American Journal of Environmental Protection
2025, Vol. 14, No. 2, pp. 36-49
https://doi.org/10.11648/j.ajep.20251402.12

Research Article

Density-Based Multi-Stage Flotation Sorting of Microplastics in Beach Sand

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Abstract

Microplastics (MPs) are ubiquitous in the environment and pose an increasing concern for the world's terrestrial and marine ecosystems due to their persistence and potential toxicity. Density sorting of MPs in beach sand, combined with heat treatment to remove impurities such as wood fragments, enhances the analysis of MP contamination. While density sorting does not identify the composition of MPs, it provides insight into their sources and potential for re-drift into the ocean. In this study, we evaluated the accuracy of a multi-stage Flotation sorting technique in separating MPs based on their density in beach sand. A major challenge in density sorting is interference from impurities such as wood fragments. To address this, heat treatment is performed to remove wood fragments. We also evaluated the effects of heat treatment on the density and weight of MPs. The findings indicate that most MPs experienced a density change and a weight loss of less than 4% and 1%, respectively, suggesting the minimal effects of the heat treatment. However, certain types of MPs, such as those containing voids (e.g., PVC-NS), showed significant density changes, which impacted their sorting behavior, resulting in some misclassification during the flotation sorting. Unless the heat treatment caused a density change, the multi-stage Flotation sorting method, including water and saturated calcium chloride (SCC) solutions, achieved high recovery rates (90%-110%) for light MPs, heavy MPs, and wood and sand mixtures. In other words, light and heavy MPs and the wood and sand mixture were separated without misjudgment and loss. Overall, this study confirms the feasibility and efficiency of multi-stage flotation sorting for MP analysis in beach sand and highlights the need to carefully consider heat treatment effects in future environmental studies on MPs.

Keywords

Microplastic, Boiling, Density Sorting, Flotation Sorting, Heat Treatment, Wood Fragments, Recovery Rates

1. Introduction

Plastics have become an indispensable and integral part of human lives. They are extensively utilized in many spheres of life owing to their convenience and superior features [1-3]. Global plastic production rose from 245 million tons in 2008 to 390.7 million tons in 2021 and is projected to reach 600 million tons in 2050 [4, 5]. Unfortunately, recycled plastics account for only 6% to 26% of manufactured plastics, meaning that 74% to 94% of plastics either end up as waste in

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Received: 6 March 2025; **Accepted:** 27 March 2025; **Published:** 17 April 2025

landfills or are directly released into the environment through various pathways [3, 6], posing a serious threat to marine ecosystems [7]. One of the most critical problems linked to plastic waste is microplastics (MPs), which are tiny plastic particles measuring less than 5 mm in size [2, 8, 9]. MPs infiltrating and contaminating the environment [10] are a growing threat to the environment and ecosystems given their potential toxicity, resilience, and persistence [11, 12]. Moreover, because of the huge amounts of plastics used in recent decades, MPs have been ubiquitously detected in all continents and the poles [13], polluting environmental matrices such as oceans, marine sediments, fresh water, wastewater, lakes, soil, air, food, organisms, and terrestrial ecosystems [2, 4, 14] even in the Antarctic and Arctic regions where human activities are restricted [4]. MPs in environmental matrices such as sediments, sand, and seawater have been analyzed [15, 16].

Effective detection and separation of MPs from environmental samples are essential for understanding their distribution and impact. Several techniques have been developed to isolate and identify MPs, each with its advantages and limitations. The U.S. National Oceanic and Atmospheric Administration's (NOAA) manual for analyzing MPs in beach samples [17] comprehensively specifies the separation and analysis of MPs, including density separation and material determination using an infrared spectrometer. Previous comprehensive studies widely used the density separation (Flotation) method to separate MPs [16, 18, 19]. Density-based separation, or flotation, is a simple and cost-effective method for isolating microplastics (MPs) using liquid density differences [15, 20, 21]. Despite its widespread use, this technique has certain limitations. Materials such as wood fragments with densities similar to some MPs can complicate the separation process [22]. Additionally, density separation alone cannot identify polymer types, making it difficult to assess the relative abundance of different plastics in a sample [22]. The method may also be less effective for very small or highly fragmented MPs, which might not exhibit clear flotation behavior [23]. Moreover, flotation is time-consuming and requires an additional treatment method [16, 24]. Filtration is a common method for isolating microplastics (MPs) from aqueous samples by trapping particles on filters with specific pore sizes [25]. However, it faces challenges with MPs smaller than 1 mm, which may pass through or be lost during the process [26, 27]. Additionally, organic materials, such as algae or plant debris, can clog the filters and interfere with MP extraction, particularly in complex samples like beach sand, leading to reduced fiber recovery [28]. Therefore, filtration alone is insufficient for comprehensive MP analysis in highly contaminated environments. On the other hand, spectroscopic methods like FTIR and Raman spectroscopy are the gold standard for MP identification, detecting polymer-specific absorption or scattering peaks. However, they are costly, require skilled personnel, and involve lengthy sample preparation, limiting scalability for large surveys. Moreover, they identify polymer types but do not efficiently separate MPs from complex matrices like sand and wood [29]. Furthermore, digestion and sieving techniques are commonly used to isolate microplastics (MPs) from sediments. However, their large-scale application is often limited by high labor costs and time-intensive processes [16].

Recent studies highlight multi-stage flotation as an effective method for improving MP separation using multiple density-based steps. Multi-stage flotation sorting has emerged as a superior technique for separating microplastics (MPs) from complex matrices, offering distinct advantages over traditional methods such as density separation, digestion, and sieving. By employing multiple flotation stages with liquids of varying densities, multi-stage flotation effectively isolates MPs based on subtle density differences. This approach significantly improves the purity of the separated MPs by progressively eliminating contaminants like wood fragments and organic debris. In contrast, traditional single-step methods often struggle to achieve comparable levels of separation efficiency [30]. Multi-stage flotation is more amenable to scaling up industrial applications than other methods. It requires relatively simple equipment and can process large volumes of material efficiently. Additionally, this method is environmentally friendly, as it minimizes the use of hazardous chemicals often associated with digestion processes [31]. The multi-stage flotation method allows for more precise separation of MPs from complex environmental matrices, reducing contamination from non-plastic materials like wood. By integrating heat treatment, the study aims to enhance the effectiveness of the flotation process, potentially improving the accuracy of MP quantification in beach sand samples.

We have previously used multiple liquids having different densities to density-sort a single type of MP. We confirmed that MPs with densities lower than the liquid floated, whereas MPs with densities higher than the liquid sank [32]. Thus, it is possible to sort MPs into two densities if a heavy liquid is used after water. Although the materials of MPs cannot be determined by density sorting, it is possible to estimate the ratio of MPs that float to those that sink in seawater. Except for hollow plastic materials, MPs with a specific gravity greater than 1 found in beach sand would originate not from the sea but from higher elevations on land. On the other hand, for MPs with a specific gravity of less than 1, their origin is not known, but their potential to re-drift into the ocean can be evaluated. Both MPs with specific gravities greater than 1 and less than 1 are mixed in beach sand [33]. Is it possible to sort them using the two-step sorting method previously described by the authors?

Wood fragments are a major contaminant in analyzing MPs in beach sand [33]. Dry wood fragments float on the water surface together with light MPs, and this can be problematic when performing density sorting. Wood fragments that are boiled to accelerate water absorption sink and can thus be removed [33]. Heat treatment such as boiling, however, may damage MPs [6, 34] through chemical degradation, poten-

tially resulting in weight loss or alterations in their physical properties, such as size or shape [35]. Furthermore, heating can cause the loss of volatile compounds, underestimating MP mass [36]. If the density of MPs is changed by heating, the floating/sinking behavior of MPs during density sorting will be altered as well, possibly resulting in erroneous sorting where MPs are classified into the wrong categories. More specifically, MPs that should float on the water's surface might be mistakenly determined as MPs that sink in water. Moreover, if weight loss occurs because of heating, the amount of MPs present in beach sand samples will be underestimated. What is the extent to which heat treatment affects the density and weight of MPs?

We focus on the following objectives in this study: (1) to evaluate the effect of heat treatment on MPs, and (2) to evaluate the accuracy of multi-stage Flotation sorting, in which MPs in beach sand are density-sorted, removing the wood and sand mixture.

Accurately isolating and identifying microplastics (MPs) from environmental samples is crucial for monitoring plastic pollution levels. The findings of this study could enhance MP monitoring strategies in coastal environments, leading to more precise assessments of pollution levels and trends over time. Reliable MP quantification is essential for developing effective policies to address plastic pollution. Additionally, the insights gained from this research could inform plastic waste management strategies, helping industries optimize recycling processes and enabling policymakers to implement more targeted regulations to reduce plastic waste in the environment.

2. Materials and Methods

2.1. Materials

2.1.1. Equipment

Commercially available scissors, nippers, cutters, shear crusher (MF10 Basic, IKA Japan Co., Ltd.), and a small mill (OML-1, Osaka Chemical) were used to shred plastic samples (hereinafter referred to as MPs). Stainless steel sieves (SANPO) with 212 μm, 1 mm, 2 mm, and 4.75 mm mesh sizes were utilized to adjust particle size distribution. For the density measurements of MPs (L size), 50 mL pycnometers, a thermometer (TT-508N, TANITA), a precision balance (ATY124, Shimadzu), and a water purifier (RFP841AA, ADVANTEC) were used. 200 mL conical beakers, a watch glass, and a hot plate (EA-DE10, ZOJIRUSHI, Japan) were used for boiling MPs. The multi-stage Flotation sorting experiment utilized 200 mL conical beakers, 300 mL glass beakers, a stainless-steel spoon, stainless-steel trays, and a dryer (DRD420DA, ADVANTEC). Liquid density was measured using a graduated cylinder and a hydrometer (Ludwig Schneider).

2.1.2. Samples

Wood is a typical contaminant found in beach sand [33]. Therefore, we utilized mixtures of MPs, wood, and sand as samples. In this study, MP samples were prepared in the laboratory using a newly available commercial plastic product as a fundamental study (Table 1). As a single wood species, cedar chopsticks (Iwai Sangyo Co., Ltd.) were obtained and crushed in a small mill to produce cedar shavings (0.425 mm to 2 mm; hereinafter referred to as wood). Toyoura silica sand was utilized as a sand component. The density of MPs (L size) was measured following the method reported by Asakura (2022) [37]. MPs with densities lower than 1 g/cm ^{3}are classified as light MPs, while those with densities higher than 1 g/cm ^{3}are classified as heavy MPs (Table 1).

Commercial kitchen detergent (Soapen Fresh Lime, Kaneyo Soap Co., Ltd.) was utilized to promote the sedimentation of small-diameter MPs (hereinafter referred to as surfactant). A saturated calcium chloride (SCC) (Miyachu Building Materials Division, Inc.) solution was utilized as a heavy liquid for the multi-stage floating sorting experiment of MPs because SCC is inexpensive and environmentally friendly [1].

2.2. Methods

2.2.1. Principle of Multi-Stage Flotation Sorting

We performed a multi-stage Flotation sorting experiment to separate MPs according to their densities from a mixture of light MPs, heavy MPs, wood, and sand. Figure 1 shows the processing procedure for multi-stage Flotation sorting and the ideal recovery of MPs. Weighed light and heavy MPs, wood, and sand were added into a beaker containing deionized water (1.00 g/cm^3) and stirred with a spoon (Figure 1a). After a while, heavy MPs (>1.00 g/cm^3) and sand (2.62 g/cm^3) sank, and light MPs (<1.00 g/cm^3) floated on the water's surface together with the dry wood (Figure 1b).

To recover only light MPs, the wood must be allowed to settle. The apparent density of dry wood (voids are filled with air) is lower than water density, whereas the true density of wet wood (voids are filled with water) is higher than water density. For instance, the density of cedar wood used in this study is 0.99 g/cm^3 when dry and 1.42 g/cm^3 when wet. Boiling promotes the wetting of wood, thereby increasing its density and causing it to settle at the bottom of the beaker (Figure 1c). After boiling, light MPs on the water's surface were collected with a spoon (Figure 1c). If water is discarded and a heavy liquid (SCC, 1.37 g/cm^3) is added instead, only heavy MPs with densities higher than that of water but lower than that of the heavy liquid will float and be recoverable with a spoon (Figure 1d). Finally, the settled wood (1.42 g/cm^3) and sand (2.62 g/cm^3) remained at the bottom of the beaker (Figure 1d). In this way, the light and heavy MPs can be collected and weighed separately from a mixed sample of MPs, wood, and sand.

2.2.2. Effects of Boiling on MPs

Table 1. Details of plastic products used in this study.

Material {heat resistance temperature (℃)}*	Description	Prepared MP size for Figures 3 to 5**			Original density (g/cm^3)	Light / Heavy***
		L size		M size		
Polyethylene (PE) {70−110}	Shopping bag (SB)	B			0.908	L
	Glove (GV)	B			0.871	L
	Rope (RP)	B			0.754	L
	Polybottle (PB)	B	MS	MS	0.934	L
	Freezer bag (FB)	B			0.919	L
Polypropylene (PP) {100−140}	PET bottle cap (BC)	B			0.925	L
	Oriented PP (OP)	B			0.888	L
	Flat plate (FP)	B			0.867	L
	Clothespin (CP)	B	MS	MS	0.905	L
	Rope (RP)	B			0.486	L
Polystyrene (PS) {70−90}	Expanded polystyrene (EP)	B			0.018	L
	Flat plate (FP)	B	MS	MS	1.084	H
	Plastic bottle label (LB)	B			1.031	H
	Compact disk case (MC)	B			1.054	H
Polyvinyl chloride (PVC) {60−80}	Food tray (FT)	B	MS	MS	0.981	L
	Pipe (PI)	B			1.424	H
	Flat plate (FP)	B	MS	MS	1.333	H
	Corrugated plate (CP)	B			1.375	H
	Non-slip sheet (NS)	B	MS		0.884	L
	Tablecloth (TC)	B			1.305	H
Polyethylene terephthalate (PET) {60−200}	PET bottle (EB)	B			1.378	H
	Egg pack (EG)	B	MS	MS	1.315	H
	Lumirror ® film (LF)	B			1.390	H
	Fruit container (FC)	B			1.336	H
Polycarbonate (PC) {120−130}	Compact disk (CD)	B	MS	MS	1.163	H
	Safety glasses (SG)	B			1.166	H
	Flat plate (FP)	B			1.166	H
Phenol-formaldehyde (PF) {150}	Pot knob (PK)	B			1.469	H

*The Japan Plastics Industry Federation (2016) [38]
**B: boiling; MS: multi-stage Flotation sorting
***L: light MPs; H: heavy MPs

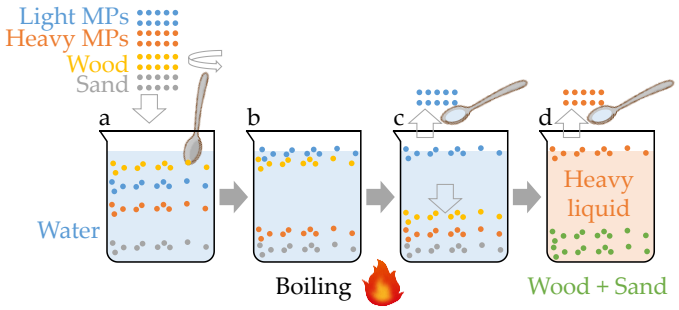


Figure 1. *The processing procedure for multi-stage Flotation sorting and ideal recovery of MPs.*

Light and heavy MPs (L size) were boiled to observe the effects of heat treatment on MP density and weight. First, 1.5 g of MPs of one plastic species (L size, n = 6) was measured and transferred into a 200 mL conical beaker, and the beaker was filled with water to 70% of its volume and stirred with a spoon. Next, the beaker containing the sample was covered with a watch glass and heated for 3 hours on a hot plate that had been preheated to 140 ℃ (set temperature). The water temperature would have reached around 100 ℃ because bubbles were observed. After boiling, the beaker was allowed to cool, and MP density was measured as described in detail by Asakura (2022) [37]. The sample was dried in an electric dryer at 80 ℃ and weighed using an electronic balance to determine its dry weight.

2.2.3. Procedure for Multi-Stage Flotation Sorting Experiment

In this experiment, a total of 16 combinations (4 types of light MPs: PE-PB, PP-CP, PS-FT, PVC-NS, and 4 types of heavy MPs: PVC-FP, PET-EG, PC-CD, PS-FP) for L size, and a total of 12 combinations (the above-mentioned 3 types of light MPs except PVC-NS (because M-sized PVC-NS was difficult to prepare owing to its ductility) and 4 types of heavy MPs) for M size, were utilized (Table 1). The experiment was performed for each combination of light and heavy MPs (e.g., PE-PB and PVC-FP) mixed with wood and sand. Wood was pre-dried in a dryer at 80 ℃ for 2 hours. Samples containing 0.5 ±0.005 g of wood, 0.5 ±0.005 g of sand, and a total of 1.5 g of light and heavy MPs in different ratios (light/heavy MPs ratios (g/g): 1.0/0.5, 0.9/0.6, 0.8/0.7, 0.7/0.8, 0.6/0.9, and 0.5/1.0 for L size (n = 6), and 1.0/0.5, 0.875/0.625, 0.750/0.750, 0.625/0.875, and 0.5/1.0 for M size (n = 5)) were weighed (Step 1, Figure A1). MPs floating on the water surface were considered light MPs (Figure 1c), and those floating on the SCC surface were considered heavy MPs (Figure 1d). In this experiment, however, misjudgment was possible because the materials of the collected MPs were not determined. For example, when equal weights of light and heavy MPs were prepared, if it was found that 30% of the light MPs sank in the water and 30% of the heavy MPs floated on the water's surface, then, this should be reported as an incorrect recovery rate; however, with the materials undetermined, it would be reported as a correct recovery rate (Figure 2). Ratios of light to heavy MPs were varied to prevent this false-positive result.

Samples were added to a conical beaker filled with water (1.00 g/cm^3) to 70% of its volume (Step 2, Figure A1), stirred, and covered with a watch glass. Then, the beaker was heated for 3 hours on a hot plate (pre-set temperature 140 ℃) (Step 3, Figure A1) to promote the sinking of wood (Step 4, Figure A1). After cooling, a small amount of surfactant was added (1/1000 of the original solution concentration) and stirred. Light MPs floating on the water surface were collected into a tray with a spoon (Step 5, Figure A1), placed in a dryer overnight to dry at 80 ℃, and weighed (corresponding to the recovered weight of light MPs). Water in the beaker was removed by decanting (Step 6, Figure A1) while being careful not to spill the settled MPs, wood, and sand. Then, SCC (1.37 g/cm^3) was added to the beaker to 70% of its volume (Step 7, Figure A1) and stirred. After waiting for 15 minutes until the wood sank and the heavy MPs floated (Step 8, Figure A1), the floating heavy MPs on the liquid surface were collected on a sieve with a mesh size of 212 μm (Step 9, Figure A1), washed thoroughly with tap water (Step 10, Figure A1), placed in a dryer overnight to dry at 80 ℃, and weighed (corresponds to the recovered weight of heavy MPs). SCC was transferred from the beaker to a graduated cylinder, and its density was measured using a hydrometer (Steps 11 and 12, Figure A1). The remaining wood and sand were diluted and washed 10 times with tap water, soaked in tap water, and left for one day (Steps 13 and 14, Figure A1). After that, the wood and sand were transferred to a tray, placed in a dryer, and dried overnight at 80 ℃ to determine the weight of the wood and sand mixture.

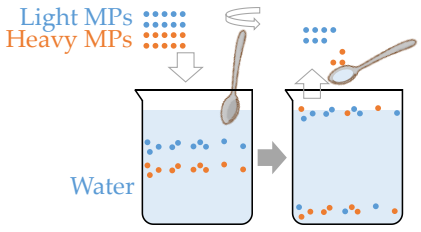


Figure 2. *An example of MPs incorrectly reported as being correctly classified, even though the MPs did not float or sink as expected. Ten light MPs were added, and ten MPs were recovered from the water surface.*

Boiling promotes water absorption. As a test, wood and water in a beaker were left overnight without boiling. Some of the wood sank while the rest remained on the water's surface.

3. Results

3.1. Effects of Boiling on MPs

Figure 3 shows the density and weight loss of MPs (L size) before and after the heat treatment. Density changes (= (boiled − original) / original) in 24 of the 28 samples were less than 4%, with some being statistically significant ($p < 0.05$). PE-RP, PS-EP, PS-LB, and PVC-NS showed a significant increase in density of more than 4% (21%, 500%, 8%, and 45%, respectively). Twenty-five of the 28 samples showed weight loss of less than 1.0%. PE-PB, PVC-NS, and PF-KP showed more than 1% weight loss (1.0%, 2.1%, and 1.2%, respectively).

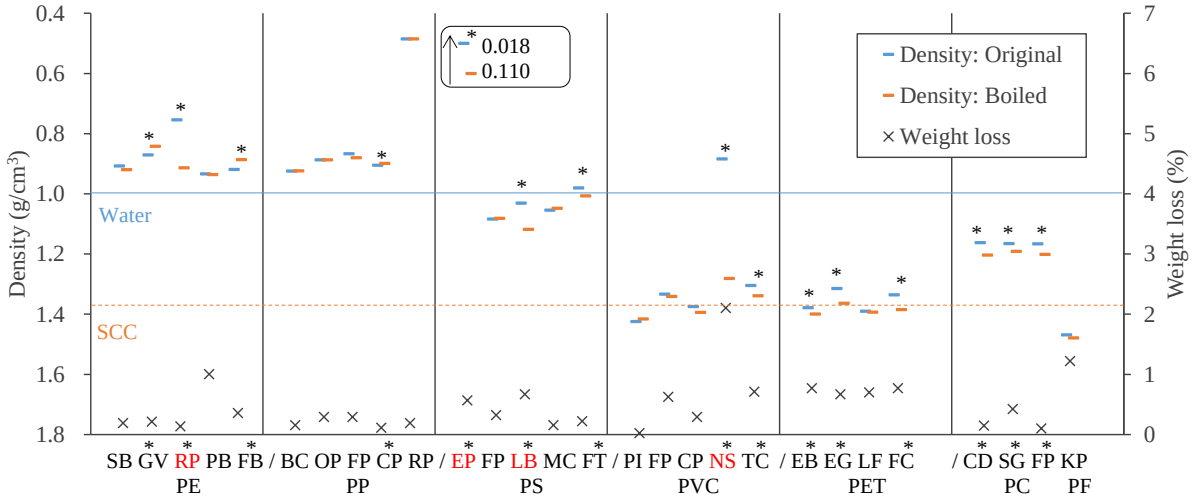


Figure 3. *Density and weight loss of MPs before and after heat treatment. n = 6. Size: L (1−4.75 mm). *: p < 0.05. Horizontal lines: density of liquids (water and saturated $CaCl_2$ solution (SCC)). Samples with red letters are those whose densities increased by more than 5% after the heat treatment. MPs whose densities lie above the horizontal line indicating the liquid density will float on the liquid surface.*

3.2. Multi-Stage Flotation Sorting Experiment of MPs, Wood, and Sand

Two examples ("as expected" and "not as expected") of the relationship between the weight of added and recovered MPs (M size, $n = 5$), wood, and sand in multi-stage Flotation sorting are shown in Figures 4a and 4b. When PE-PB (light) was added with PET-EG (heavy), along with wood and sand, respectively, the weights of the recovered light MPs, heavy MPs, and wood and sand mixture were almost the same as the weights of the added MPs, wood, and sand (Figure 4a). On the other hand, when PS-FT (light) was added with PVC-FP (heavy), along with wood and sand, the weights of the recovered light and heavy MPs differed from the weights of the added MPs (Figure 4b). The weight of the recovered heavy MPs exceeded that of the added heavy MPs, possibly because of the increase in density of the light MPs due to boiling, which prevented them from floating on the water surface; these light MPs might have subsequently floated on SCC and, thus, were identified as heavy MPs. In this scenario, the recovery rate of heavy MPs would exceed 100%. However, the weights of the recovered mixture of light and heavy MPs and of the mixture of wood and sand were almost equal to the added weights (Figure 4b).

Figure 5 shows the recovery rates (the recovered weight/the added weight) of L- and M-sized light MPs, heavy MPs, and the wood and sand mixture in the multi-stage Flotation sorting experiment for all samples. When using L-sized PE-PB and PP-CP (light), the recovery rates of the light and heavy MPs and the wood and sand mixture ranged from 94% to 105% (Figure 5a, left side). On the other hand, when using M-sized PE-PB and PP-CP (light), the recovery rates of the light and heavy MPs and the wood and sand mixture ranged from 90% to 106% (Figure 5b, left side). In other words, an increase in range was noted.

When using L-sized PS-FT and PVC-NS (light), the recovery rates of the light and heavy MPs and the wood and sand mixture were almost 0%, 150% to 400%, and nearly 100%, respectively (Figure 5a, right side). When using M-sized PS-FT (light), the recovery rates of the light and heavy MPs and the wood and sand mixture were 7% to 63%, 140% to 280%, and almost 100%, respectively (Figure 5b, right side).

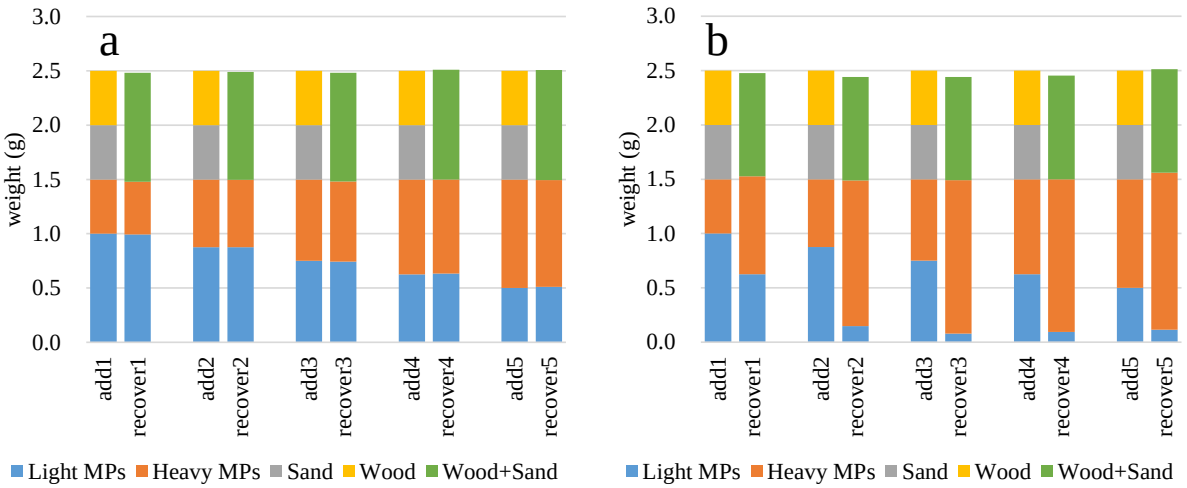
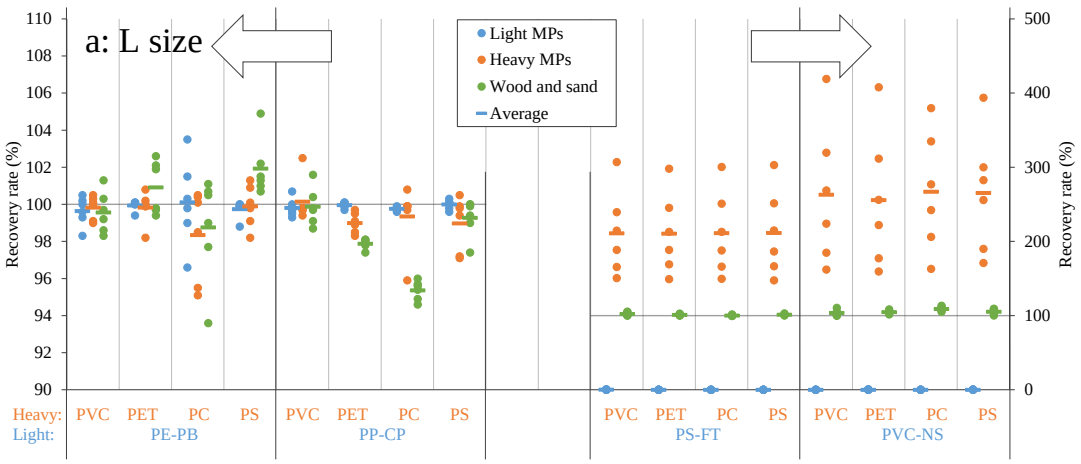


Figure 4. *Added and recovered amounts in multi-stage Flotation sorting. MP size: M (0.212−1 mm). (a) PE-PB (Light) and PET-EG (Heavy); (b) PS-FT (Light) and PVC-FP (Heavy).*

4. Discussion

4.1. Effect of Heat Treatment on MPs Analysis

If the effect of heat treatment on density is minimal, the particles should exhibit the expected floating or sinking behavior in density sorting. Most samples showed a density change of less than 4%, suggesting that heat treatment had a minimal impact on MP density during the analysis. Three of the four MPs showing the greatest density change (PE-RP, PS-EP, and PVC-NS) share a common characteristic of being materials that contain voids. Therefore, they have relatively low original densities, even within the same type of plastic. For example, whereas the densities of most PVC materials fall in the range of 1.2 to 1.4 g/cm^3, the original density of PVC-NS is less than 1.0 g/cm^3. We speculate that the original density of PVC-NS was probably measured when its voids were filled with air. If this were the case, the density difference would be due to the method of density measurement rather than the effect of boiling. Alternatively, it is possible that the density approached its true value after boiling. On the other hand, the principle of boiling that caused dry wood to sink could be understood from the density change in PVC-NS. A dishwashing sponge floats on the water's surface when dry but eventually sinks as it gradually absorbs water. However, if removed from the water and squeezed to expel the absorbed water, it will float again. What this suggests is that it is difficult to define the density of particles containing air voids. The original density of PVC-NS will probably approach the density after boiling if it is left in water without boiling. Considering this, it is unlikely that the original density shown in Figure 3 is the dry density, as PVC-NS was in contact with water for a short time when its original density was measured. When the density of MPs exceeds the density of the liquid due to boiling, it has a significant impact on density sorting, as in the case of PS-FT and PVC-NS. However, as mentioned above, the original density is unclear. PS-FT and PVC-NS would be heavy MPs, not light MPs as originally categorized.



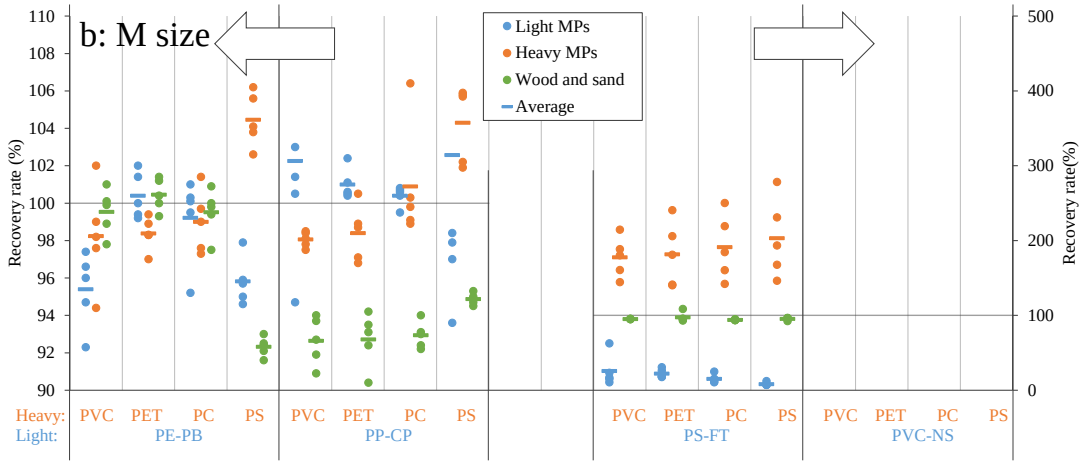


Figure 5. *Recovery rates of light MPs, heavy MPs, and wood and sand mixtures in multi-stage Flotation sorting. (a) n = 6, L size MPs (1−4.75 mm); (b) n = 5, M size MPs (0.212−1 mm).*

Boiling had a minimal impact on MP weight, with most samples losing less than 1%. Thus, heat treatment had a limited impact on the weight of MPs during the analysis. Even for MPs that showed a significant change in density due to boiling, the maximum weight loss was 2%. This indicates that the rate at which microplastics disappear due to boiling is low. PS-EP, which showed a 500% change in density, had a weight loss of less than 1%.

Previous comprehensive studies used heat as a pre-treatment to digest organic matter attached to the surface of MPs [1, 39, 40], followed by drying of the sediment sample [40-42] to allow for accurate measurement of MPs [13]. The NOAA manual also recommends heat treatment [17]. Plastics can be damaged/degraded by heat, light, UV radiation, high salinity, elevated microbial activity, and other factors [2, 4, 6, 43]. Some types of MPs were completely lost after boiling in water; boiling tests confirmed that temperatures higher than 70 ℃ were responsible for the loss [35]. On the contrary, another study found no significant damage to MPs even after heating at 75 ℃ for 30 min [44]. A future study will be required to clarify which types of MPs are susceptible to heat treatment.

4.2. Accuracy of Multi-Stage Flotation Sorting of MPs from Sand-Containing Impurities

In multi-stage Flotation sorting, a mixture of materials is sorted by density. The sorting accuracy is high if the ratio of the recovered weight to the added weight is around 100%. When PE-PB and PP-CP were used as light MPs, multi-stage Flotation sorting resulted in high recovery rates for both light and heavy MPs and the wood and sand mixture in the range of 90% to 110% (Figure 5, left side). The variation in recovery rate was greater when M-sized MPs were used instead of L-sized MPs, suggesting that the presence of smaller particles can lower sorting accuracy. Particle size is a vital factor in establishing the extraction potential of MPs [26, 45]. Multiple mixing ratios of light to heavy MPs were used, with differences of up to four-fold ((1.0/0.5)/(0.5/1.0) = 4); however, the recovery rates were within the range of 90% to 110%. In other words, there were no misjudgments between the light and heavy MPs.

When PS-FT and PVC-NS were used as light MPs, their recovery rates were 63% or lower, whereas the recovery rates for heavy MPs were 140% or higher. It is possible that the density of the light MPs increased due to boiling, resulting in a behavior similar to that of heavy MPs. However, as mentioned above, PS-FT and PVC-NS would be heavy MPs, not light MPs as originally categorized.

However, when the recovery rate was calculated for MP mixtures (light and heavy MPs) without distinguishing between light and heavy MPs, it fell within the range of 90% to 110%. This means that multi-stage sorting has high accuracy, at least for sorting MPs from other materials such as wood and sand.

For all samples and mixing ratios, the recovery rate was in the range of 90% to 110% for the wood and sand mixture, suggesting that wood and sand were separated from the MPs without misjudgment and losses.

False positives- misidentifying non-plastic particles as microplastics—can lead to an overestimation of pollution levels, causing unnecessary concern and potentially misguiding environmental policies. Even with a high recovery rate (90% to 110%), inaccurate identification reduces the reliability of results. To prevent this, studies should employ advanced validation methods such as FTIR or Raman spectroscopy. Ensuring accuracy provides a clearer understanding of pollution levels and supports more informed decision-making in research and policy.

However, this method could play a key role in advancing microplastic monitoring by providing more accurate, reliable, and efficient detection of microplastics in marine environments. With precise quantification of microplastic pollution, it can help identify the specific sources and concentrations of pollution in sediments and sands. This data is crucial for

shaping policies and regulations aimed at reducing plastic waste. For instance, industries like shipping, fishing, and coastal development could be held more accountable for their plastic discharges, leading to stricter regulations, improved waste management practices, and the development of technologies to minimize microplastic pollution. By offering better monitoring tools, this method would enable more targeted and effective strategies to reduce microplastic contamination in coastal ecosystems.

4.3. Limitations and Future Directions

The study focused on a specific set of MPs with known densities, which were synthetically created from commercially available plastic. However, environmental MPs from real-world samples may exhibit different physical and chemical properties, potentially affecting their behavior in density sorting. Therefore, it is essential to conduct studies using naturally occurring MPs from beach sand and marine sediments to validate the method's accuracy under real-world conditions.

Plastics degraded by UV exposure may undergo greater density and weight changes in response to heat treatment. This study primarily focuses on the effects of heat on the density and weight of MPs, rather than their chemical changes. However, heat treatment can induce both physical (e.g., size and shape) and chemical alterations in MPs. We recommend that future studies assess these changes using spectroscopic analysis (e.g., FTIR, Raman spectroscopy) to determine whether heat affects MP properties and to evaluate any heat-induced changes in MPs.

This study did not compare multi-stage flotation sorting with other sorting methods, such as filtration, sieving, chemical and enzymatic digestion, or spectroscopy. Future research should assess the effectiveness of multi-stage flotation sorting in comparison to other conventional MP separation techniques for sorting MPs from marine sediments. This should include a control experiment using a standard sorting method to determine the most effective approach for different environmental samples.

The study does not explicitly clarify how false positives and false negatives were addressed. To avoid false positives and negatives in MP identification, we utilized MP ratios. However, there remains a possibility that some microplastics (MPs) were misclassified due to density changes after boiling. Post-boiling, density alterations may have caused some MPs to shift between floating and sinking fractions, leading to misclassification. This could result in false negatives if MPs moved to an unexpected fraction and were overlooked or false positives if non-MPs were incorrectly identified as MPs. A future study incorporating control experiments, such as pre- and post-boiling MP characterization using spectroscopy (e.g., FTIR or Raman), would help clarify these classification uncertainties.

In addition, parameters (explanatory variables) that explain changes in density and weight (dependent variables) were not explored. At least, we were able to consider explanatory variables in two categories: MPs with and without air voids. A future study is warranted to evaluate the separation accuracy when samples collected from a sandy beach are subjected to multi-stage Flotation sorting.

5. Conclusions

This study evaluated the accuracy of multi-stage flotation sorting, which separates microplastics in beach sand based on density. The effects of heat treatment on the density and weight of MPs were also evaluated, as boiling is performed to eliminate wood fragments contained in beach sand as an impurity that interferes with density sorting. The main findings are summarized below.

(1) Most samples showed density changes and weight losses of less than 4% and 1%, respectively, suggesting that the effects of boiling were minimal, and hence, heat treatment had a limited impact on MPs during the analysis.

(2) Unless boiling causes a density change, multi-stage Flotation sorting had high recovery rates in the range of 90% to 110% for light and heavy MPs and the wood and sand mixture. In other words, light and heavy MPs and the wood and sand mixture were separated without misjudgment and loss.

The accuracy of the multi-stage flotation sorting process is influenced by the particle size of MPs, with smaller particles showing more variability in recovery rates. This method effectively separates MPs based on their densities, providing useful insights into their origins and potential for re-drift, although it cannot determine the material composition of the MPs. Overall, this approach presents a reliable technique for analyzing MPs in beach sand, especially when addressing common contaminants like wood.

Abbreviations

MP	Microplastic
NOAA	National Oceanic and Atmospheric Administration
SCC	Saturated Calcium Chloride

Supplementary Material

The supplementary material can be accessed at https://doi.org/10.11648/j.ajep.20251402.12

Acknowledgments

Special thanks are extended to Ms. Yui Chinju and Ms. Yukari Tokiyasu.

Author Contributions

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Hiroshi Asakura: Conceptualization, Methodology, Visualization, Supervision, Project administration, Funding acquisition, Writing – review & editing

Funding

This work is supported by the JAPAN SOCIETY FOR THE PROMOTION OF SCIENCE (JSPS), a Grant-in-Aid for Scientific Research C (20K12208). The APC was funded by NAGASAKI UNIVERSITY.

Data Availability Statement

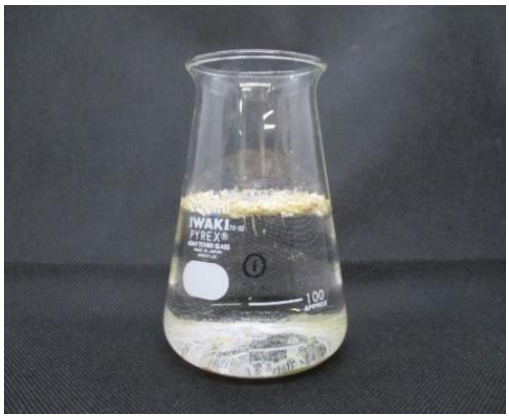
Data are available in the Supplementary Materials.

Conflicts of Interest

The authors declare no conflicts of interest.

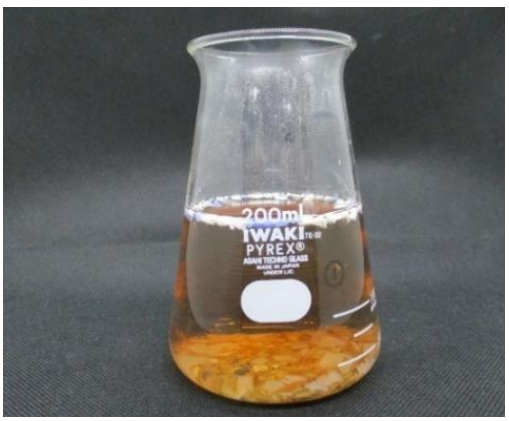
Appendix

Step 1: Weighing samples

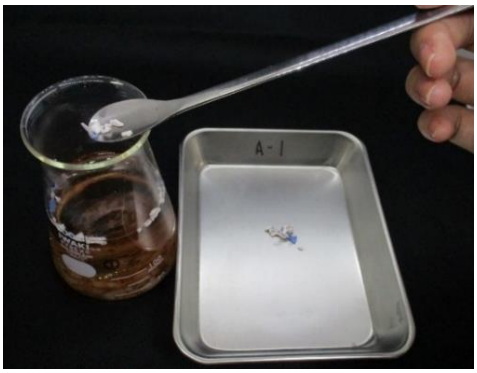


Step 2: Beaker containing water and samples

Step 3: Heating beakers



Step 4: Beaker after boiling



Step 5: Collecting light MPs

Step 6: Removing water

Step 7: Adding SCC

Step 8: Floating of heavy MPs

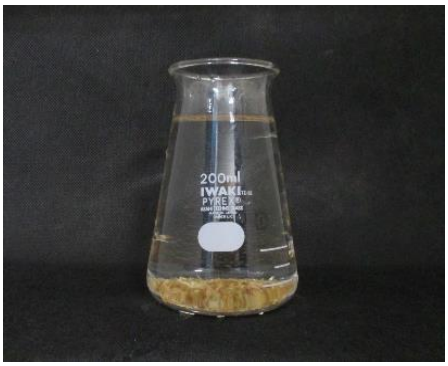
Step 9: Collecting heavy MPs

Step 10: Washing heavy MPs

Step 11: Removing SCC

Step 12: Measuring SCC density

Step 13: Dilute washing of wood and sand



Step 14: Washing wood and sand

Figure A1. *Procedure for multi-stage Flotation sorting.*

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