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# Controlling Small Particles for Two-Step Density Sorting of Simulated Microplastics: Overcoming Surface Tension Effects with Surfactants

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## ABSTRACT

Infrared spectrometers are commonly recommended for analyzing microplastics (MPs) in sediment samples. However, these instruments are costly and time consuming, limiting the scope of surveys and our understanding of the distribution and long-term variation of MPs. Although it is challenging to determine MPs by floatation sorting, it is possible to estimate the ratio of MPs that float and sink in seawater. The study employed floatation sorting to confirm whether MPs with densities lower than the liquid float and those with densities higher sink, even for MPs smaller than 1 mm. As expected, large MPs (1 to 4.75 mm in size) with densities higher than that of the liquid sank. Unexpectedly, small MPs (212 µm to 1 mm) with densities higher than the liquid's density also floated. Assuming the unexpected floating was due to surface tension, we added a surfactant to lower it, causing MPs with densities higher than the liquid's to either sink as expected or accelerate sinking. Thus, with the use of a surfactant, even small MPs can be sorted by density if a heavy liquid is used after water.

## HIGHLIGHTS

- Large MPs (1 to 4.75 mm) with densities higher than those of the liquid sink as expected.
- Small MPs (212 µm to 1 mm) with densities higher than the liquid may unexpectedly float due to surface tension.
- Adding a surfactant reduces surface tension, accelerates the sinking of small MPs.
- Using a heavy liquid after water enables effective density-based sorting of small MPs.

## 1. INTRODUCTION

Plastics have become an indispensable material that is widely used in all aspects of our daily lives owing to their low price, durability, versatility, lightness, water repellency, and ductility (Luo et al., 2022; Li et al., 2018). Global production of plastics increased from 245 million tons in 2008 to 390.7 million tons in 2021 (Shukla et al., 2024), and is predicted to reach 600 million tons in 2050 (Yoganandham et al., 2023). However, only 6 to 26% of these plastics are recycled and the remaining 94% become plastic waste that continuously accumulates in landfills or directly enters the environment through various routes (Yang et al., 2022; Huang et al., 2021).

One of the most critical problems brought about by plastic waste is microplastics (MPs). MPs are small plastic particles with sizes less than 5 mm that have entered and polluted the environment (Wang et al., 2022; Wu et al., 2019). MPs are present in all marine ecosystems at varying concentrations and approximately 245 million tons are discharged into the marine environment annually (Alimba and Faggio, 2019). MPs have been widely detected in marine

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sediments, river sediments, soil, air, freshwater, wastewater, food, multiple organisms, and terrestrial ecosystems in recent decades (Chang et al., 2022; Huang et al., 2022; Cutroneo et al., 2021). MPs have a large hydrophobic surface and a rigid organic structure that can adsorb a variety of organic and inorganic pollutants, such as polycyclic aromatic hydrocarbons, polychlorinated biphenyls, heavy metals (including Cu, Zn, Cd, Cr, Pb, Co, Ni, Mn, Fe, Ag, and Hg), pharmaceuticals, and personal care products. Pollutant adsorption by MPs may lead to pollutant enrichment, which may increase local concentration in soil and exert combined effects on plants (Yang et al., 2022; Xiang et al., 2022). Therefore, it is necessary to know the distribution and long-term trends of MPs to prevent environmental pollution.

MPs have been detected in the surface and subsurface waters of the Atlantic Ocean, the Northeastern Pacific Ocean, and Arctic Polar waters, as well as in the surface waters of the North Sea, the Adriatic Sea, the Bohai Sea, and the South China Sea (Akdogan and Guven, 2019). The largest regional releases of MPs are in India and South Asia (18.3%), followed by North America (17.2%), Europe and Central Asia (15.9%), China (15.8%), East Asia and Oceania (15.0%), South America (9.1%), and Africa and the Middle East (8.7%) (Ang et al., 2022). Examples of MP densities in sandy beaches/coastal areas include 45-220 particles/kg (p/kg) in India (Tiwari et al., 2019), 232 p/kg in Bangladesh (Banik et al., 2022), 2.4-2.8 p/kg in the USA (Plee and Pomory, 2020), 338-1,270 p/kg in Norway (Olsen et al., 2020), 60-610 p/kg in South China (Zhang et al., 2019), and 61 p/kg in Mexico (Beckwith and Fuentes, 2018).

The separation methods of MPs in collected beach sediment samples can be classified mainly into physical, chemical, and biological methods (Tirkey and Upadhyay, 2021). The U.S. National Oceanic and Atmospheric Administration's (NOAA) manual for analyzing MPs in beach samples (Masura et al., 2015) comprehensively specifies the separation and analysis of MPs. Many studies have focused on MP analysis in such environmental media as marine, sand, and sediment (Soursou et al., 2023; Nabi et al., 2022) using the density separation (floatation) method (Tiwari et al., 2023; Nabi et al., 2022; Prata et al., 2019). The floatation method is easy and quick to perform and widely used to isolate/extract MPs from sand and sediment samples using a saturated salt solution (Crutchett and Bornt, 2024; Zhang et al., 2021). Salts for density separation include sodium chloride (NaCl), sodium iodide (NaI), zinc chloride ($ZnCl_2$), calcium chloride ($CaCl_2$), manganese (II) sulfate ($MnSO_4$), potassium formate ($CHKO_2$), and sodium polytungstate (SPT) (Soursou et al., 2023; Tirkey et al., 2021; Van Cauwenberghe et al., 2015). These heavy liquids are used to float not only MPs with low densities but also MPs with high densities that sink in water.

The NOAA manual recommends the use of an infrared spectrometer for the analysis of separated MP samples. This instrument determines not only whether the collected particles are plastic or not, but also the type of plastic material, thus facilitating the identification of the source of MPs. However, if material determination were required during MP surveys, the surveys would be limited to those conducted by professional researchers. Aside from being costly and time-consuming, the use of an infrared spectrometer limits surveys and prevents us from understanding the distribution and long-term variation of MPs. GESAMP (2019) recommends that surveys be conducted by citizen scientists to gather more information on the environment. Even though a survey by citizen scientists alone cannot determine the materials of MPs, it can provide supporting data on the materials. Asakura (2022) performed floatation sorting of MPs larger than 1 mm in size using water and saturated calcium chloride (SCC) solution and confirmed that MPs with densities lower than the density of the liquid floated and those with densities higher than the density of the liquid sank. This means that MPs can be sorted into two density levels if SCC solution is used after water. Although it is impossible to determine the materials of the MPs, it would be possible to estimate the ratio of MPs that float to those that sink in seawater. Assuming a certain land area, MPs with a specific gravity larger than 1 would originate not from ocean debris but from higher elevations on land. On the other hand, in the case of MPs with a specific gravity smaller than 1, it cannot be distinguished whether they drifted from the sea or land, but the MPs can be evaluated as having the potential to re-drift into the ocean. In this way, knowing the ratio of floating to sinking MPs in beach sediments, in addition to the amount of MPs present, gives us additional information about the current level of contamination and the possibility of contamination in the surrounding area, even without using an infrared spectrometer.

MPs larger than 1 mm in size are relatively large particles. Do small MPs behave as expected, i.e., as reported in Asakura's study? This is because large and small particles have different specific surface areas and are therefore affected differently by surface tension. In this study, we address the following questions. (1) Do MPs measuring less than 1 mm switch between floating and sinking depending on the density of the liquid? (2) If the MPs do not show the expected behavior as shown in (1), is there any way to improve the situation?

## 2. METHODOLOGY

### 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, and 4.75 mm mesh sizes were utilized to adjust particle size distribution. For density measurements of MPs (L-size used), several 50 mL pycnometers, a thermometer (TT-508N, TANITA), a precision balance (ATY124, Shimadzu), and a water purifier (RFP841AA, ADVANTEC) were used. For the floatation sorting experiment, 300 mL glass beakers, a stainless-steel spoon, stainless-steel trays, and a dryer (DRD420DA, ADVANTEC) were used. For liquid density measurement, a graduated cylinder and a hydrometer (Ludwig Schneider) were utilized.

*2.1.2 Samples*

To prepare simulated MP samples, several types (PE, PP, PS, PVC, PET, PC, and PF) of plastic products (Table 1) were crushed and passed through a stainless-steel sieve to obtain L- (1 mm to 4.75 mm) and M- (212 µm to 1 mm) sized MPs.

The density of L-sized MPs was measured (Asakura, 2022). MPs with densities lower and higher than 1 g/cm$^3$ are called light and heavy MPs, respectively (Table 1). Saturated calcium chloride (SCC, Miyachu Building Materials Division, Inc.) solution was used as a heavy liquid for floatation sorting experiments of MPs because SCC is environmentally friendly and affordable (Debraj and Lavanya, 2023). Commercial kitchen detergent (Soapen Fresh Lime, Kaneyo Soap Co., Ltd.) was used to prepare the diluted detergent solution (hereinafter referred to as surfactant).

**Table 1.** Details of plastic products

| Material | Description | Prepared MP size* for Figure 3 | | | | | | Density (g/cm$^3$) | Light / Heavy |
|---|---|---|---|---|---|---|---|---|---|
| | | L size | | M size | | | | | |
| | | W | C | W | WS | C | CS | | |
| Polyethylene (PE) | Shopping bag (SB) | x | | | | | | 0.908 | L |
| | Polybottle (PB) | x | x | x | x | x | x | 0.934 | L |
| | Rope (RP) | x | | | | | | 0.754 | L |
| | Glove (GV) | x | | | | | | 0.871 | L |
| | Freezer bag (FB) | x | | x | | | | 0.919 | L |
| Polypropylene (PP) | PET bottle cap (BC) | x | | x | | | | 0.925 | L |
| | Flat plate (FP) | x | | x | | | | 0.867 | L |
| | Clothespin (CP) | x | x | x | x | x | x | 0.905 | L |
| | Rope (RP) | x | | | | | | 0.486 | L |
| | Oriented PP (OP) | x | | | | | | 0.888 | L |
| Polystyrene (PS) | Expanded polystyrene (EP) | x | | x | x | | | 0.018 | L |
| | Flat plate (FP) | x | x | x | x | x | x | 1.084 | H |
| | Plastic bottle label (LB) | x | x | x | x | | | 1.031 | H |
| | Compact disk case (MC) | x | x | x | x | | | 1.054 | H |
| | Food tray (FT) | x | x | x | | x | x | 0.981 | L |
| Polyvinyl chloride (PVC) | Pipe (PI) | x | x | x | x | x | x | 1.424 | H |
| | Flat plate (FP) | x | x | x | x | x | x | 1.333 | H |
| | Corrugated plate (CP) | x | x | x | x | x | x | 1.375 | H |
| | Tablecloth (TC) | x | x | | | | | 1.305 | H |
| | Non-slip sheet (NS) | x | x | x | | | | 1.218 | H |

**Table 1.** Details of plastic products (cont.)

| Material | Description | Prepared MP size* for Figure 3 | | | | | | Density (g/cm$^3$) | Light / Heavy |
|---|---|---|---|---|---|---|---|---|---|
| | | L size | | M size | | | | | |
| | | W | C | W | WS | C | CS | | |
| Polyethylene terephthalate (PET) | PET bottle (EB) | x | x | x | x | x | x | 1.378 | H |
| | Egg pack (EG) | x | x | x | x | | | 1.315 | H |
| | Lumirror ® film (LF) | x | x | x | x | | | 1.390 | H |
| | Fruit container (FC) | x | x | x | x | | | 1.336 | H |
| Polycarbonate (PC) | Compact disk (CD) | x | x | x | x | x | x | 1.163 | H |
| | Safety glasses (SG) | x | x | x | x | x | x | 1.166 | H |
| | Flat plate (FP) | x | x | x | x | | | 1.166 | H |
| Phenol-formaldehyde (PF) | Pot knob (PK) | x | | x | x | | | 1.469 | H |

## 2.2 Methods

### *2.2.1 Principle of floatation sorting*

The present study was conducted with light and heavy plastics to determine plastic behavior in individual floatation experiments. Figure 1 shows an ideal floatation sorting experiment. Weighed (indicated as initial amount, Figure 1) light or heavy plastics are added to individual beakers containing deionized water and stirred with a spoon (Ideal 1 and Ideal 2, Figure 1). After waiting for a while, the light plastics (indicated as floating matter, Figure 1) float on the water surface and are recovered using a spoon (Ideal 1, Figure 1) as the density of light plastics is lower than water density. On the other hand, heavy plastics settle at the beaker's bottom and thus cannot be collected from the water surface (Ideal 2, Figure 1). To collect the heavy plastics, water in the beaker is exchanged with a heavy liquid (i.e., SCC). The heavy plastics float on the liquid surface and are recovered by a spoon (Ideal 3, Figure 1). In this way, light and heavy plastics can be collected separately (two-step density sorting) from mixtures if floatation sorting is performed in water followed by a heavy liquid (Ideal 4, Figure 1).

However, errors occur in actual floatation sorting. Figure 2 shows the failure of the floatation sorting experiment. After mixing light plastics with water, some behave like heavy plastics, sinking to the bottom, whereas those that float on the water's surface are recovered by a spoon (Failure 1, Figure 2). The sinking is believed to result from binding with other particles or the beaker's inner surface. On the other hand, after mixing heavy plastics with water, some behave as light plastics, floating on the water's surface, and these are recovered (Failure 2, Figure 2). This is thought to be due to surface tension or combination with air bubbles. This unexpected behavior of plastics can lead to errors in the estimation of floating and settling fractions. The ratio of the amount recovered to the initial amount is used to determine the recovery rate. In this study, the weight of the particles is measured rather than the number. The unexpected behavior of heavy plastics can be prevented by adding a surfactant to the water to reduce surface tension and promote particle settling at the bottom (Solution for Failure 2, Figure 2).

### *2.2.2 Procedure for floatation sorting experiment*

The floatation sorting experiment of single MPs (L and M size) was conducted to observe light and heavy plastic behavior in several liquids such as deionized tap water, SCC, tap water with surfactant, and SCC with surfactant. The concentration of the surfactant was 1/1,000 of the original solution. First, dry trays were weighed. The beaker (300 mL) was filled with 300 mL of liquid and measured (0.5±0.005 g) MPs of one plastic species (n=6 for L size and 5 for M size) were added to it. Due to experimental constraints, preparing simulated M-sized MPs is more challenging than preparing L-sized MPs, which is why the number of M-sized replicates is reduced. The liquid in the beaker was gently stirred with a spoon for 1 minute to accelerate the sinking or surfacing of MPs and remove air bubbles. After a few minutes, floating MPs were scooped out with a spoon and kept in a tray for drying in a dryer (80°C), and the dry weight was determined.

We verified whether the floating or sinking of MPs could be controlled by changing the density of the liquid. For this floatation sorting experiment, we used light (PC-CD, 1.163 g/cm$^3$) and heavy (PVC-FP, 1.333 g/cm$^3$) M-sized MPs (n=5). Water (1.00 g/cm$^3$) and low (diluted $CaCl_2$ solution, 1.30 g/cm$^3$), and high (SCC, 1.37 g/cm$^3$) density solutions with and without surfactants were used as liquids.

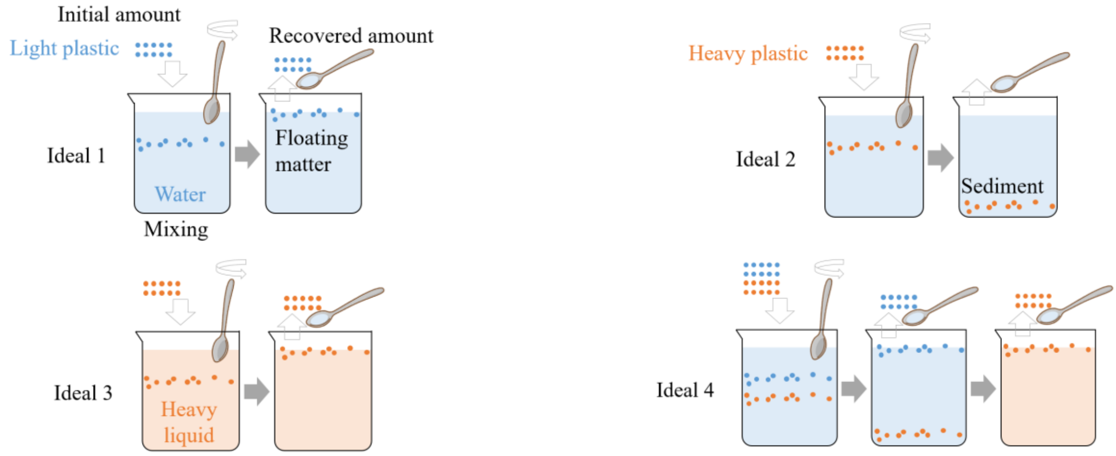


**Figure 1.** Ideal floatation sorting experiment

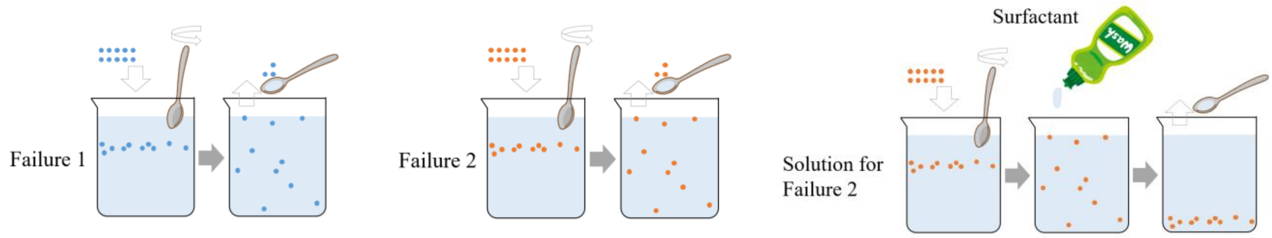


**Figure 2.** Failure and success in the floatation sorting experiment

## 3. RESULTS AND DISCUSSION

### 3.1 Floatation sorting of MPs using water and SCC solution

Figure 3 shows the relationship between the density of MPs and the recovery rate by floatation sorting. The L- and M-sized light MPs floated on the water's (1.00 g/cm$^3$) surface with recovery rates reaching nearly 100% (Figures 3(a), (b), (c)). In water, most of the L-sized heavy MPs settled at the bottom with a recovery rate of approximately 0% (Figure 3(a)). In contrast, some M-sized heavy MPs tended to float, resulting in a higher recovery rate than the L-sized heavy MPs (Figure 3(b), area shaded in orange). When a surfactant was added to water, the M-sized heavy MPs settled and the recovery rate dropped to nearly 0%, except in the case of PS-FP (Figure 3(c), area shaded in orange).

The L- and M-sized MPs, whose densities are lower than that of SCC (1.37 g/cm$^3$), floated to the surface, resulting in almost 100% recovery rates (Figures 3(d), (e), (f)). For MPs with densities higher than that of SCC, the L-sized MPs settled at the bottom (Figure 3(d)), while the M-sized MPs floated to the surface, resulting in a 100% recovery rate (Figure 3(e)). However, when a surfactant was added, the sedimentation of the M-sized MPs was accelerated, and the recovery rate decreased to 86% (Figure 3(f)).

In any case, the presence or absence of a surfactant did not influence the approximately 100% recovery rates of MPs whose densities are lower than that of the liquid.

### 3.2 Floatation sorting of MPs using diluted $CaCl_2$ solution

Figure 4 shows the relationship between the volume mixing ratio (heavy liquid/pure water) and the density of the liquid mixture. The predicted density is calculated using the following equation.

$$\text{Predicted density} = \frac{\text{Water density} + (\text{Mixing ratio} \times \text{Heavy liquid density})}{(1 + \text{Mixing ratio})}$$

The actual density of the liquid mixture is almost equal to the predicted density. This means that liquids with the required density (between water and SCC) can be easily prepared.

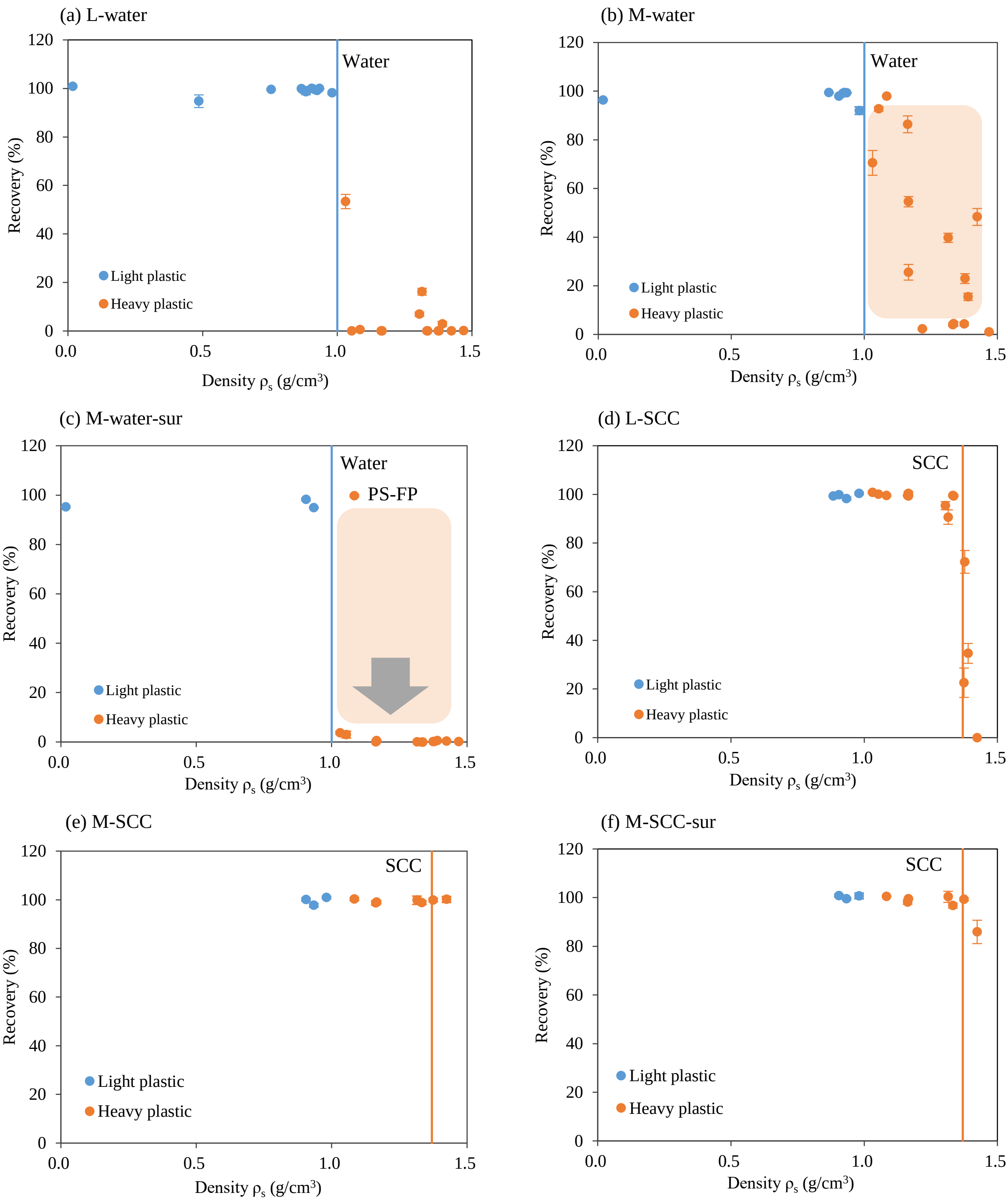


**Figure 3.** Relationship between the density of MPs and the recovery rate by floatation sorting. n=6 (L: 1 mm to 4.75 mm) or 5 (M: 212 µm to 1 mm). Vertical line: density of liquid (water or SCC). Error bar: standard error (α=0.05). SCC: saturated $CaCl_2$; sur: surfactant.

We also examined the behavior of M-sized MPs by using light (PC-CD) and heavy (PVC-FP) MPs in liquids with various densities. We found that the recovery rate varied with the density of the liquid (Figure 5). When water (1.00 g/cm$^3$) was used as a liquid, PC-CD (1.163 g/cm$^3$) floated on the water surface and the recovery rate was 86%. In contrast, when a surfactant was used with water, PC-CD settled at the bottom and the recovery rate was 0.1%. PVC-FP (1.333 g/cm$^3$) settled when water and water with a surfactant were used as the liquid, and the recovery rate was approximately 4.0% and 0.0%, respectively.

In the diluted $CaCl_2$ solution (1.30 g/cm$^3$), PC-CD (1.163 g/cm$^3$) floated, achieving a recovery rate of

98%. In the same solution, some PVC-FP (1.333 g/cm³) also floated, with a recovery rate of nearly 30%. However, after adding a surfactant, most of the PVC-FP settled, reducing the recovery rate to just 2.9%.

In SCC (1.37 g/cm³), both PC-CD and PVC-FP floated regardless of the presence or absence of the surfactant, and the recovery rate exceeded 96%.

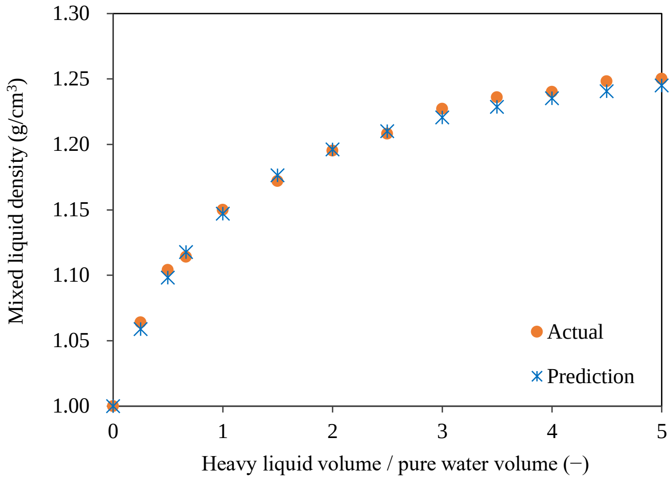


**Figure 4.** Relationship between volume mixing ratio and density of liquid mixture when heavy liquid (SCC) is mixed with pure water.

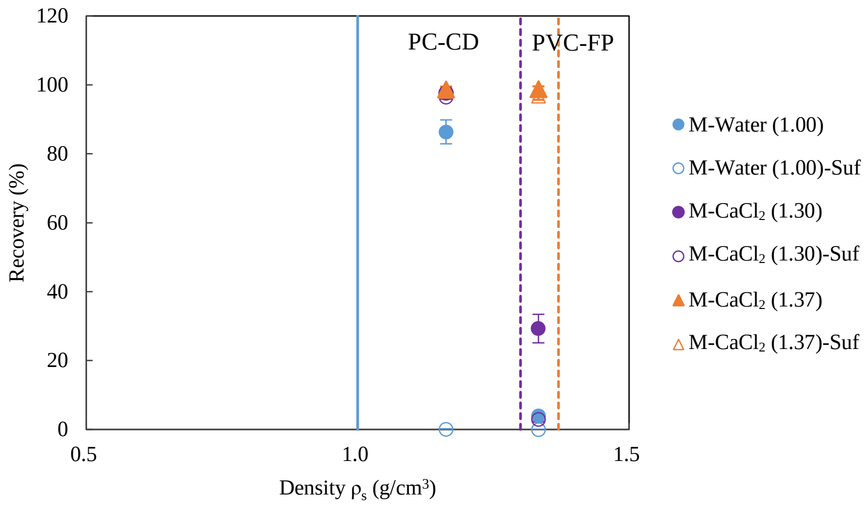


**Figure 5.** Recovery rates of MPs in liquids with various densities. Values in parentheses in the legend indicate the density of the liquid used (g/cm³); the colors of the symbols match the colors of the lines representing the density of the liquid used. n=5. Size: M (212 µm to 1 mm). Vertical lines: density of liquids. Error bar: standard error (α=0.05).

## 4. DISCUSSION

### 4.1 Behavior of small MPs during floatation sorting

In floatation sorting, particles with densities lower than the density of the liquid used should float and particles with densities higher than that of the liquid used should sink (expected floatation behavior). As shown in Figures 3(a) and 3(d), L-sized (1 mm to 4.75 mm) MPs exhibited the expected sinking behavior in water and SCC. However, M-sized (212 µm to 1 mm) MPs with densities higher than that of the liquid floated, contrary to expectations (Figure 3(b) (area shaded in orange) and Figure 3(c)). This means that MPs with low densities are overestimated (and MPs with high densities are underestimated) in the two-step density sorting. Assuming that this unexpected floating was due to surface tension, we added a surfactant to lower the surface tension, and MPs with densities higher than that of the liquid either sank as expected (Figure 3(c), area shaded in orange) or showed accelerated settling (Figure 3(f)), except for a few.

It is easy to dilute SCC to create a liquid with the desired density. The actual densities agreed with the predicted densities obtained from the calculation

(Figure 4). Even in liquids with densities between those of water and SCC, some M-sized (212 µm to 1 mm) MPs with densities higher than that of the liquid floated, contrary to expectations, but sank as expected when a surfactant was added (Figure 5).

MPs with densities lower than that of the liquid floated as expected regardless of the density of the liquid or the presence or absence of surfactant (Figures 3 and 5). This means that MPs with low densities are not underestimated in the two-step density sorting.

Various brine solutions including NaCl, NaBr, NaI, and $ZnBr_2$ were used in density separation experiments to recover different MPs (Nabi et al., 2022; Zhang et al., 2021). Several MPs (500 µm to 3 mm; PE, PP, PVC, PET, PS, EPS, and PUR) exhibited recovery rates of 99%, 96%, 97%, 91%, 92%, 68%, and 96%, respectively, from marine sediments when a saturated NaCl solution (1.2 $g/cm^3$) was used first, followed by a NaI solution (1.8 $g/cm^3$) in density separation experiments (Nuelle et al., 2014). The recovery rates of PVC and PET, which have higher densities than the NaCl solution (Table 1), exceeded 90% due to the use of a high-density NaI solution. Our research results can corroborate this result. Quinn et al. (2017) extracted MPs from marine sediments using tap water and several saturated salt solutions of varying densities including NaCl, NaBr, NaI, and $ZnBr_2$. The recovery rates were higher for smaller (200 to 400 µm) MPs than for larger (800 to 1,000 µm) MPs, increasing as the liquid density increased. We also found that the recovery rates were high for small particles. Other density separation experiments gave similar results—MP size influenced the recovery rate, namely, small MPs have higher recovery rates than large MPs (Nabi et al., 2022; Vermeiren et al., 2020; Coppock et al., 2017). If we simply want to recover small MPs by floating sorting, no countermeasures are necessary, and we will be able to recover more heavy MPs than expected. However, to separate MPs by density, we need a surfactant to control the flotation behavior of small MPs, as indicated by this study's results.

Based on the present study we conclude that the improved density sorting method can be used in environmental monitoring and microplastic research to obtain more reliable data on MP distribution in sediments and water bodies aiding in pollution assessment and mitigation efforts.

### 4.2 Limitations and further study

Some soft plastics were difficult to shred and did not yield M-sized particles. MPs whose surfaces have been degraded by sunlight or contaminated with microorganisms or oil may behave differently from undamaged MPs.

In this study, we performed a preliminary two-step density sorting experiment to separate light plastics from heavy ones. In the future, we will conduct a two-step density sorting experiment to identify factors that inhibit expected sorting efficiency.

This study serves as a foundational investigation for the density sorting of simulated MPs. However, in the future, we will validate the method using real environmental samples, particularly sediment from sea beaches and seawater, with added MPs of known quantities to assess its applicability.

In this study, we used tap water as a substitute for seawater because the densities of tap water (1.00 $g/cm^3$) and seawater (1.03 $g/cm^3$) are very similar. However, in future work, we plan to focus on using seawater to better align with real-world conditions.

## 5. CONCLUSION

We examined whether MPs with densities lower than the liquid density float and MPs with densities higher than the liquid density sink by performing floatation sorting experiments. We included MPs less than 1 mm in size. Our main findings are described below.

(1) Large MPs (1 mm to 4.75 mm) with densities higher than that of the liquid sank, as expected. On the other hand, small MPs (212 µm to 1 mm) with densities higher than that of the liquid floated, contrary to expectations. MPs with densities lower than that of the liquid floated, as expected, regardless of the liquid density and the presence or absence of surfactant.

(2) Assuming that the unexpected floating was due to surface tension, we added a surfactant to lower the surface tension, and MPs with densities higher than that of the liquid either sank or showed accelerated sinking, except for some MPs.

Thus, with the aid of a surfactant, even small MPs can be sorted into two groups with different densities if heavy liquid is used after water.

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## AUTHOR CONTRIBUTIONS

Conceptualization, Methodology, Visualization, Supervision, Project Administration and Funding Acquisition, H. Asakura; Investigation and Writing - Original Draft Preparation, M.A. Islam; Writing - Review and Editing, H. Asakura, S.A. Mamun, K. Nakagawa, K. Shimizu, M. Yagi, A. Ussawarujikulchai.

## DECLARATION OF COMPETING INTEREST

The authors declare no conflict of interest.

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