

A separation of plastic debris in Saigon river sediment using a microplastic sediment separator

Bao-Son Trinh*, Dam Nguyen Vu, Pham Thi Bich Luyen, Pham Cam Huy



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ABSTRACT

Plastic debris was separated from Saigon river sediment using a microplastic sediment separator (MPSS) for the first time. A separation fluid of $ZnCl_2$ solution (1.6 kg/L) was used to separate lower density materials (floating on the top) from higher density materials (sinking in the bottom). Plastic and plastic-like materials were collected and analyzed by an FTIR spectrometer to determine plastic composition. The results show that a total of 350 items (0.0485 g) of plastic debris were extracted from 1,500.00 g dry sediment, including 11 items (0.0130 g) of macroplastics (> 5 mm) and 339 items (0.0355 g) of microplastics (0.3–5 mm). Polyethylene (PE), polypropylene (PP), and PE-PP mixture were the most major plastic types with 225, 85, and 21 items, respectively. Others, including polystyrene (PS), polyurethane (PU), silicon, rubber, and nylon were also found in the sediment sample. Fragments were the major shape with 300 items while other shapes of sheets, fibers and beads were also recorded. The advantages of MPSS are high capacity, high separation efficiency, and low-cost operation. The disadvantages of this apparatus are high pollution due to using $ZnCl_2$, bottom stirrer is easily stuck, and uncertain efficiency with small microplastic particles (< 0.3 mm). A significant high number of microplastics in sediment environment indicates that microplastics could be a potential risk for benthic organisms and aquatic food web. Various types and shapes of plastics also suggest that macroplastics and microplastics in sediment could originate from various sources. It is said that, for the first time in Vietnam, plastic debris, in particular with microplastics, can be efficiently extracted from a large amount of sediment sample (up to 1.5 kg dry sediment). These findings could be an interesting reference for researchers who may want to obtain a large amount of microplastics for further investigation of microplastic toxicology.

Key words: Microplastics sediment separator, river sediment, density, separation

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INTRODUCTION

Plastic debris in aquatic environment has drawn great attention in recent years. It was easily found in surface water, water column, and sediment^{1,2}. Plastics exhibit high resistance to biodegradation with half-lives varying from days to centuries³. Low density plastics, e.g. polypropylene (PP, 0.855-0.946 kg/L), polyethylene (PE, 0.926-0.940 kg/L), or polystyrene (PS, 0.96-1.04 kg/L), can float in surface water while high density plastics, e.g. polyethylene terephthalate (PET, 1.38 kg/L), polyoxymethylene (POM, 1.41 kg/L), epoxy resin (1.1-1.4 kg/L) or polyvinyl chloride (PVC, 1.1-1.45 kg/L), are expected to sink in sediment. This simple prediction may be misleading because over time most surfaces develop a biofilm or form aggregations which could eventually cause even buoyant plastics to sink⁴.

Sediment environment is suggested to be a long-term sink for plastic debris⁵. Table 1 summarizes maximum concentrations of microplastics (< 5 mm) separated from sediments worldwide. Recently, several

methods of microplastic separation have been published. Most of these methods are based on the principle of density which causes the floating of low-density materials and sinking of high density materials. In 2015, the National Oceanic and Atmospheric Administration (NOAA) of the United States of America⁶ reported laboratory methods for microplastic separation in marine sediment samples. In brief, an amount of wet sediment (400 g) was soaked in lithium metatungstate solution ($d = 1.6$ kg/L, 300 mL) to separate microplastics. In 2016, Quinn *et al*⁷ validated the recovery rates of different salt solutions for microplastic separation. An amount of marine sediment (66.66 g) with 0.066 g microplastic were transferred in sodium chloride (NaCl), sodium bromide (NaBr), sodium iodide (NaI) and zinc bromide ($ZnBr_2$) for the study. In 2017, Coppock *et al*⁸ suggested an improved small-scale sediment-microplastic isolation unit to separate microplastics from sediments. Similarly, an amount of dry sediment (30–50 g) was soaked in $ZnCl_2$ solution (1.5 kg/L, 700 mL) to separate microplastics. Nevertheless, these methods were

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implemented with a small amount of sediment samples (tens to hundreds of grams) while a larger amount of microplastics could be essential for various purposes of microplastic investigation. For instance, people need to analyze not only composition of plastic debris but also other associated chemicals such as persistent organic compounds^{9,10}, and bacteria¹¹. In addition, toxicological assays for investigating the impacts of microplastics on aquatic organisms also need a large amount of microplastics.

Since the early years of 2010s, a microplastic sediment separator (MPSS) or the so-called Munich Plastic Sediment Separator was launched in Munich, Germany. This apparatus can operate with a large amount of sediments (up to 6 L of wet sediment) and therefore is expected to separate a larger amount of microplastics than previous methods. In 2012, Imhof *et al*²⁰ was the first author who evaluated the efficiency of the MPSS. A volume of 1 L of the river Würm sediment was inoculated with artificial reference plastics (ARPs) of different size-classes (large microplastics 2–5 mm; small microplastics 0.040–0.309 mm). It is reported that the recovery rates of this apparatus were reported of 100 % for large microplastics (1–5 mm) and 95.5 % for small microplastics (< 1 mm). In 2016, Zobkov and Esiukova²¹ re-evaluated the efficiency of this apparatus with ARPs (fluorescent PET with thickness of 0.46 ± 0.39 mm and side sizes of 0.90 ± 0.39 mm) and without ARPs in natural marine bottom sediments. The results were then compared with the separation efficiencies of the MPSS method published by Imhof *et al*²⁰ and the modified separation method published by NOAA⁶. It was reported that while the ARP extraction efficiency from natural sediments by the MPSS was really high ($97.1\% \pm 2.6\%$) the extraction efficiency of marine microplastics was considerably lower (13–39 %) than that obtained with the NOAA's method. Zobkov and Esiukova²¹ suggested that the MPSS is a useful tool for Microplastic extraction from large sediment samples but it is needed to further testing and elaboration of standardized extraction procedures.

According to our knowledge, since the publication of Zobkov and Esiukova²¹, no further study using MPSS has been published. Furthermore, there are no previous studies using the MPSS to separate plastic debris in riverine sediments, particularly in Saigon river sediment. This study, therefore, aims to test the MPSS for separation plastic debris in a Saigon river sediment. Wet peroxide oxidation, Fourier-transform infrared (FTIR) spectroscopy, and stereomicroscope techniques were then applied to identify quantity and quality of plastic debris.

MATERIALS AND METHODS

Sediment sample

Sampling site ($10^{\circ}45'03.4''\text{N}$ $106^{\circ}44'43.6''\text{E}$) was located near Phu My (PM) bridge, a downstream position of the Saigon rivers where it just flows through Ho Chi Minh city (Figure 1) before confluence to Dong Nai rivers and pouring to the South sea of Vietnam at Can Gio estuary. This site is expected to be a sink of plastic debris because the rivers could transport a large amount of solid waste including plastic debris from urban and industrial areas sitting along the upstream. Sediment samples were collected at three different positions (~10 kg/position) across the rivers (Figure 1a) using a Hydrobios bottom sampler (Ekman-Birge type, box sizes of $15 \times 15 \times 20$ cm, grasping area of 225 cm^2 , Figure 1b). The samples were stored in 15 L stainless steel buckets with closed caps and transported to laboratory in the same day. These replicates were then wellly mixed together to finally get a composite sediment sample. It was kept in dry and dark area at room temperature ($25\text{--}28^{\circ}\text{C}$) until used. Physico-chemical characteristics of the PM sediment sample was analyzed in laboratory and presented in Table 2.

Chemicals

Zinc chloride solution: ZnCl_2 (27 kg) purchased from Hsien Ang Industry Co., Taiwan with 98.3 % purity was mixed with DI water (20 L) to get the final ZnCl_2 solution with density of 1.6 kg/L. Hydrogen peroxide (30 %) and H_2SO_4 were purchased from Fisher Chemical, USA. Iron Sulfate Heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) with 99 % purity was purchased from Xilong Scientific Co., China. Other chemicals were used at analytical grade.

Microplastic separation using MicroPlastic Sediment Separator

Sample pre-treatment: the sediment sample was dried (60°C , 48 h). Dry sample (1,500.00 g) was ground by a mortar and pestle and then sieved by a 2 mm sieve to remove any large and hard components (> 2 mm) which could lock the stirring motor. The upper-sieve portion (≥ 2 mm) was inspected to collect all plastic and plastic-like materials by a forceps and a stereomicroscope $\times 30$ (Olympus SD30, Japan). Large plastic and plastic-like materials were washed with distilled water and dried at room temperature ($25\text{--}28^{\circ}\text{C}$, 72 h) before analyzing by an FTIR spectrometer. The under-sieve portion (< 2 mm) was applied for microplastic separation using a microplastic sediment separator.

Table 1: Maximum concentrations of microplastics separated from sediments worldwide. All concentrations are expressed as either number of items kg⁻¹ dry sediment or mg kg⁻¹ dry sediment.

Country	Location	Maximum concentration	Unit	Refs.
India	Ship-breaking yard	89	mg kg ⁻¹	12
UK	Beacha	9	# kg ^{-1b} 13	14
UK	Estuarinea	35	# kg ^{-1b}	14
UK	Subtidala	86	# kg ^{-1b}	14
Singapore	Beach	16	# kg ⁻¹	15
UK	Sewage disposal site	15	# kg ^{-1b}	16
Belgium	Harbor	391	# kg ⁻¹	17
Belgium	Continental shelf	116	# kg ⁻¹	17
Belgium	Beach	156	# kg ⁻¹	17
Tunisia	Lagoon-channel of Bizerte	3,000-18,000	# kg ⁻¹	18
Canada	Lake Ontario	760	# kg ⁻¹	19
Canada	Etobicoke Creek	28,000	# kg ⁻¹	19

^a Only fiber concentrations were reported.

^b Original unit (# fibers 50 mL⁻¹ sediment) converted using an average sediment density of 1600 kg m⁻³ and 1.25 as average wet sediment/dry sediment ratio.¹³

Table 2: Physico-chemical characteristics of the Phu My sediment sample

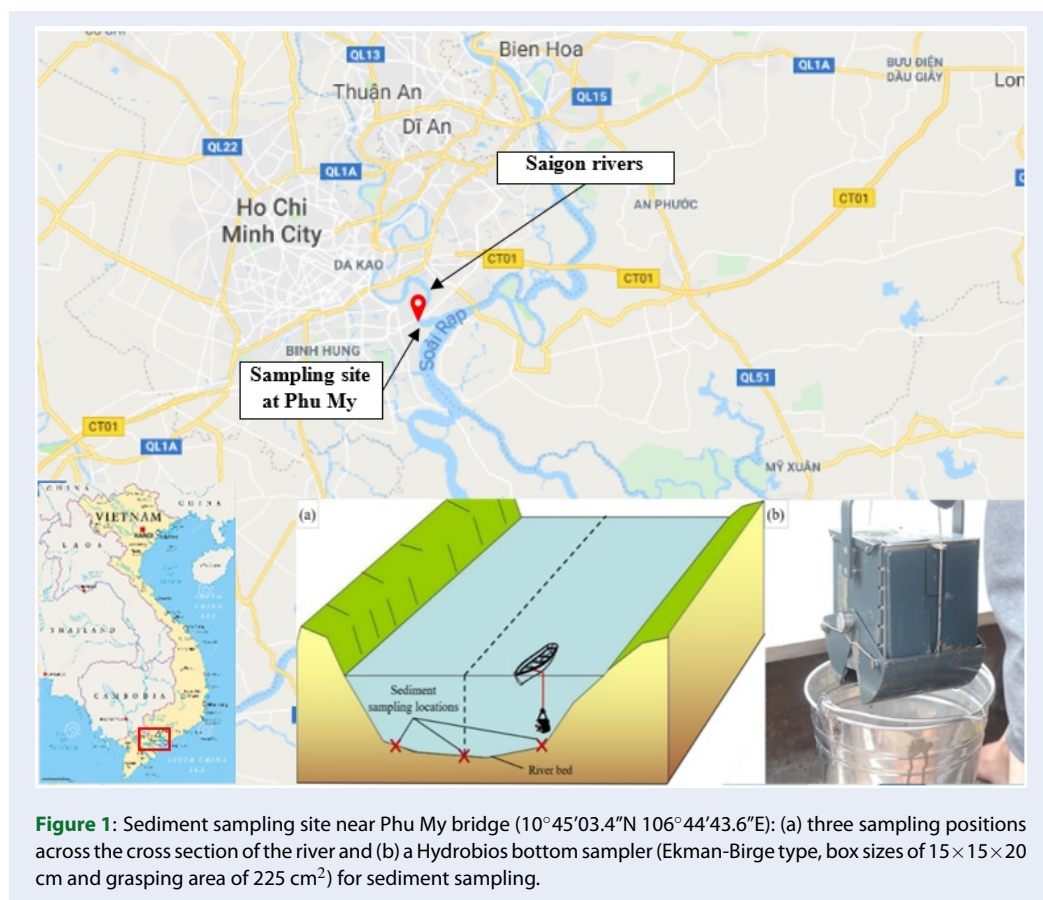
Parameters	Particle size distribution			TOC	Moisture
	Sand (0.075-0.250 mm)	Silt (0.002-0.075 mm)	Clay (< 0.002 mm)	(mg OC/ g sed)	(%)
Values	46.7 %	43.0 %	10.3 %	3.23	1. 63.35

Microplastic sediment separator (MPSS): MPSS manufactured by Hydro-Bios Apparatebau GmbH, Germany was used to extracting plastic debris from the composite sediment sample. Based on different densities of materials, plastic debris, normally with low densities varying 0.9–1.4 kg/L, can be separated from sediment through a high-density separation fluid e.g. ZnCl₂ solution (1.6 kg/L). The advantages of this method are high capacity (up to 6 L of wet sediment sample for a single run) and high efficiency (up to 100 % recovery rate for large plastic items (> 1 mm) and 95.5 % recovery rate for small plastic items (< 1 mm))^{20,22}.

Figure 2 presents the structure of an MPSS. It is divided into three major components: (1) a sediment container: (17 L) equipped with a rotor for maintaining a constant stirring (0–30 rpm); (2) a standpipe: with gradually reduced diameter for achieving

a high particle concentration; and (3) a sample chamber: equipped with two glass tubes for easy supervision of the separation process.

Microplastic separation process by MPSS: at first, the bottom standpipe was mounted on the sediment container. A volume of ZnCl₂ solution (~25L, 1.6 kg/L) was filled in the standpipe up to about ~85 % high. The stirring motor was turned on to agitate the solution (~25 rpm). While the motor was revolving, the sediment (under-sieve portion, < 2mm) was gradually introduced to ZnCl₂ solution through the open sediment inlet flange. It was noted that high feeding rate could result to the increase of possibility of plastic items being pulled down and buried in the sediment. The stirring motor continued to work for 2 h to allow a first separation where the floating materials, including plastic debris and organic materials, had enough time to separate from the sinking sediment.



After stirring, the separation fluid was settled down for 2 h. The top standpipe and the sample chamber were then installed. A fresh ZnCl₂ solution was introduced using the bottom valve to elevate the fluid level and lift the floating materials through the open valve into the sample chamber (Figure 2a). The ball valve was then closed. The bottom valve and the vent screw were opened to lower the fluid. The sample chamber, containing trapped floating materials, was detached. By turning the sample chamber upside-down and opening the ball valve, the trapped floating materials were collected by draining the separation fluid using a vacuum filter.

Oxidation process: The trapped floating materials were oxidized by a wet peroxide oxidation protocol published by NOAA⁶ for a second separation where floating organic materials were decomposed. Briefly, Fe₂SO₄ solution (0.05 M, 20 mL) was added to a beaker containing the trapped floating materials and agitated by a magnet fish. Hydrogen peroxide (30 %, 20 mL) was gradually transferred to the mixture and heated to 75 °C. As soon as gas bubbles were observed at the surface, the beaker was removed from the hot-

plate and placed in the fume hood until boiling subsided. The solution was heated to 75 °C for an additional 30 minutes. If bubbles were observed, another 20 mL hydrogen peroxide 30% was added. This step was repeated until no bubbles were visible. The oxidized solid materials were then transferred to a 0.3-mm sieve and the upper-sieve portion (≥ 0.3 mm) was collected for a final plastic separation by a forceps under a stereomicroscope (Olympus SD30, Japan) before analyzing by an FTIR spectrometer.

Plastic identifying process: The large (≥ 2mm) and small (≥ 0.3 mm) upper-sieve portions were identified for plastic types by an FTIR spectrometer (FTIR-6600 equipped with an ATR Pro One Single-Reflection, Jasco, Japan). Infrared radiation of 500–4000 cm⁻¹ and a spectral resolution of 4 cm⁻¹ with 35 scans were applied to identify a specific spectrum of the testing material. The spectrum was then compared with the KnowItAll™ spectral library (Wiley Science Solutions) with an acceptable matching ratio of higher than 75%. According to popular plastic classification based on size, large plastic items were listed as macroplastics (≥ 5 mm) and the smaller one are listed as microplastics (0.3–5 mm).

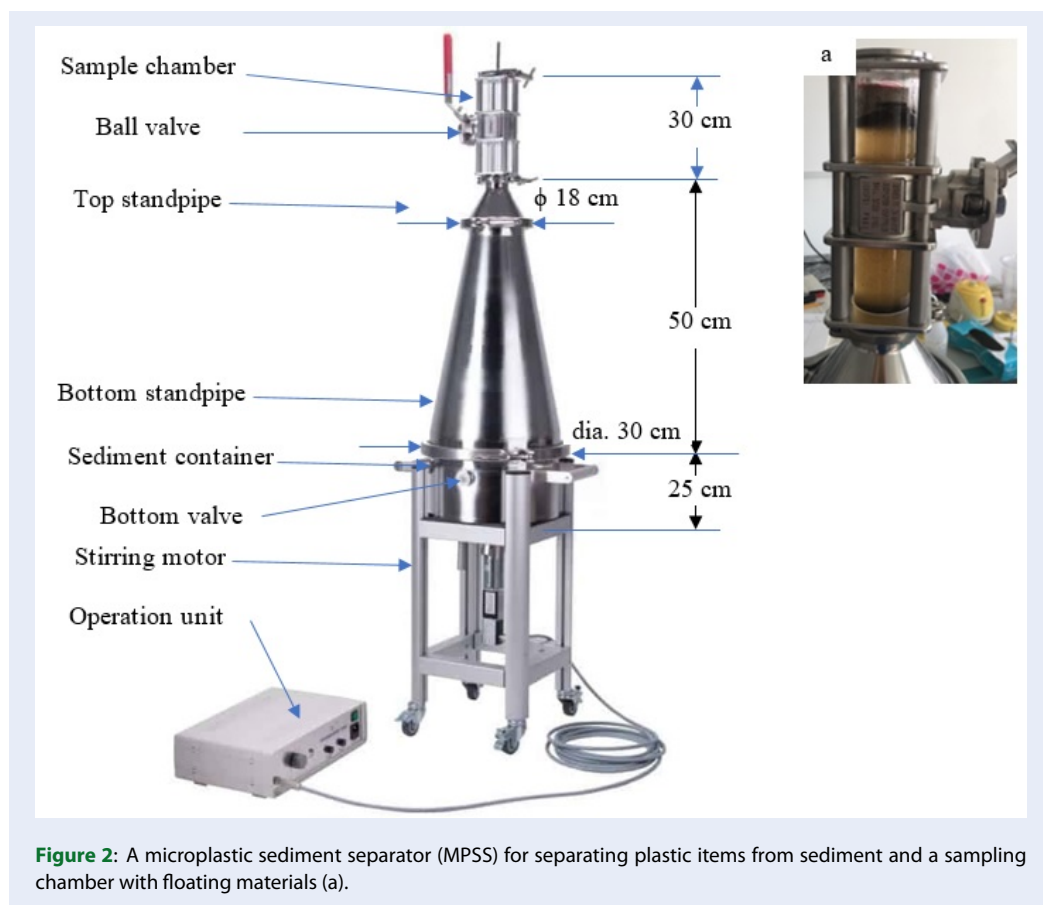


Figure 2: A microplastic sediment separator (MPSS) for separating plastic items from sediment and a sampling chamber with floating materials (a).

RESULTS AND DISCUSSION

Plastic debris extracted from the sediment sample was presented based on size, weight, types and shapes. According to size, plastic debris was classified as macroplastics (≥ 5 mm) and microplastics (0.3–5 mm) (Figure 3). There was a total of 350 items of plastic debris extracted from 1,500.00 g dry sediment (233 items/kg sediment), including 11 items of macroplastics (7 items/kg sediment) and 339 items of microplastics (226 items/kg sediment) (Figure 4a). A significant high number of microplastics in sediment environment indicates that microplastics could be a potential risk for benthic organisms and aquatic food web. Many previous studies reported the accumulation of microplastics in benthic organisms such as blue mussel *Mytilus edulis* L.²³, freshwater mussel *Dreissena polymorpha*²⁴.

Based on weight, a total of 0.0485 g plastic debris (32.3 mg plastic/kg dry sediment) was collected, including 0.0130 g of macroplastics (8.7 mg/kg) and 0.0355 g microplastics (23.7 mg/kg) (Figure 4b). This number shows that microplastic mass is higher (approximately

3 times of this study) than the mass of microplastic in sediment.

Based on plastic types, polyethylene (PE) is the most major type with 225 items. Polypropylene (PP) and polystyrene (PS) are the second and third major types with 85 and 23 items, respectively (Table 3). Other plastic types of polyurethane (PU), a mixture of PE and PP, silicon, rubber, and nylon were also found with the values of 5, 4, 4, 3 and 1 items, respectively (Figure 4c). This observation is consistent with the results reported by Lahens, Strady¹ that the major types of macroplastics in surface water of Saigon rivers were PE (79%) and PP (15%). In water column, plastic debris in surface water will be sunk in sediment according to its density and other environmental factors^{4,25}. Based on plastic shapes, fragments were the major shape with 300 items. Other shapes of sheets, fibers and beads were also found with the values of 21, 20, and 9 items, respectively (Figure 4d). Table 3 summarizes the number items of plastic debris according to types and shapes which were extracted from the 1,500.00 g sediment sample. This finding is again consistent with the observations reported by¹.

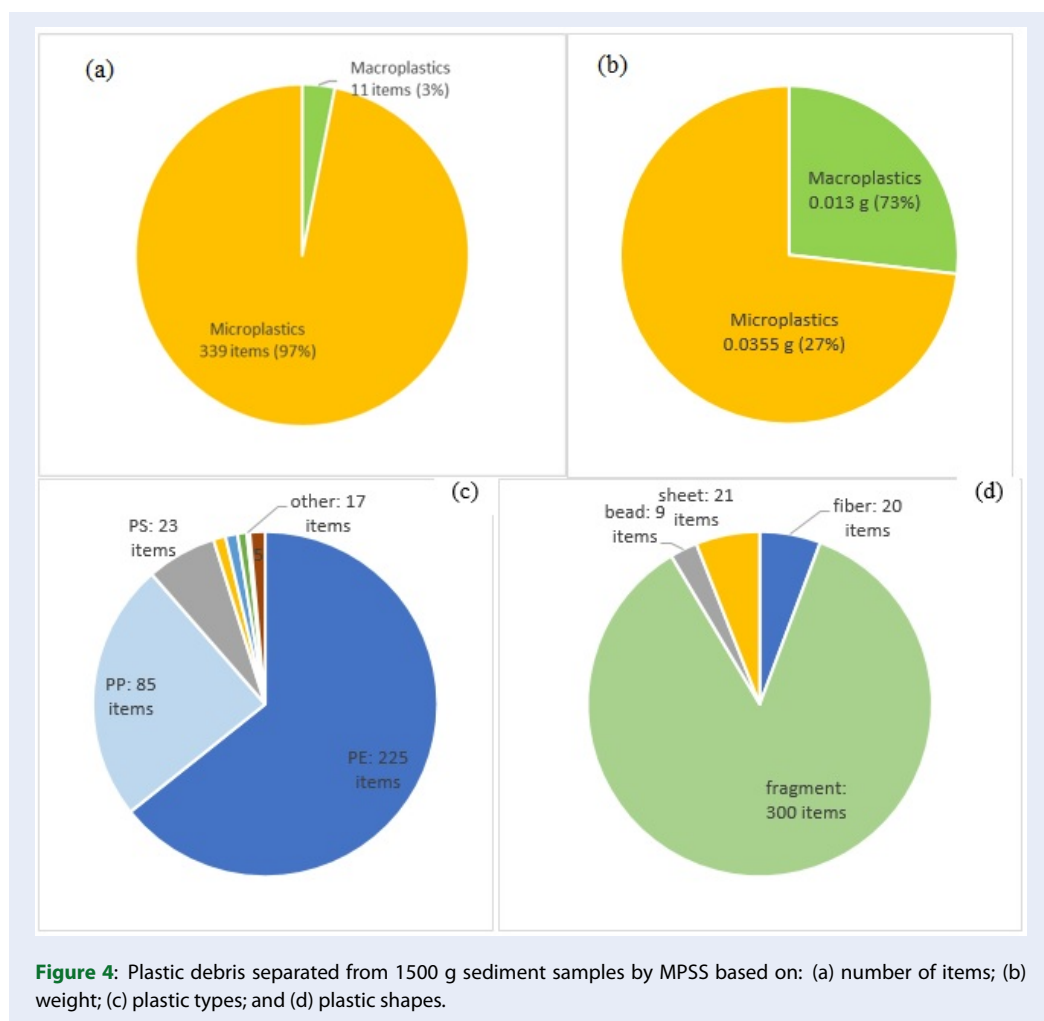
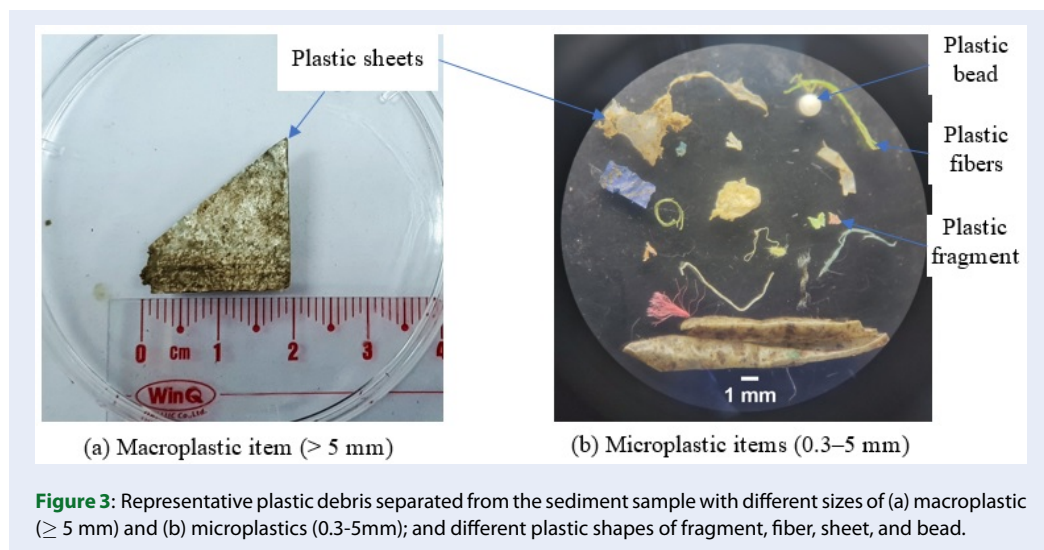


Table 3: Numbers of plastic debris (items), including macroplastics (MaPs) and microplastics (MiPs), extracted from 1,500.00 g of Phu My sediment according to plastic types and shapes.

Plastic types	Plastic shapes	Fragment (items)	Fiber (items)	Sheet (items)	bead (items)	Total (items)
MaPs	PE	0	3	4	0	7
		0	2	0	0	2
		2	0	0	0	2
MiPs	PE	197	5	11	5	218
		71	9	2	1	83
	PE+PP	16	0	2	3	21
		2	0	2	0	4
	Silicon	4	0	0	0	4
	Rubber	3	0	0	0	3
	Nylon	1	0	0	0	1
PU	4	1	0	0	5	
Total (items)		300	20	21	9	350

Not many previous studies reported the quantity of microplastics in sediments based on weight (see Table 1). It could be because many microplastic items are so small and light that it is so difficult to determine their mass. Infrequently, Reddy *et al*¹² reported the maximum concentration of microplastics in a ship-breaking yard in India was of 89 mg/kg dry sediment. This concentration is higher than our finding of 23.7 mg/kg. Different flow rates of the Saigon rivers in our this study could result to the concentrations of microplastics in sediments.

CONCLUSION

Microplastic sediment separator (MPSS) was successfully used for the first time in Vietnam to separate plastic debris, including macroplastics (>5 mm) and microplastics (0.3–5 mm), from natural river sediment. The advantages of this apparatus are (1) high capacity, up to 1,500 g dry sediment/time; (2) high efficiency with microplastics having particle sizes larger than 0.3 mm; and (3) low-cost operation with ZnCl₂ solution. The disadvantages of this apparatus are (1) high aquatic pollution due to using ZnCl₂ solution; (2) the bottom stirrer is easily stuck by hard and large (> 2 mm) solid particles; and (3) uncertain efficiency with microplastics having particle sizes smaller than 0.3 mm.

A significant high number of microplastics in sediment environment indicates that microplastics could be a potential risk for benthic organisms and aquatic food web. Various types and shapes of plastics also

suggest that macroplastics and microplastics in sediment could originate from various sources. It is said that, for the first time in Vietnam, plastic debris, particular with microplastics, can be efficiently extracted from a large amount of sediment sample (up to 1.5 kg dry sediment). These findings could be an interesting reference for researchers who may want to obtain a large amount of microplastics for further investigation of microplastic toxicology.

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LIST OF ABBREVIATIONS

- ARPs Artificial Reference Plastics
- DI DeIonized
- Fe₂SO₄ Ferric Sulfate
- FTIR Fourier-Transform Infrared spectroscopy
- H₂SO₄ Sulfuric acid
- MaPs MacroPlastics
- MiPs MicroPlastics
- MPSS MicroPlastic Sediment Separator
- NaBr Sodium Bromide
- NaCl Sodium Chloride
- NaI Sodium Iodide
- NOAA National Oceanic and Atmospheric Administration
- OC Organic Carbon

PE PolyEthylene
 PET PolyEthylene Terephthalate
 PM Phu My
 POM PolyOxyMethylene
 PP PolyPropylene
 PS PolyStyrene
 PU PolyUrethane
 PVC PolyVinyl Chloride
 TOC Total Organic Carbon
 UK United Kingdom
 USA United States of America
 ZnBr₂ Zinc Bromide
 ZnCl₂ Zinc Chloride

CONFLICT OF INTEREST

The researchers declare that there is no conflict of interest during and after the study.

AUTHOR CONTRIBUTION

Dr Bao-Son Trinh is the principal investigator of this study who has designed and implemented the assays and finally written this report.

Dam Nguyen Vu, Pham Thi Bich Luyen, and Pham Cam Huy were the researcher who assisted Dr. Trinh to complete this project.

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