 cm^{-1} which represent C-H aromatic stretching and C=C aromatic group, respectively. Next, SEM method was used to determine the morphology and size of the microplastics which give sizes ranging from 200-500 μm for each sample. Furthermore, optical microscope was used to determine the color and shape of the microbeads. The results showed that extracted microbeads come from various colors such as white, purple, pink, brown and colorless while the shape observed was spherical, granular, and irregular. Next, insect net and bucket techniques were used in sampling of surface water. The successfully filtered solids were analysed using WPO method and characterize by ATR-FTIR technique. The microplastics found in surface water sample were from various shape including fragment, film, pellet, foam, and tube with the majority come from PE type polymer plastic. Hence, this study proved the presence of microplastic in personal care products that available in Malaysia as well as in surface water.

Keywords: Facial scrubs; microbeads; polyethylene; polymer; polystyrene

ABSTRAK

Mikroplastik primer yang merangkumi mikromanik ditambahkan ke dalam produk kosmetik dan penjagaan diri sebagai agen penyental dan kebanyakannya digunakan kerana keseragaman dan sifat pengelupasan yang berkesan. Namun kerana ukurannya yang kecil, mikroplastik tidak dapat disaring oleh kilang pengolahan air dan mengalir ke saluran air lalu menimbulkan ancaman besar kepada kehidupan air. Oleh itu, kajian ini bertujuan untuk mengesan kehadiran mikroplastik di dalam produk penjagaan diri dan air permukaan. Mikroplastik daripada sampel penjagaan diri diekstrak menggunakan air suam pada suhu 60 ⁰C dan kemudian dicirikan menggunakan ATR - FTIR untuk menentukan komposisi polimer. Berdasarkan spektrum, mikroplastik yang diekstrak disahkan sebagai polietilena dengan puncak penting pada 2800 - 2900 cm^{-1} (peregangan C-H) dan jalur sederhana pada 700 cm^{-1} yang menunjukkan adanya rangkaian etilena. Mikroplastik yang diekstrak juga dikenal pasti sebagai polistirena dengan puncak penting muncul pada 3300 dan 1600 cm^{-1} dengan puncak lemah tambahan pada 1400 cm^{-1} masing-masing mewakili regangan aromatik C-H dan kumpulan aromatik C=C. Seterusnya, kaedah SEM digunakan untuk menentukan morfologi dan ukuran mikroplastik yang memberikan ukuran antara 200-500 μm untuk setiap sampel. Selanjutnya, mikroskop optik digunakan untuk menentukan warna dan bentuk mikromanik. Hasil kajian menunjukkan bahawa mikromanik yang diekstraks berasal daripada pelbagai warna seperti putih, ungu, merah jambu, coklat dan tidak berwarna sedangkan bentuk yang diperhatikan berbentuk bulat, berbutir dan tidak teratur. Seterusnya, teknik jaring serangga dan baldi digunakan dalam pengambilan sampel air permukaan. Pepejal yang berjaya disaring telah dianalisis menggunakan kaedah WPO dan dicirikan oleh teknik ATR-FTIR. Mikroplastik yang terdapat dalam sampel permukaan air adalah daripada pelbagai bentuk termasuk serpihan, filem, pelet, busa dan tiub dan berasal daripada plastik polimer jenis PE. Oleh itu, kajian ini membuktikan adanya mikroplastik dalam produk penjagaan diri yang terdapat di Malaysia dan juga di permukaan air.

Kata kunci: Lulur muka; mikromanik; polietilena; polimer; polisterina

INTRODUCTION

Recently, demand for plastics keep increasing every year due to their versatile properties and low manufacturing cost (Tomasovic 2014). In addition, plastic production increased almost 200 times per year (Ritchie & Rosser 2018) and in 2017 global plastics production reached 348 million metric tons. Nonetheless, increasing amount of plastic production each year coincides with the rising of plastics pollution problem nowadays. Plastics have become one of the main factors of sea pollution and it was reported that more than 13 million plastics flowed into the sea every year and around 100 000 aquatic life died annually (UNEP 2018). Most of the plastics were either come from household waste or being dumped at the landfill directly or indirectly. Improper waste management contribute to water and air pollution problem as well as health problem (Ahmad et al. 2018; Tiew et al. 2019) These wastes were being carried out by wind or rain into the drain, which then moved into the sewage or river and will eventually end up into the sea (Moore 2015). Generally, plastics are known as non-biodegradable materials and require a very long time to decompose. Plastic is preferably used in industry because it is significantly easy to manufacture, low cost production and more resourceful (Noh et al. 2019) Common plastics use in industries including polyethylene (PE), polystyrene (PS), polyvinyl chloride (PVC), polyamide, and nylon.

Furthermore, plastics can be classified based on their size which are macroplastics and microplastics. Macroplastics are polymers with size more than 5 mm, can be seen by naked eyes and do not have direct effect on the food chain (Lehner 2015). Macroplastics are usually used in our daily life include food containers (Iksan & Ahmed 2019), plastic bottles, shopping bags, medical appliances (Rajabi et al. 2019), and electronic devices. Next, microplastics are pieces of plastics with size less than 5 mm to nm and can be divided into primary and secondary classes. Primary microplastics consist of microbeads found in cosmetics and personal care products, resin pellets used in plastics manufacturing process and industrial abrasive materials (Westphalen & Abdelrasoul 2018). Meanwhile, secondary microplastics come from breaking down of larger plastic into smaller pieces (UNEP 2016) that occur due to photodegradation, weathering or hydrolysis process (Lehner 2015). Since 2014, issues on microplastics have become one of the important problems because of their bad effect on the food chain and food web, which ultimately affect human as the main recipient in the food web. Research on microplastics have been widely conducted to understand the real condition of microplastics in marine water, main sources of this problem, the impact into the food chain as well as the toxicology of microplastics toward aquatic life (Carr et al. 2016; Reisser et al. 2013).

Microplastics found in cosmetic and personal care products are known as microbeads which refer to solid particles with size range from 1 - 1000 µm (Leslie 2014). Microbeads can be classified to natural and plastics microbeads. Plastics microbeads are commonly used because they are easily obtained and involve low cost of production. Besides, plastics microbeads are also safe to use and have greater exfoliating properties to remove dead skin (Cosmetic Europe) as well as they can be produced with variety of sizes and provide uniformity to the products (Yelena 2013). Polyethylene is one of the polymers that frequently used in the cosmetics and personal care products which act as adhesive, binder, emulsion stabilizers and can increase the density of the products. The PE compound also mixed with glycol to increase the viscosity and as one of the added values to the products. The mixture used in cosmetic and personal care products is saved and does not have any bad effect towards human skin (Fruitjer-polloth 2015).

Today microplastics from cosmetics and personal care products have become one of the contributors towards the marine pollution problems. Due to their tiny sizes and various colors, aquatic life may consume them as a food. One of the studies carried out in Philippines proved the presence of microplastics in the stomach of the Manila shell sp. *Venerupis philippinarum* (Davidson & Dudas 2016) which showed that microplastics had enter the food chain of marine life. Microplastics become an issue to marine pollution because they are very small and cannot be filtered out during the water treatment process, which then flowed into the marine waterways (Westphalen & Abdelrasoul 2018). Microplastics were found in the sample after water treatment process from one of the treatment plants in US and from the sample of the river which received the effluent from the same plant (Wu et al. 2017). Research in primary microplastics are still less conducted and consider as insignificant (Cheung & Fok 2017) especially in Malaysia. In this study, Tasik Cempaka and Sungai Langat which are located in Bangi were chosen because of their important role in water supply, fishing, waste disposal, agriculture and industry (Gasim et al. 2009). Sungai Langat is one of the most important water sources for two-third state in Selangor (Hamzah et al. 2019). Water quality for Sungai Langat and Tasik Cempaka were classified under class III which mean slightly polluted (Ahmad et al. 2015; Basheer et al. 2017). Study to determine the presence of microplastics in Malaysia marine water are crucial to determine the water condition and method to overcome this problem. Therefore, this study was carried out to investigate the presence of microplastics in personal care products and to determine either microplastics have contributed towards plastic pollution in Malaysia's marine water.

MATERIALS AND METHODS

CHEMICALS

The chemicals and solvent used in this study included iron sulphate ($FeSO_4$), hydrogen peroxide (H_2O_2) and sodium chloride ($NaCl$) were purchased from Sigma Aldrich.

SELECTION OF COSMETICS AND PERSONAL CARE PRODUCTS

Six samples of personal care products were obtained from local market and labelled with A-F letter, respectively, which include toothpaste, facial, and body scrub. The products were selected based on the top selling brands in each shop and some of the products have also stated polyethylene in their ingredients. Other than that, the products were also chosen based on the survey and study conducted by 'Beat the Microbead' organization which listed out the beauty product that contain microplastics.

EXTRACTION OF MICROPLASTICS FROM PERSONAL CARE PRODUCTS

Sample A (20 mL) was added into warm water (60 ℃) and stirred with glass rod until the soluble portion was dissolved. The mixture was left overnight to ensure that it was completely dissolved. Then, the retained microbeads were filtered using vacuum pump and dried in the oven at 45-50 ℃ for 24 h. All the steps were repeated for the other five samples.

WATER SAMPLING METHOD

Water samples were taken from Sungai Langat (Figure 1) and Tasik Cempaka at four different locations (Figure 2) to get a good sample representative. Coordinate for sampling points in Sungai Langat and Tasik Cempaka are stated in Tables 1 and 2, respectively. The sampling method was divided using two techniques depending on the location of the sampling. First, bucket technique is used for sampling in the lake area where the bucket was thrown into the lake and the water will fill up the bucket before it was transferred into 1 L sampled bottle. The water sample was filtered directly, and the solid particles were transferred into the beaker and covered with aluminium foil. While the second method was using an insect net which applies in the river water. The insect net was put into the river water for 1 h approximately. Then, all the retained microplastics were washed into the beaker and covered with aluminium foil. All the retained solid particles from both samples were brought into the lab for further analysis. Furthermore, the different sampling method was used for different location depends on the condition of the water. Insect net technique was used for sampling in river water, while bucket method was used in lake area. Next, all the water samples were filtered, and the solid particles obtained were dried in the oven overnight. After completely dried, the samples were further analyzed using wet peroxide oxidation (WPO) technique where the solid particles were differentiated by density. All the suspected microplastics floated onto the mixture of the solution and filtered again before dried until constant weight.
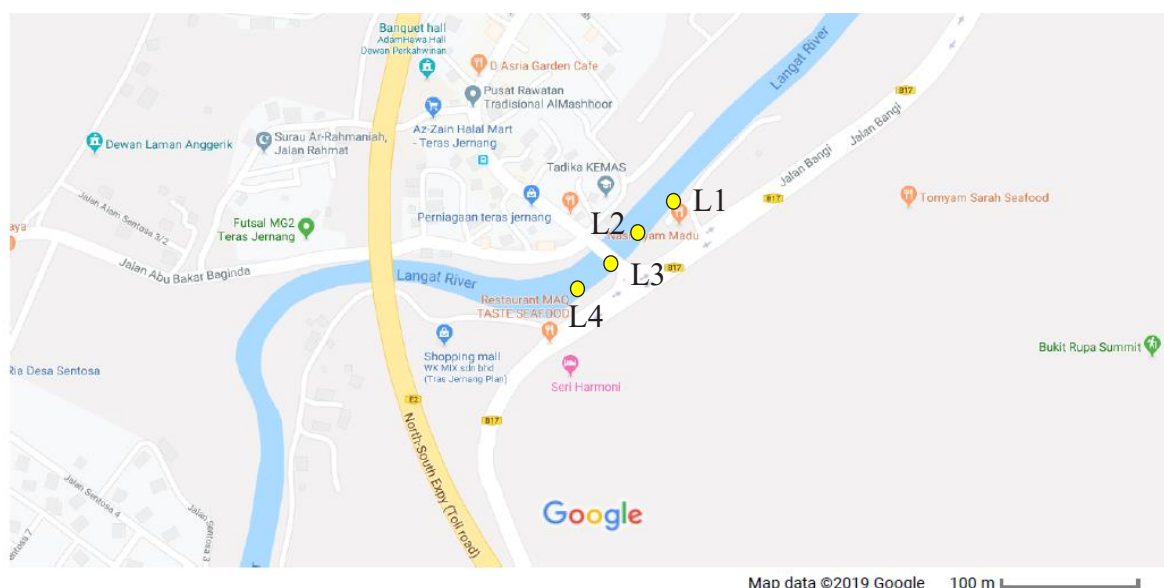

FIGURE 1. Sampling point at Sungai Langat

TABLE 1. Coordinate for sampling point at Sungai Langat

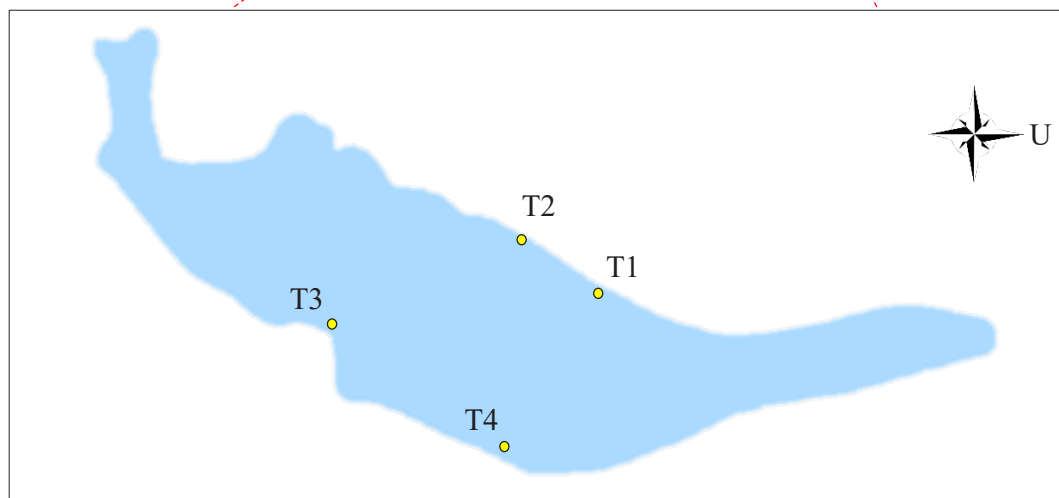| Points	Longitude (N)	Latitude (E)
L1	2°55'54.1"	101°46'33.3"
L2	2°55'55.1"	101°46'35.0"
L3	2°55'52.8"	101°46'31.9"
L4	2°55'52.3"	101°46'30.9"

FIGURE 2. Sampling point at Tasik Cempaka

TABLE 2. Coordinate for Sampling Point at Tasik Cempaka

Points	Longitude (N)	Latitude (E)
T1	2°57'37.1"	101°45'34.3"
T2	2°57'36.5"	101°45'33.9"
T3	2°57'30.8"	101°45'35.1"
T4	2°57'35.6"	101°45'37.9"

REMOVAL OF NON-PLASTICS MATERIAL

Method for water samples analysis was referred with slight modifications from a study by Masura et al. (2015) and Westphalen and Abdelrasoul (2015). Generally, the method is known as wet peroxide oxidation (WPO) technique which used to remove non-plastics material from the samples. The water samples were filtered using a stainless steel filter with a size of 37 μm. All the solid particles were dried in the oven at 40-50 °C for 24 h. After that, 20 mL of $FeSO_4$ solution (0.05 M) was poured into the beaker contained all the filtered solid particles. Next, around 20 mL of H_2O_2 solution was poured into the mixture and slowly stirred using a glass rod. The mixture was covered with watch glass and put at room temperature for 5 min. The mixture then was heated and stirred at 75 °C and the reaction was observed. The mixture was heated for 30 min and if there was any possibility of spilling, distilled water was added to slow the reaction. Then, 6 g of NaCl was added for every 20 mL of the mixture and heated back to dissolve all the salt particles. Lastly, the mixture was cooled at room temperature before being filtered and all the microplastics was dried in the oven.

IDENTIFICATION AND VISUALIZATION OF MICROPLASTICS

The microplastics obtained from personal care products and surface water samples were observed under the optical microscope at 400× magnification to identify their shape and color. Then, Attenuated Total Reflection Fourier Transform Infrared, ATR-FTIR (model Agilent Cary 630 FTIR Spectrometer) was used to determine the polymer composition and no preparation sample needed. Next, the results were compared with the library spectra (Jung et al. 2018; Khalik et al. 2018). Furthermore, Scanning Electron Microscopy, SEM (model Carl Zeiss Evo Ma 10 UK) was used to confirm the morphology of the microplastics in the personal care products and cosmetics

and waterways. Microplastics pieces were mounted onto the aluminium SEM stubs using double sided carbon sticky tabs. Samples were then coated with gold using a sputter coater and ready to be analyzed.

RESULTS AND DISCUSSION

EXTRACTION AND IDENTIFICATION OF MICROPLASTICS

Microplastics extracted from personal care products and surface water samples were observed under the optical microscope. From the result, microplastics extracted from personal care products have different shapes of microplastics including spherical, granular, and irregular shape, while the colors were varying from each sample such as orange, white, pink, purple, and colorless (Table 3). Different brands of products have different color and shape of microplastics. These results were similar with finding by previous study (Cheung & Fok 2017; Fendall & Sewell 2009; Napper et al. 2015).

Next, extracted microplastics from water samples were observed under optical microscope to determine its shape (Figure 3) and color (Table 4). The microplastic particles that was successfully filtered were from fragment, granule, tube, film, foam, and pellet shape. These results were similar with finding by Eriksen et al. (2013), Khalik et al. (2018), Mason et al. (2016), and Zhao et al. (2014). From the river sample, 12 pieces (pcs) of microplastics were founded with red, blue, green, white and grey color. Meanwhile a total of 5 pcs microplastics were found in Tasik Cempaka with blue, green and white color only. The existence of microplastics in the water sample showed that these particles were released into the marine environment because they were not filtered out during water treatment process or indirectly released from the household and industrial waste. All plastic particles with size less than 5 mm were continued for further analysis.

TABLE 3. Color and shape of the extracted microplastics from personal care product
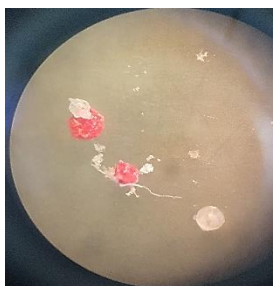
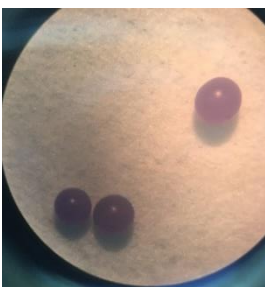| Samples	A	B	C
Image			
Color	Orange White	Orange Brown Colorless	Pink Colorless
Shape	Irregular Granular	Irregular Granular	Granular Irregular
Samples	D	E	F
Image			
Color	Brown	Purple	White Colorless
Shape	Irregular	Spherical	Irregular Granular

TABLE 4. Classification of microplastics based on colour

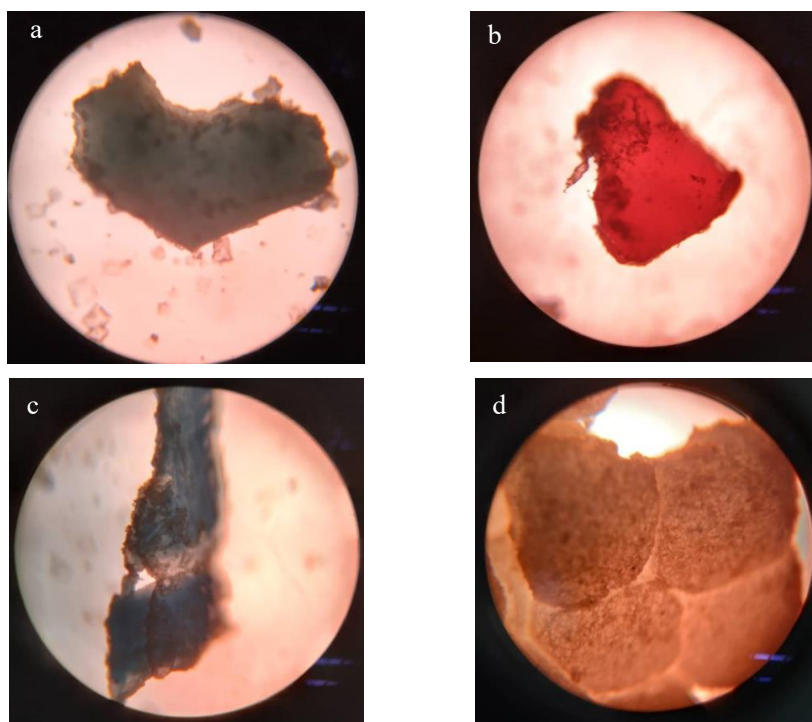| Sampling location	Classification based on color (pcs)					Total number of microplastics (pcs)
	Red	Blue	Green	White	Grey	
Sungai Langat	5	2	1	3	1	12
Tasik Cempaka	-	2	2	1	-	5

FIGURE 3. Image of microplastics sample under optical microscope,
(a) (b) fragment (c) film, and (d) foam

SCANNING ELECTRON MICROSCOPY ANALYSIS

SEM method was used to determine the morphology and size of the extracted microplastic particles. Samples A and B were chosen as the representative for PS and PE, respectively. Visualization of extracted microplastics from personal care products can be differentiated between smooth and rough forms (Napper et al. 2015). Sample A shows a rough surface of microplastics (Figure 4), while sample B have both smooth and rough forms of microplastics (Figure 5). Other than that, from SEM technique the size of the microplastics particles also determined which ranging from 200-500 µm. These sizes match with the definition of microbeads stated by Leslie (2014), where microplastics are small particles found in the cosmetic and personal care products with size form 1-1000 µm. Figure 6 shows the SEM images for river and lake sample. From the result, the sizes of the filtered microplastics was 4.9 for river sample and 3.1 mm for lake sample. Only two samples from both river and lake samples were determined by SEM because the mass for other samples were not enough to be analyze. Hence, these two samples were taken to represent the samples from each location.
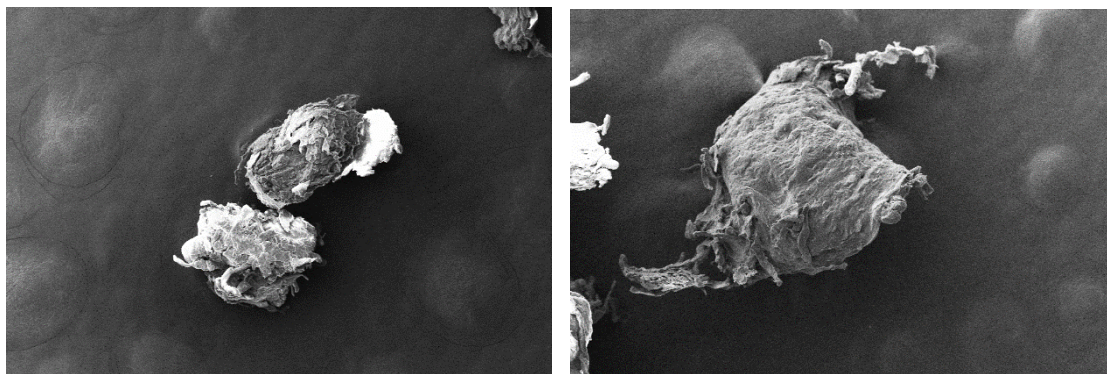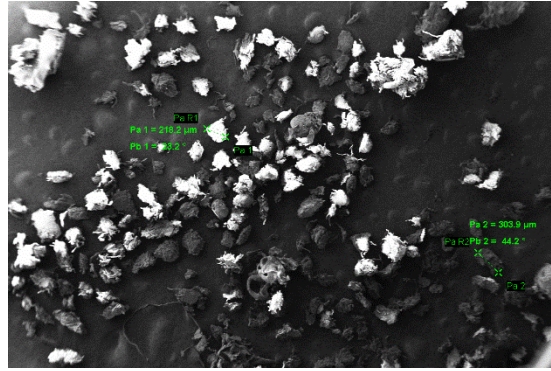
FIGURE 4. SEM Image for extracted microplastics from sample A with
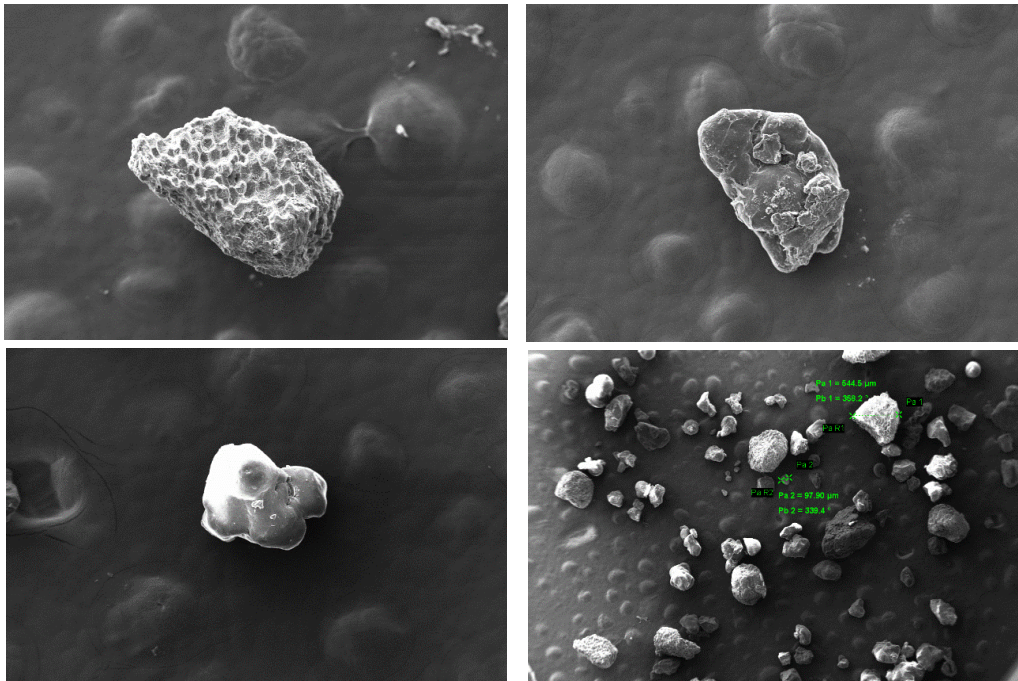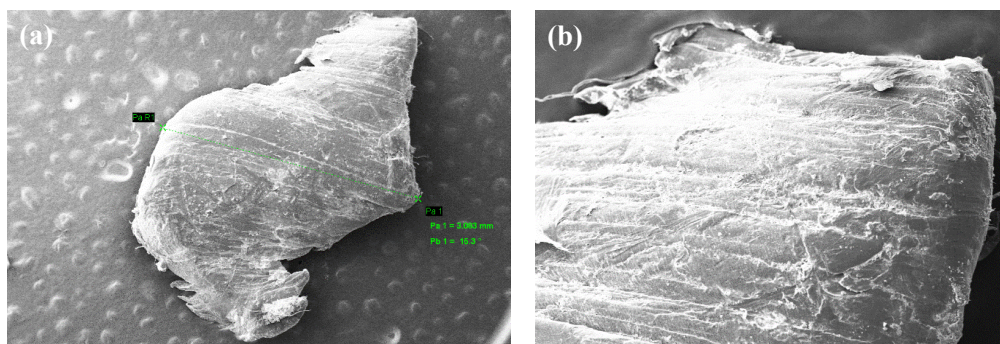100× magnifications

FIGURE 5. SEM Image for extracted microplastics from sample B with
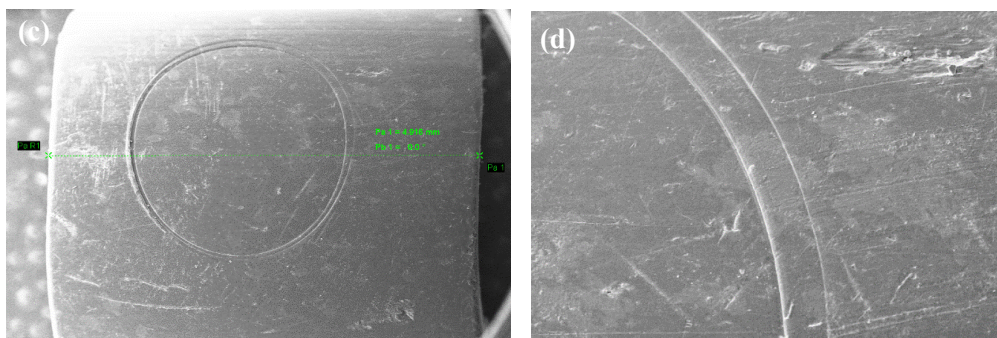100× magnifications

FIGURE 6. SEM image for extracted microplastics from river sample
(a, b) and lake sample (c, d) with 100× magnifications

ATTENUATED TOTAL REFLECTION FOURIER TRANSFORM
INFRARED ANALYSIS

Characterization of the microplastics sample were continued with ATR-FTIR to determine the polymer composition in each sample. ATR-FTIR results showed that sample A and D (Figure 7) came from polystyrene (PS) which shows the significant peaks including medium band around 3340 cm^{-1} for aromatic C-H with additional strong peak in fingerprint region at 1036 cm^{-1}. Absorption band for aromatic C=C stretching also was observed at 1634 and 1448 cm^{-1} (Table 5) (Jung et al. 2018; Khalik et al. 2018).

Next, from IR spectra sample B, C, E, and F were made up of polyethylene (PE). The IR analysis also conducted for stock PE as the reference peak (Figure 8). In addition, microplastics particles found in water samples also come from PE polymer type (Figure 9). The significant peaks were shown at 2920 and 2851 cm^{-1} for C-H$_2$ bending, 1463 and 1403 cm^{-1} for C-H$_2$ deformation with medium band, while weak band was observed at 1103 cm^{-1} for -H-C-H- twisting and wagging and peak at fingerprint region (724 cm^{-1}) with medium band was spotted which proved the present of ethylene network in the compound. Results for individual wavenumber for each sample were shown in Tables 6 and 7 for both personal care products and cosmetics as well as water samples, respectively.
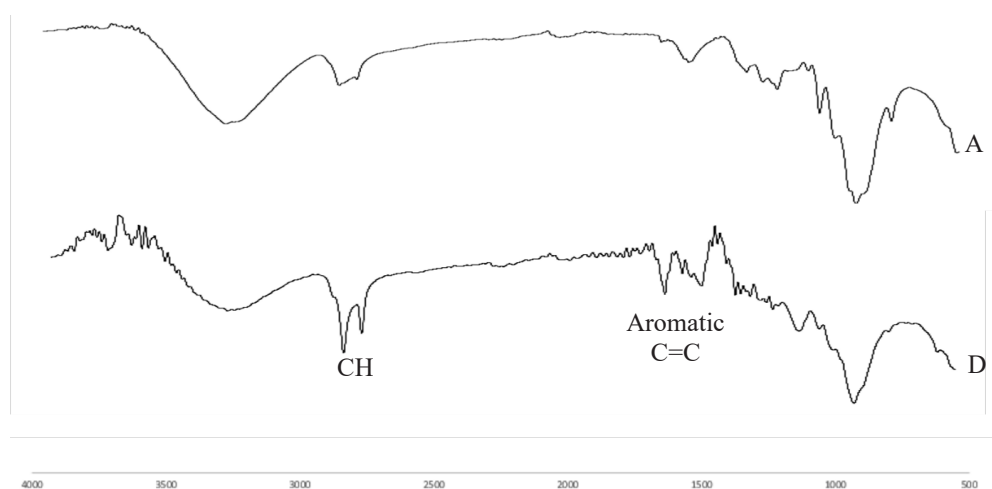

FIGURE 7. The IR spectra for microplastics extracted from
personal care products sample A & D

TABLE 5. Data comparison for IR result with reference peak for PS with sample A & D

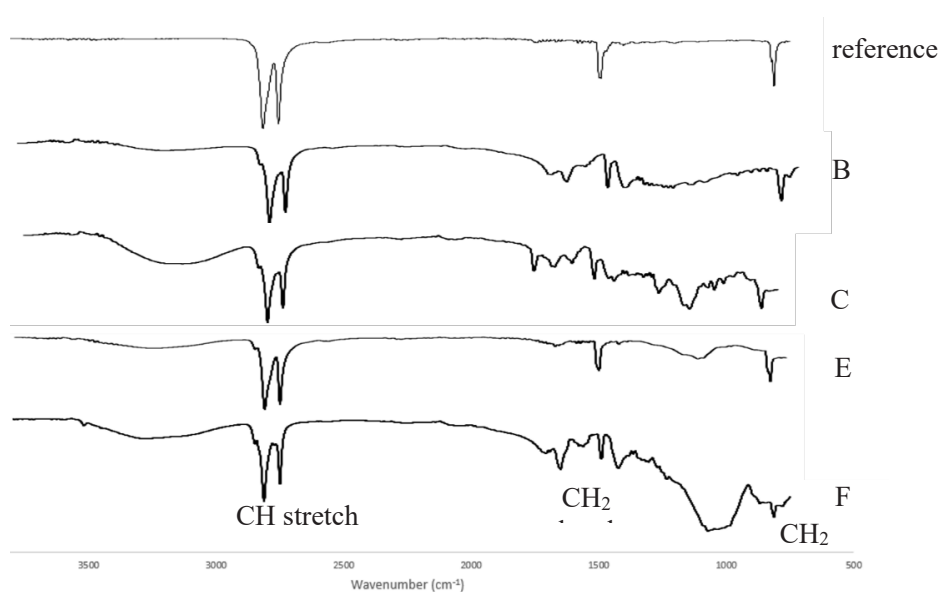| Functional group	Reference frequency (cm^{-1}) (Jung et al. 2018; Khalik et al. 2018)	Sample A (cm^{-1})	Sample D (cm^{-1})
C-C aromatic stretching	3200 - 3367	3340	3314
CH$_2$ stretching	2900 - 2800	2915-2851	2915-2844
C=C aromatic stretching	1637 1523 & 1409	1634 1447	1724 1589 & 1464
C-H aromatic stretching (oop)	1105 - 1033	1036	1023

FIGURE 8. The IR spectra for microplastic samples
extracted from personal care products sample B, C, E & F

TABLE 6. Data comparison for IR result with reference peak for stock PE with sample B, C, E & F

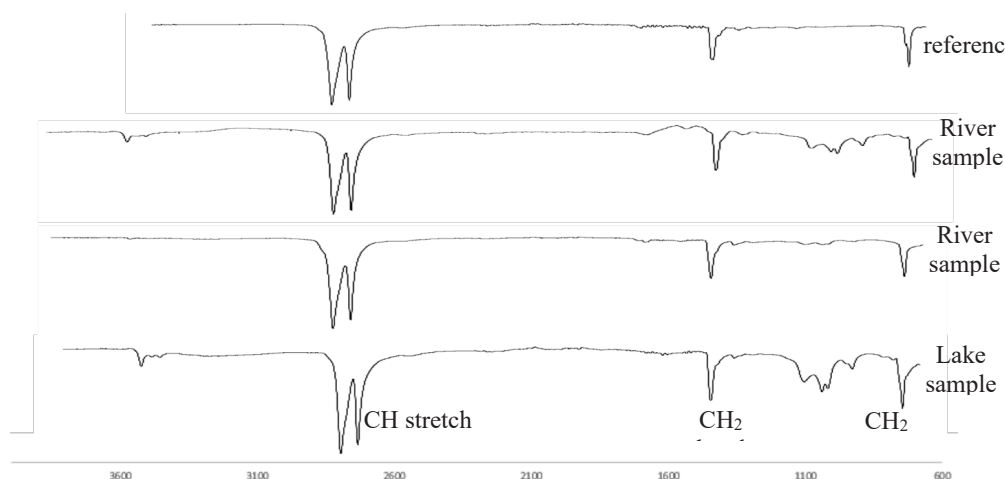| Functional group	Reference frequency (stock PE)	Sample B (cm^{-1})	Sample C (cm^{-1})	Sample E (cm^{-1})	Sample F (cm^{-1})
C-H sp^3 stretching	2911 & 2845	2920 & 2851	2919 & 2851	2910 & 2846	2912 & 2845
CH$_2$ bending	1460	1463 & 1403	1468 & 1398	1460	1472 & 1385
CH$_3$ bending	1361	1382	1375	1374	1389
Stretching H-C-H deformation	1144	1103	1066	1030	1009
Stretching CH$_2$-CH$_2$ network	715	724	724	714	720

FIGURE 9. The IR spectra for microplastic extracted from
water samples

TABLE 7. Data comparison for IR result with reference peak for stock PE with water samples

Functional group	Reference frequency (stock PE)	River 1 (cm⁻¹)	River 2 (cm⁻¹)	Lake 1 (cm⁻¹)
C-H sp³ stretching	2911 & 2845	2920 & 2851	2922 & 2853	2910 & 2846
CH₂ bending	1460	1472	1470	1460
CH₃ bending	1361	1366	1380	1379
Stretching H-C-H deformation	1144	1012	1050	1039
Stretching CH₂-CH₂ network	715	719	724	721

CONCLUSION

Microplastics pollution have been widely studied because of their adverse effect on the environment and marine life. In this study, the main objective was to investigate the presence of microplastics in personal care products that available in Malaysia and to determine the presence of microplastics from surface water in Bangi, Selangor. Microplastics from personal care products are known as microbeads and can be extracted using distilled water at 50-60 °C, filtered and dried for 24 h. The microscopic technique was used to identify the color and shape of the microplastics in each sample. The presence of microplastics in personal care products can be proven by FTIR spectroscopy as well as SEM analysis. All the six products were confirmed to have microplastics content with four of them were from PE while the other two was from PS polymer. The results from the SEM analysis also found that the size of the microplastics extracted was in the range 98-300 μm. Based on the FTIR spectrum, the functional groups for the specific polymer can be proven by the presence of significance peaks of C-H stretching, H-C-H stretching (deformation) and stretching of the

CH$_2$-CH$_2$ network that refer to PE-type polymers for samples B, D, E, and F. Furthermore, FTIR spectrum of sample A and D with significance peak observed for aromatic C-C and C=C stretching which indicate PS polymer. Microplastics also founded in water samples and analyzed as PE which sizes range of 3-4 mm.

Furthermore, the sampling locations were chosen to facilitate the sampling process with the nearby area and this was in line with the objective of the study which to determine the presence of microplastics from Bangi, Selangor. In addition, the sampling technique for the surface water varies according to the type of location and the circumstances surrounding the selected location. Sampling for lake water was using bucket because of the lake condition which contaminated with various other solid materials. Meanwhile, for the river location, insect net was used because the heavy flow of the river was not suitable with the bucket technique.

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Nur Hapizah Suardy, Nurfaizah Abu Tahrim & Suria Ramli*
Department of Chemical Sciences
Faculty of Science and Technology
Universiti Kebangsaan Malaysia
43600 UKM Bangi, Selangor Darul Ehsan
Malaysia

Nurfaizah Abu Tahrim & Suria Ramli*
Center for Water Research and Analysis (ALIR)
Faculty of Science and Technology
Universiti Kebangsaan Malaysia
43600 UKM Bangi, Selangor Darul Ehsan
Malaysia

Suria Ramli*
Polymer Research Center (PORCE)
Faculty of Science and Technology
Universiti Kebangsaan Malaysia
43600 UKM Bangi, Selangor Darul Ehsan
Malaysia

*Corresponding author; email: su_ramli@ukm.edu.my