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RECOVERY OF MICROPLASTICS FROM A SYNTHETIC WATER

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The impact of microplastics (MPs) on aquatic organisms and human health is a growing concern. The purpose of this paper is to detect and identify laboratory-generated MPs in an artificial water. Recovery and repeatability of MPs was conducted with environmentally relevant LDPE, PET, PP, PS waste, for which the number, shape and color are known. The data show that the method applied for detection of MPs is proper for colored pellets, foils, fragments, and filaments and indicated a recovery about 83%. A recovery of 60±10% was achieved in the case of colorless foils from LDPE detected through the optical microscopy.

Keywords: plastic waste, microplastics, optical microscopy, FTIR, size, recovery, repeatability

1. Introduction

Celluloid was the first commercially successful plastic, which was invented by John Wesley Hyatt in 1869 [1]. The modern use of the term ‘plastic’ was first coined by Leo Hendrick in 1909. Today, plastic’ is a generic term used to describe a wide range of materials [2] applied in human life, from plastic bags and writing pens to pipes and electrical equipment; consequently, plastic production increases so as to meet the growing needs of the growing population [3]. As a whole, polyethylene (PE), polypropylene (PP), polystyrene (PS), and polyethylene terephthalate (PET) are compositions commonly used in the textile, packaging, automobile, electronic, and electrical industries [4]. For example, PP and PE are frequently applied in food packaging, cosmetic containers, and furniture. PET is generally used in beverage bottles, cosmetics, and textile containers. Globally, the amount of plastic production increased from 1.7 million tons in 1950 to 359 million tons in 2018, resulting in an increase of plastic waste [5,6]. It has been estimated that annually, 4.8 to 12.7 million tons of produced plastics end up in the marine environment [7].

Large chunks of plastic waste, called macroplastics, degrade in the environment into smaller and smaller pieces of plastic. The term ‘microplastic’

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(MPs) generally refers to any piece of plastic smaller than the size of 5 mm to 1 μm across the longest dimension [8,9]. The pieces of plastic smaller than 1 μm are considered nanoplastics (NPs). MPs can be classified as primary and secondary according to their actual source [10]. Primary MPs can be found in household items, personal hygiene products, facial cleansers, toothpaste, exfoliating creams, medicines [11], and anthropogenic activities such as compressed air blasting and 3D printing items [12,13]. It has been estimated that around 6% of liquid skin cleansers sold in the European Union, Switzerland, and Norway contain MPs, of which over 93% are polyethylene (PE) [1]. Due to the continuous fragmentation of plastic waste under the environmental factors, the generated MPs are frequently transported in the aquatic ecosystems through wastewater collection and treatment systems [14,15]. Secondary MPs result from the decomposition of plastic litter. The literature highlights large sources of these secondary MPs: plastic fishing gear, waste generated by ships or disposed of after recreational activities, organic waste, paints containing synthetic polymers, composting additives, fibers released from hygiene products, or synthetic textiles [4,16]. Wear and abrasion of synthetic textiles during use and drying have been identified as important pathways for microplastic fiber formation [17].

Microplastics have been detected in seawater [18], marine species and sediments from the coastal areas [19], sea salts [20], drinking water intended for human consumption [21], yogurt [22], air [23], recycling plastic facilities [24], and soil [25].

MPs has raised significant concerns relating to environmental and human health due to uptake of other toxic chemicals from aquatic environment such as polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbon (PAHs), antibiotics, and heavy metals [26,27,28] leading to serious diseases, including cancer [29].

The purpose of this study is to validate a detecting method of MPs, in triplicate, by adding a known number of MPs in an artificial water, similar with that from rivers.

2. Experimental parts

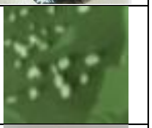
2.1. Materials

The plastic waste used for the method validation were: colorless and black foils of low-density polyethylene (LDPE) and PP, respectively, originating from food packaging, blue fragments of PET from drinking water bottles, white pellets of expanded polystyrene (PS) from plastic items packaging, ethylene-propylene (C2-C3) copolymer as fragments, which is frequently used in the automotive, electronics and the construction industry, and multicolor PET fibers resulted from textile. Selected plastic waste were manually cut into small pieces under 5 mm size dimension. To confirm that the chosen plastic waste fell within the dimensions of microplastics, they was separated into fractions ranging from 4000 to 32 µm. Before manually cut of plastic waste, their characterization by ATR-FTIR (Interspec 200-X Spectrophotometer, Interspectrum, Estonia) was done.

Table 1 shows the detailed characteristics of MPs investigated for further validation method from water matrix.

Table 1

Physical parameters of the MPs used for spiking of artificial water

MPs type	Color	Shape	Density (g/cm^3)	Visual image
Expanded PS	White	Pellets	0.0300	
PP	Black	Foils	0.9743	
C2-C3	White	Fragments	0.9805	
PET	Blue	Fragments	1.4501	
LDPE	Colorless	Foils	0.9714	
PET	Red, black, green, colorless, blue	Filaments	1.3575	

2.2. Sample preparation

To prepare artificial water in laboratory following chemical reagents p.a. were added to a Berzelius beaker: NaCl 24.53 g/L, $MgCl_2x6H_2O$ 5.20 g/L, Na_2SO_4 4.09 g/L, $CaCl_2$ 1.16 g/L, KCl 0.695 g/L, $NaHCO_3$ 0.201 g/L, KBr 0.101 g/L, H_3BO_3 0.027 g/L. Anhydrous $CaCl_2$, $MgCl_2x6H_2O$, NaCl, $NaHCO_3$ were purchased from Chimreactiv SRL, Bucharest; anhydrous Na_2SO_4 was acquired from Chemical Company, Iasi; KCl was acquired from Basic-Poch SA, Sowińskiego, Poland; KBr was from Sharlab SL., Sentmenat Spain; and H_3BO_3 was from Merck KGaA, Darmstadt, Germany.

The distilled water was added till 1000 mL. The resulted solution was homogenized into an ultrasonic bath, then, it was filtered under vacuum through a glass filter paper with 1.2 µm pore sizes.

The synthetic water prepared in the laboratory was characterized by an electrical conductivity of 41.83 ± 0.70 mS/cm, a total dissolved solids content of 24.92 ± 0.08 g/L (multiparameter type CONSORT C862, Turnhout, Belgium), a pH of 7.07 ± 0.04 (multiparameter CONSORT C831, Turnhout, Belgium), a concentration of chlorine ions of 15.1 ± 0.02 g/L, and a salinity of 26.4.

The prepared artificial saline water was spiked with 82.3 mg of mixed MPs formed
from PS, LPDE, PET, PP, and C2-C3 copolymer having different morphologies. The solution was mixed into a Digital Heated Ultrasonic Cleaner and, then, filtered by using a cascade filtration configuration. The purpose to use artificial water comes from the large MPs widespread in aquatic environment and the major of plastics floating in water [30,31].

To avoid the occurring of MPs in laboratory, a few precautions have been taken during the processing of samples:

- the personnel wore clean white cotton laboratory coats and nitrile gloves
- the air circulation during laboratory was stopped
- the work surfaces were cleaned with 70% ethanol and rinsed with distilled water
- the distilled water was pre-filtered before use, by using glass filter papers, 47 mm diameter, 1.0 µm pore size ((Hahnemühle, Dassen, Germany)
- the negative controls (n = 3) are performed simultaneously with MPs processing to estimate the errors provided due to contamination.

2.3. Method validation

The approximately mixed 82.3 mg of MPs consisting of 10 fragments of PET, 10 pellets of expanded PS, 10 fragments of C2-C3 copolymer, 10 foils of LDPE, 10 foils of PP, and 65 filaments of PET was introduced in a Vibratory Sieve-Shaker „Analysette 3" tool equipped with stainless steel sieves with pore dimensions of 4 mm, 1 mm, 125 µm, 90 µm, and 32 µm. The MPs fractions separation, in triplicate, was done at an amplitude of 1 mm for 5 minutes. The MPs with known sizes were immersed into a volume of 1000 mL artificial water and dispersed on the electrical plate provided with magnet, under 50 rpm. A cascade of successive stainless-steel mesh sieves (Test sieves, ISO 3310-1, Fritsch GmbH, Idar-Oberstein, Germany) with sizes of 4 mm, 1 mm, 125 µm, 90 µm, and 32 µm was used to separate the MPs. The content of each sieve was rinsed carefully with distilled water and transferred to a vacuum pump assuring a maxim pressure of 2.5 bar (KNF, Type Laboport N96) equipped with a glass microfiber filter, GF/A with pore sizes of 1.6 µm. After washing each filter containing MPs with distilled water, it was introduced into a glass Petri dish with lid (Φ 10 cm) and kept for drying at room temperature for further detection. An optical microscope OLYMPUS BX 51 M, Olympus Corporation, Tokio, Japan) with Olympus Stream Essential 1.9.3

Software was used to examinate the MPs in light polarized light, for morphology, dimension and color characteristics.

The recovery was determined by the ratio of the retrieved MPs as number to those introduced in artificial water, while repeatability, as RSD%, was measured by the ratio of the standard deviation to the mean of the recovered MPs.

3. Results

3.1. ATR-FTIR analysis

Fig. 1 shows the ATR-FTIR spectra performed for the plastic waste subjected for MPs detection.

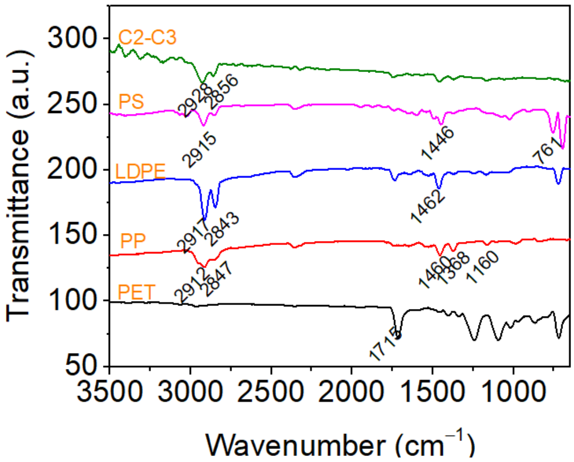


Fig. 1. ATR-FTIR spectra of selected plastic waste

The LDPE waste is characterized by two absorption peaks at 2917 cm^{-1} and 2843 cm^{-1} which correspond to the valence vibration of the asymmetric and symmetric CH_2 group, respectively. The peak at 1462 cm^{-1} is attributed to the CH_2 valence group. PET is characterized by an intense absorption band located at the frequency of 1715 cm^{-1}, which corresponds to the carbonyl group specific to polyester. PP shows a broad peak with the maximum at 2912 cm^{-1} (valence C-H) and absorption bands at 1460 cm^{-1} (CH_2), 1368 cm^{-1} (CH_3) and 1160 (CH_3 rocking, C-C of valence) [32]. The PS spectrum highlights the peaks in the range 2855-3067 cm^{-1} which are attributed to the valence vibration of the C-H group in the benzene nucleus and another peak at 1446 cm^{-1} attributed to the valence vibration of the aromatic ring. PS and C2-C3 copolymer were also identified based on DSC (differential scanning calorimetry) analysis which is not presented in this study.

FT-IR spectroscopy is a common analytical method used for the identification of MPs [33].

3.2. Characterization of MPs

The weight % distribution of selected MPs on the sieve sizes is illustrated in Fig. 2.

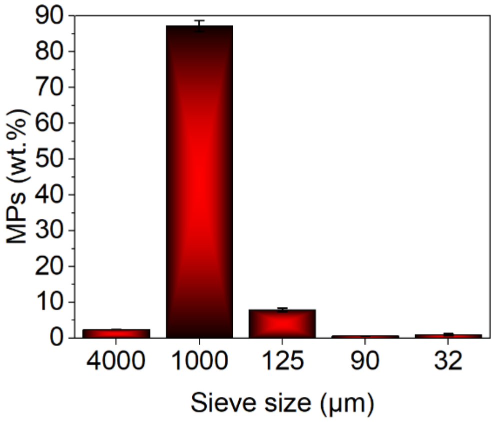


Fig. 2. Weight percent of MPs distribution on the test sieves

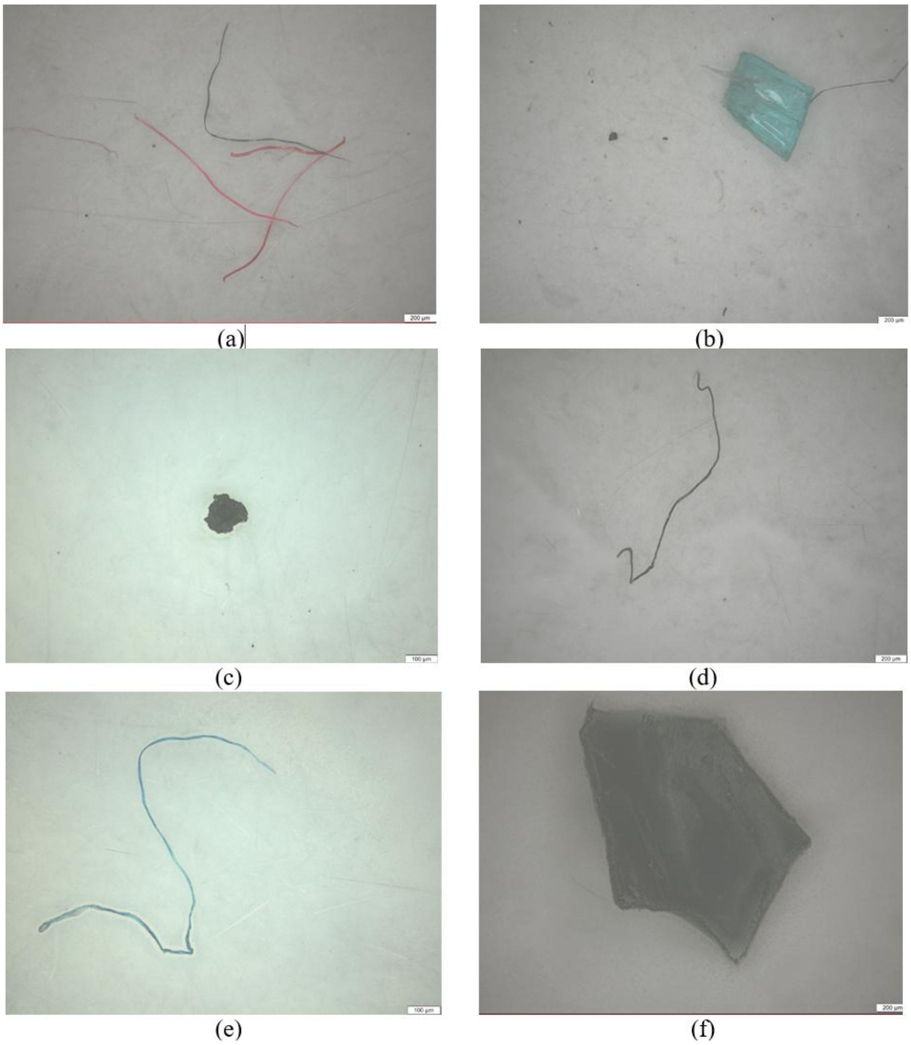
According to Fig. 2, it was observed that the most weight of MPs was widespread on the sieve with 1 mm size (87.2±1.5%), followed by that of 125 µm (7.89±0.5%). However, this behavior was not meet in the case of MPs collected from real water, when the abundance of MPs increases with the decreasing of the sieve size. The use of different mesh size enables a more thorough comprehension of the distribution and impact of MPs in various environments and their potential for ingestion by aquatic organisms. The majority of MPs ingested by marine invertebrates and fish are often smaller than 300 µm in diameter [34].

Fig. 3 illustrates some images of MPs taken from the optical microscopy examination.

Optical microscopy is the best method employed for the immediate identification of MPs. This method allows the detection of MPs with different morphologies, color and size. However, it leads to the misidentification and underestimation of MPs arising from the colorless and unclear shape of MPs [35].

The recovery % calculated for each MP type, in triplicate, is shown in Table 2.

MPs show distinct morphologies, such as pellets, fragments, foil, and filaments, and colors. The MPs fabricated in laboratory from known plastic waste exhibit predominantly filament shape, as the most MPs analyzed in literature, and large chromatic colors (Fig. 4).



(a) (b) (c) (d) (e) (f)

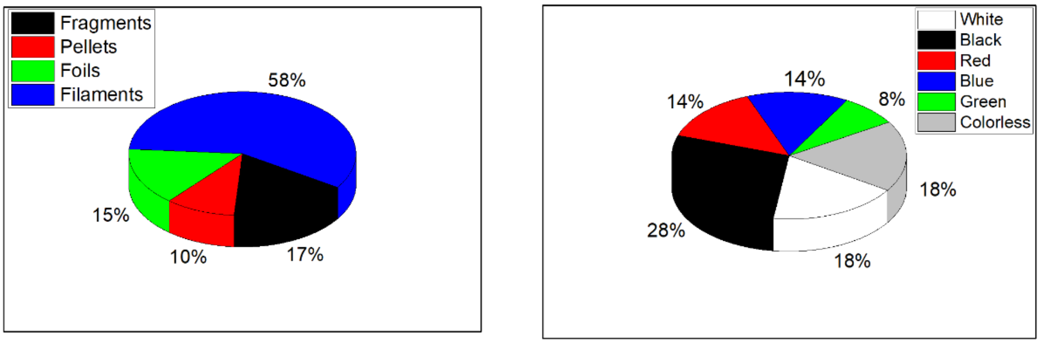
Fig. 3. Optical microscopy images of MPs collected on: (a) 32 µm, magnification 50x; (b) 90 µm, magnification 50x; (c), (d) 125 µm, magnification 100x and 50x; (e) 1000 µm, magnification 100x; (f) 4000 µm, magnification 50x

Table 2

Recovery for detection of MPs from synthetic water

	No. of MPs introduced in synthetic water	No. of MPs recovered on each test sieve					Recovery, %
		4000 µm	1000 µm	125 µm	90 µm	32 µm	
Test no. 1	10 fragments of PET			2	3	4	90
	10 pellets of expanded PS	6	4				100
	10 fragments of C2-C3 copolymer	4		2		2	80
	10 foils of LDPE			2	3		50

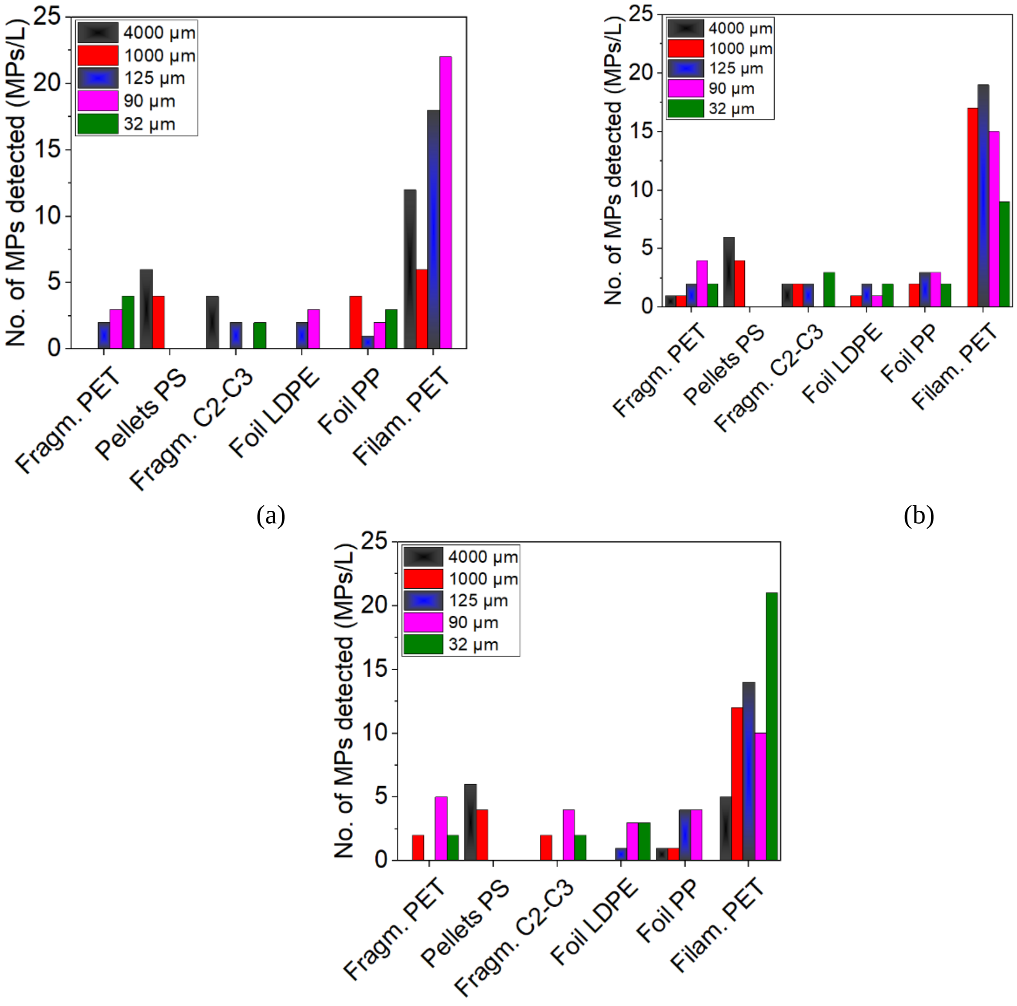
	10 foils of PP		4	1	2	3	100
	65 filaments of PET	12	6	18	22		89.2
Test no. 2	10 fragments of PET	1	1	2	4	2	100
	10 pellets of expanded PS	6	4				100
	10 fragments of C2-C3 copolymer	2	2	2		3	90
	10 foils of LDPE		1	2	1	2	60
	10 foils of PP		2	3	3	2	100
	65 filaments of PET		17	19	15	9	92.3
Test no. 3	10 fragments of PET		2		5	2	90
	10 pellets of expanded PS	6	4				100
	10 fragments of C2-C3 copolymer		2		4	2	80
	10 foils of LDPE			1	3	3	70
	10 foils of PP	1	1	4	4		100
	65 filaments of PET	5	12	14	10	21	95.5



(a) (b)

Fig. 4. Characteristics of MPs detected through optical microscopy for the first test. (a) Morphology; (b) Color

Fig. 5 shows the number of MPs detected for each plastic waste type after the separation through a cascade of sieves.



(a) (b)

(c)

Fig. 5. Number of MPs detected by optical microscopy based on the test sieve size. (a) Test no. 1; (b) Test no. 2; (c) Test no. 3

For three replicates, the number of MPs detected on each sieve size differs (Fig. 5). This depends on the manually cut of plastic waste. It can be observed that the pellets of PS show the same abundance on 4000 µm and 1000 µm.

Fig. 6 shows the parameters for validation of this method in laboratory.

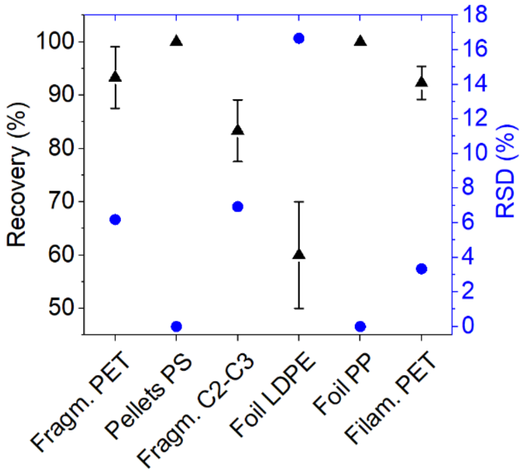


Fig. 6. Recovery and repeatability of method for detection of MPs prepared in artificial water ▲ Recovery, %, ● Repeatability, %

The validation results showed an average recovery of 60%±10% for the LDPE, 83%±5.7% for C2-C3 copolymer, 92.3%±3% for filaments of PET, 93.3%±5.7% for fragments of PET, and 100% for pellets of PS and foils of PP (Fig. 6). This method was found to have a good repeatability (RSD <7 %) for all shape of MPs with exception of colorless LDPE foil. Due to the colorless, LDPE MPs were more difficult to detect, which explains the lower repeatability. From Fig. 6 it was observed that the most difficult to identify is the colorless foils of LDPE.

Another limitation of this procedure is that the artificial water did not contain suspensions and organic matter, which make the detection difficult. This might lead to overestimation of microplastic abundance in real environment.

4. Conclusions

Microplastics monitoring data were achieved by investigating the recovery and repeatability of known MPs derived from plastic waste immersed in artificial water. A mixed of fragments, pellets, foils, and filaments from known LDPE, PS, PP, PET, and C2-C3 copolymer was used for recovery from synthetic water. Isolated particles from sieves with sizes of 4000 µm, 1000 µm, 125 µm, 90 µm, and 32 µm were characterized by using optical microscopy based on abundance, shape and color.

The overall recovery of the applied method was 60%±10% for colorless LDPE and 83.3%±5.7% for fragments of C2-C3 copolymer, and 100% for PS pellets and black foils of PP. The repeatability % was < 7% for fragments of PET and C2-C3 copolymer, foils of PP, pellets of PS, and filaments of PET. This methodology could be a preliminary trial to be applied for MPs detected from real environments.

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