



# **Communication The Standard and Reverse Mode Operation of a Hydrocyclone for Microplastic Separation**

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**Abstract:** Harmonization in the analytical framework is needed to detect, define and further categorize plastics released into the environment. In the range of particles smaller than 200  $\mu$ m, hydrocyclones (HCs) have proven their capacity in removing microplastics efficiently by offering technical advantages at low operational costs. This publication aims to expand scientific knowledge by introducing four commercially available, low-priced microplastics to a pilot-scale HC setting. The physicochemical characteristics of particles as well as the separation efficiency of the test rig were investigated in depth. Particles with a density of >1000 kg/m<sup>3</sup> passed the primary vortex and were discharged into the underflow, allowing us to employ standard mode operation. Particles with a density of <1000 kg/m<sup>3</sup> entered the secondary vortex and were removed through the overflow. As expected, separation efficiencies were found to be higher for particles revealing a greater density difference when compared with the mobile phase water. Furthermore, an increase in the inlet volume flow revealed significant positive impacts on the separation efficiency for three plastics to a certain threshold. Data on standard and reverse mode operations presented in this publication can lay out an important source for the harmonization and standardization of future HC research, with the goal of overcoming plastic pollution by developing economically competitive separation processes.

**Keywords:** hydrocyclone; particle separation; reverse mode operation; separation efficiency; plastic debris

## 1. Introduction

Plastic products have become indispensable in our daily life [1], but their extensive production in combination with an underdeveloped waste management have caused a significant disposal of plastic debris in different sizes virtually present in all environmental compartments [2]. Macroplastics enter the environment directly or indirectly, depending on the state of waste management in its various sectors of use. Due to the influence of UV radiation from the sun and mechanical impacts, macroplastic particles later break down into secondary microplastics and nanoplastics [3,4]. Primary microplastics are incorporated directly into products (e.g., cosmetics and paints) as a functional ingredient and find their way into the environment through their application. The ubiquitous presence of microplastics (MPs) in all aquatic and terrestrial environments [5] up to the highest mountain ranges [6,7] together with their uncertain mechanical and toxicological effects pose a significant threat for all species including human beings [8–11]. Hence, more and



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**Copyright:** © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). more international and national bills and directives address these polluters and call for a harmonization in the analytical frameworks in order to define and further categorize plastic debris as well as for standardized, accurate, traceable and robust methodologies, from sample preparation to detection, to obtain comparable data on the status of plastic pollution [12–14].

As reported before, the availability and validity of the respective data decrease with the size of investigated particles [15]. Manta trawls and other net systems (80–300  $\mu$ m) are tremendously discriminating particles smaller than their nominal mesh size; in situ pump systems and fractionated filtration devices allow a lower threshold. However, for the quantitative sampling of particles that are >10  $\mu$ m, filtration is impeded by fast clogging [15,16]. Consequently, centrifugal separators, such as hydrocyclones (HCs), that are widely applied in several industries (e.g., [17–20] and others) have found their niche in applications dealing with plastic pollution. Separation is based on density differences in the two phases, e.g., solid MP particles and a liquid medium, and the principle of two forces. A centrifugal force pushes the phase with the higher density to the wall, creating a primary vortex that passes through the underflow and a drag force pulling the phase with the lower density into the center, thereby creating the secondary vortex leaving through the overflow (vortex finder) [21]. HCs bear the advantage of good scalability, high throughput, continuous operation, structural simplicity at low operation costs, low energy requirements and no secondary pollution [17,22].

Studies have revealed promising results for separating plastic particles in liquid matrices; however, they prominently rely on shredded plastic particles [1,23,24], which consequently bear difficulties when it comes to the comparison of design variables in the HC setup, e.g., the cone angle, the length of the cylindrical section and many more, thereby effecting the separation efficiency of MPs [23].

This study aims to investigate the separation characteristics of four important, commercially available and low-cost MP bulk solids in a bench-scale hydrocodone setting in depth. Authors propose that data on standard and reverse mode operations presented in this publication can lay out an important source for the harmonization and standardization of future HC-based research, with the goal being to overcome plastic pollution by developing economically competitive separation processes.

#### 2. Materials and Methods

#### 2.1. Material Selection and Characterization

The following plastics or elastomers, listed with their respective trade names, were investigated in this study: one high-density polyethylene (HDPE) powder (ET306010), one polypropylene (PP) powder (Eltex P KS001PF), one polystyrol (PS) powder (PrimeCast 101) and one polymethyl methacrylate (PMMA) powder (DEGACRYL LP 55/03) were obtained from four different companies (Table 1).

Three of these plastics—HDPE, PP and PS—were properly described in the preliminary publication of these authors [14]. Particle density, size distribution and the PMMA shape were determined following the same instructions presented in that previous study. Other basic information was taken from technical information reports and data sheets provided by the manufacturers. All the characteristics for PMMA are presented in Table 1. The particle size distribution of all four investigated plastics is stated in Figure 1.

#### 2.2. Separation Experiments

For particle separation, a test rig (see Figure 2, adapted from [25]) was used throughout the experiments. Water (20 °C) was pumped with the circulation pump P1 (KWPK065-040-0250, KSB Austria GmbH, Wiener Neustadt, AUT) from the clean water vessel through the flowmeter F001 (type S030, Bürkert Austria GmbH, Mödling, AUT) to the HC unit Z1. Test particles, introduced with a final feed concentration of 0.5 kg/m<sup>3</sup>, were injected using a metering device (piston injection with the stepper motor M4). The HC underflow was pumped (P2) through the flowmeter F002 (type 2551, Georg Fischer Piping Systems

Ltd, Schaffhausen, CH) to the absolute filter F1 (0.75  $\mu$ m; LLT-BFBE-2-304-1B and LT-AR-232-WS-1-P-R; HENNLICH GmbH, Suben, AUT) for the gravimetrical determination of the separated particles. The overflow was transferred to the absolute filter F2 (0.75  $\mu$ m; LLT-BFBE-2-304-2B and LT-AR-232-WS-2-P-R; HENNLICH GmbH, Suben, AUT). Dry mass of particles was determined after drying at 70 °C for 72 h (completely dry).

**Table 1.** Results of the separation experiments according to variation of particle type, hydrocyclone operation mode and underflow rate. The  $\Delta max$  (+) and  $\Delta min$  (–) show the difference between the average value and the minimum and maximum measured value.

Hydrocyclone Operation	Manufacturer	Particle Type	Particle Name	Particle Density	Median Diameter	Inlet Flow Rate	Underflow Rate	Separation Efficiency	Δmax (+)	Δmin (–)
-	-			$kg/m^3$	μm	m <sup>3</sup> /h	%	%	%	%
Standard mode	FVONIK	PMMA	Degacryl	1200.6	520	5	0	98.42	0.33	0.29
			LP 55/03	1200.0	520		10	98.54	0.32	0.34
	EOS	PS	PrimeCast 101	1068.7	85	5	0	3.01	2.18	1.15
							10	20.69	0.62	0.42
							20	35.65	0.62	0.35
							30	50.32	0.45	0.89
							40	64.29	0.43	0.61
							50	79.00	1.19	2.34
							60	85.83	1.25	0.66
							70	90.36	0.65	0.39
							80	92.34	1.66	0.90
							90	93.85	0.66	0.69
Reverse mode	GOODFELLOV	W HDPE	ET306010 Eltex P KS001PF	949.6 881.9	118	5	0	- - 0.00		
							10			-
							20			
							30			
							40	0.15	1 50	1 50
							50	3.15	1.59	1.59
							60	13.96	1.37	1.37
							70	40.25	2.73	2.02
							80	89.01 00.12	2.90	2.94
							90	99.12	0.70	1.51
	INEOS	РР					10	0.00		-
							20			
							20	716	1 1 /	266
							40	23 58	0.70	2.00
							50		2.04	1.52
							60	71 68	0.65	1.02
							70	91.00	2.12	2.50
							80	96.76	1.04	1 20
							90	90.70	0.74	1.27
							20	22.20	0.74	1.55



**Figure 1.** Particle size distribution (PSD, cumulative) of the microplastic particles used in the experiments. Laser diffraction measurements were conducted using a Malvern Mastersizer 2000E (Malvern Panalytical Ltd., Malvern, UK) with diffraction indexes of 1.54 (ET306010), 1.49 (Eltex P KS001PF), 1.59 (PrimeCast 101) and 1.49 (Degacryl LP 55/03). Parts of the particle details were already published in a previous study [14].



**Figure 2.** Experimental setup of the test rig. FIC: flow indication control; LI: level indication; M: motor; P: pump; PD: pressure difference; PI: pressure indicator; S: hose; TI: temperature indication and V: Valve. Adapted and re-designed from a previous study of the authors [25].

Separation efficiency of dried particles was determined according to Equation (1) where  $\varepsilon$  is the separation efficiency,  $m_0$  the mass of the dried empty filter before the experiment and  $m_1$  the mass of the dried loaded filter.

$$\varepsilon = \frac{m_{1, filter1 - m_{0, filter1}}}{m_{1, filter1 - m_{0, filter1} + m_{1, filter2} - m_{0, filter2}}$$
(1)

To evaluate energy consumption, the pressure drop between the inlet and the overflow of the HC was measured under variation of the feed flow rate (F001) without underflow. The geometrical dimensions of the used HC, based on an empirical geometrical optimization study [26], are shown in Figure 3.



**Figure 3.** Hydrocyclone dimensions on the basis of a previous empirical study [26] with variation of the inlet diameter, vortex-finder diameter, vortex-finder length, cone angle and underflow configurations.

### 3. Results and Discussion

Overall, a total number of 96 separation experiments (32 treatments, n = 3) were carried out on the test rig (Figure 2). PMMA and PS particles, both at  $\rho > 1000 \text{ kg/m}^3$ , left with the underflow due to centrifugal force (=standard mode). HDPE and PP particles ( $\rho < 1000 \text{ kg/m}^3$ ) did so with the overflow (=reverse mode operation). Table 1 and Figure 4 reveal the average separation efficiencies (n = 3) for all four investigated polymers, including minimum and maximum measured values.



**Figure 4.** Standard (**left**) and reverse mode (**right**) operations of the hydrocyclone (5 m<sup>3</sup>/h inlet flow rate) with selected polymers and variation of the underflow rate.

PMMA separation was found to be straightforward, revealing percentages of 98.42% and 98.54% at underflow rates of 10% and 20%, respectively. Particle properties of a relatively large density difference ( $\rho$ : 1200.6 kg/m<sup>3</sup>) to water ( $\rho$ : 1000 kg/m<sup>3</sup>) coupled with relatively large particle size (520 µm) allowed for optimal separation at a low underflow rate. Consequently, there is no need to increase the underflow rate, so maximum (100%) generation of purified liquid is possible. For PS, separation was found to be more challenging, due to minor density differences in relation to water and smaller particle size (Table 1 and Figure 4, left). With 0% underflow, only 3% separation efficiency can be reached. Separation efficiency continuously increased between 10% and 70% underflow rates and thereafter slightly increased at underflow rates of 70% to 90%, thereby revealing efficiencies of 90.36%, 92.34% and 93.85%, respectively.

HDPE particle separation (Figure 4, right) started at a 50% underflow rate (separation efficiency of 3.15%) and thereafter rapidly increased, reaching quantities of 99.12% separation efficiency at a 90% underflow rate. Consequently, at an underflow rate of 50%, almost 50% ( $2.5 \text{ m}^3/\text{h}$ ) of purified liquid can be generated, while all particles are concentrated in the 2.5 m<sup>3</sup>/h unpurified liquid (overflow of the hydrocyclone). Even greater efficiencies could be detected for PP particles due to greater differences in density (881.9 kg/m<sup>3</sup> compared with 949.6 kg/m<sup>3</sup> for HDPE). The hydrocyclone was found to be capable of retaining 99.35% of suspended particles at a 90% underflow rate. Furthermore, separation was induced at a 30% underflow rate, revealing a respective average efficiency of 7.16%.

Generally spoken, particles with a density of >1000 kg/m<sup>3</sup> are more likely to pass the primary vortex and be discharged into the underflow and particles with a density < 1000 kg/m<sup>3</sup> are more likely to enter the secondary vortex and be removed through the overflow [27]. Moreover, the greater the density difference between the particles and water, and the stronger the underflow rate are the higher the centrifugal force acting on the particles is, hence increasing overall separation efficiency.

Three other studies revealed promising results using shredded polymers. In a study by Yuan et al. [1], a composition of PET ( $\rho$ : 1300–1380 kg/m<sup>3</sup>) and PVC ( $\rho$ : 1380–1500 kg/m<sup>3</sup>) fragments that was mingled (mass ratio of 1:3.28) and grinded to a mean particle size of 0.75 mm was introduced into a CaCl<sub>2</sub> solution ( $\rho$ : 1280 kg/m<sup>3</sup>) and fed into the test rig in 2% concentrations (v/v). Test results revealed an efficiency above 80%, with purities of PVC and PET reaching 93.2% and 94.5%, respectively. The same polymers were also applied by Fu et al. [23]. The authors grinded PET ( $\rho$ : 1.220 kg/m<sup>3</sup>) and PVC bottles ( $\rho$ : 1310 kg/m<sup>3</sup>) to reach a mass ratio of 3.28:1 suspended in a CaCl<sub>2</sub> solution ( $\rho$ : 1280 kg/m<sup>3</sup>) and a 3% feeding regime. At a cone angel of  $12^{\circ}$ , the authors revealed efficiencies of up to 96.6% for PET in the overflow and 83.4% for PVC in the underflow. Carlson [24] presented a publication on the composition of HDPE ( $\rho$ : 970 kg/m<sup>3</sup>) and PP ( $\rho$ : 900 kg/m<sup>3</sup>) that was uniformly mixed according to a ratio of 1:1 and a particle size ranging from 239 to 1096  $\mu$ m. The authors used a light medium to reach efficiency of 78.8%. PP was found to have reduced from 1.0% in feed to 0.2% in the underflow. Separation in these publications was conducted using CaCl<sub>2</sub> as the liquid medium. The results presented here reveal high capacities for all four investigated plastics, hence allowing more functional applications in existing infrastructure (e.g., wastewater streams and others).

Energy consumption was evaluated using the characteristic pressure drop–volume flow rate diagram of the HC, which is displayed in Figure 5 according to an underflow rate of 0.



**Figure 5.** Pressure drop as a function of the feed volume flow rate (underflow rate = 0). Through multiplication of the pressure drop and the volume underflow rate of each set point, the energy consumption of the hydrocyclone can be estimated. For a typical setpoint (5 m<sup>3</sup>/h), the power consumption of the hydrocyclone is 66 W (simplified consideration neglecting individual coefficients of performance).

#### 4. Conclusions

HCs treating MPs face unfavorable conditions such as small particle sizes and low density differences. However, due to their beneficial characteristics, HCs bear the potential to fill the analytical gap for microplastic particles smaller than 200  $\mu$ m in large scale applications.

This study provides a database of the physicochemical properties and separation efficiencies of four commercially available, low-priced MPs. Particles were separated either through standard or reverse mode operations, thereby proofing the flexibility of a HC system in separating fractions with different density characteristics. As expected, higher density differences between target microplastic particles and water had to be compensated with underflow rates raised to a certain threshold. Removal of MPs for all four investigated plastics was shown to be efficient by using water as a liquid medium rather than CaCl<sub>2</sub> solution, thereby allowing for a level of functionality that is more applicable in practice. Another characteristic feature of this study is the usage of commercially available microplastic particles in contrast to the particles that were self-made by other authors. This leads to high potential for the harmonization of separation experiments in the microplastic sector.

In future studies, more plastic types and size-distributions must be investigated in depth to allow us to understand released primary and secondary microplastic debris more deeply as well as its removal from ecosystems through HC applications.

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