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Quantification of Microplastics in Urban Compost-Amended Farmland Soil Using an Elutriation Device

Luigi Paolo D'Acqui [1], Sara Di Lonardo [1,2,*], Martina Grattacaso [1], Alessandra Bonetti [1] and Ottorino-Luca Pantani [3]

[1] Research Institute on Terrestrial Ecosystems (IRET), National Research Council (CNR), Via Madonna del Piano 10, 50019 Sesto Fiorentino, Italy; luigipaolo.dacqui@cnr.it (L.P.D.); martina.grattacaso@iret.cnr.it (M.G.); alessandra.bonetti@cnr.it (A.B.)
[2] National Biodiversity Future Center (NBFC), Piazza Marina 61, 90133 Palermo, Italy
[3] Dipartimento di Scienze e Tecnologie Agrarie, Alimentari, Ambientali e Forestali, Piazzale delle Cascine 18, 50144 Firenze, Italy; ottorino-luca.pantani@unifi.it
* Correspondence: sara.dilonardo@cnr.it

Abstract

Microplastics (MPs) present in farmland soils, where urban compost has been distributed since 2005, were extracted using a device based on elutriation, a method developed for marine sediments but not yet used in soil. Since (i) fine earth (diameter < 2 mm) is the standard fraction used for soil analysis and (ii) the size of MPs contained in urban compost may exceed that value, MP were recovered from both the entire soil and fine earth. The recovered MPs pieces were weighed, counted, and characterized using FTIR photoacoustic spectroscopy (FTIR-PAS). Both the mass and number of recovered MPs pieces (>34 µm) were comparable to those reported in the literature for soils. Polystyrene, polyethylene, and polypropylene are the primary polymers. Nevertheless, some issues were highlighted: (i) the importance of sampling the soil by volume, and (ii) the need of analyzing the entire soil sample rather than just the fraction below 2 mm, commonly used in soil analysis; (iii) the necessity of breaking up (i.e., by ultrasonication and/or dispersion) soil aggregates that may withstand the elutriation process.

Keywords: agriculture; urban compost; FTIR-PAS; elutriation; pollution

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1. Introduction

Human activities, such as industry, transport, and agriculture, release enormous quantities of CO_2 into the atmosphere, contributing to the greenhouse effect and climate change. One possible action to counter this phenomenon is to safeguard the soil organic carbon (OC) content [1] by increasing the content of organic substances in the top 40 cm of the soil [2]. To achieve this goal, participants at the United Nations' 21st Climate Change Conference "COP21" in 2015 proposed different agronomic practices, in particular the use of compost, which contributes to the increase of soil organic matter owing to its stable OC content. Thus, recycling organic waste through composting or fermentation and subsequent application to agricultural land is an environmentally sound practice for returning nutrients, trace elements, and humus to the soil [3,4]. In Europe and worldwide, regulations regarding compost purity, particularly concerning plastic contamination, are evolving. In Europe, the Fertilising Product Regulation (EU 2019/1009) establishes harmonized standards for compost and other fertilizing products across the European Community. Despite these standards, challenges persist regarding plastic contamination in compost.

However, most household and municipal/urban solid waste compost contains materials such as plastics [5,6], which can be a source of undesirable constituents in soil [7–10]. Sieving and sifting procedures can significantly reduce, but never completely eliminate, the presence of these contaminants. Some studies have focused on identifying and quantifying MPs in compost and compost-amended soil, rather than evaluating mitigation strategies. However, some recent research and pilot-scale initiatives have begun exploring potential innovations aimed at reducing plastic contamination in compost [11].

Microplastics have different compositions and properties. Most MPs have hydrophobic surfaces, which may alter their water-holding capacity, transport, and soil availability [12,13]. Due to their high adsorption capacity, MPs may alter the adsorption-desorption behaviors and availability of nutrients or contaminants in organic forms, thereby changing soil fertility and contaminant toxicity [14–16]. In particular, two soil characteristics, soil porosity and organic matter content, can significantly influence MPs' entrapment and mobility in the soil. Higher porosity can facilitate the movement and redistribution of MPs, especially the smallest MPs. Conversely, low-porosity, compacted, and fine-textured soils may trap MPs more effectively [17–20]. Organic matter promotes the formation of soil aggregates and provides binding sites for MPs, thereby reducing their mobility. A high organic matter content affects water retention and microbial activity, influencing MPs degradation. Conversely, MPs can bind with soil organic matter, making them less accessible to decomposers and slowing the breakdown of organic material. A 10% reduction in the decomposition rate of SOM was observed at an MP concentration of 0.5%, further compromising nutrient cycling and soil fertility. These interactions also slow the mineralization of carbon and nitrogen, which are essential processes for maintaining soil organic carbon levels and overall soil fertility. A 5–10% decrease in soil organic carbon due to disrupted SOM decomposition has been documented [21]. Together, these factors determine how MPs are retained, transported, or potentially biodegraded in the soil environment, thereby affecting their ecological impact and the effectiveness of extraction methods [22].

Microplastics have been found everywhere, from high-mountain lakes to deep-sea sediments and in temperate and tropical aquatic systems [23,24]. However, MPs' pollution in soil is a relatively new issue that cannot be overlooked. Some authors have hypothesized that MPs contamination on land might be 4–23-fold larger than that in the ocean [25], while others have suggested that agricultural soils alone might store more MPs than oceanic basins [26].

For this complex matter, sampling techniques, extraction methods, analytical procedures, and even units of measurement for soil samples have not yet been standardized.

Separation, filtration, sieving, and sorting of MPs are the four main steps that can be distinguished during the laboratory processing of samples [27]. To separate MPs, flotation methods similar to those proposed by [8] and later by [28] can be used. These methods use the density of materials to differentiate plastic pieces from natural particles found in the soil. This includes the analytical steps required to accelerate the separation of particles, such as the use of saturated salt solutions and centrifugation [28,29]. Plastic pieces are then separated from the supernatant obtained from density separation by passing the solution containing the plastic pieces over a filter, usually aided by vacuum [30]. Moreover, MPs can be separated from samples by sieving and sorting. Materials retained by the sieve can be collected and sorted, while those that pass through are typically discarded. The use of sieves with different mesh sizes allows for the sorting of MPs according to their size [31]. All the procedures described above are costly in terms of time and materials when the sample sizes are large. Overall, MPs represented a very small proportion of the sample. Consequently, any means of concentrating them can be beneficial in terms of time and resource savings.

Among the environmental matrices in which MPs were found, sediments share some aspects with soils, such as the presence of mineral and organic solid phases. They usually lack a relevant gas phase; thus, they may have different chemical-physical interactions with MPs due to factors such as the aggregation state of the particles, redox potential, pH, and particle size distribution. Nevertheless, methods for MPs characterization in sediments can potentially be adjusted for soils, but their applicability still needs to be evaluated [32,33]. In particular, the method pioneered by Claessens et al. [31] is based on the principle of elutriation, which separates lighter and smaller particles from others using an upward stream of air and water.

In other words, the use of a device similar to that built by [31] appears to be a viable method for removing predominant soil mineral particles and concentrating MPs in a smaller volume for further processing.

After the separation/extraction step, visual inspection is a common method for identifying MPs based on type, shape, degradation stage, or color as sorting criteria. Chemical and physical characteristics (e.g., specific density) can be used to complete the visual inspection. Nevertheless, the high number of papers on current methods for MPs extraction and identification in soils has several limitations that impact the accuracy, efficiency, and comparability of the results. Among the principal drawbacks are the heterogeneity of the soil matrix, sample preparation and extraction efficiency, MPs size detection limits, contamination, accuracy in the identification of MPs, quantification limits, and lack of standardized protocols [34,35].

The chemical composition and mass of MPs can be determined using pyrolysis coupled with gas chromatography and mass spectrometry (Py/GC/MS) [27,36–38] or thermal extraction desorption gas chromatography (TED-GC, [37]). Both methods allow the chemical identification of plastic polymers and the determination of their molecular weight; however, they destroy the sample [39]. In contrast, spectroscopic methods, such as Fourier Transform InfraRed (FTIR) and Raman spectroscopy, enable the evaluation of the size and number of pieces, as well as polymer identification, without destroying MP pieces. Spectroscopic methods identify MPs polymers through their molecular vibrations [40]; however, to date, they have not been extensively tested in soil. Considering that Wang et al. [41], in a review of analytical methods to extract MPs from soil, suggested focusing on realistic environmental concentrations and surrogates that mimic the types, shapes, and sizes of MPs/NPs in natural environments, we selected an agricultural soil amended with municipal solid waste compost in an inland hilly area of Italy. Solid compost has been applied in large quantities since 2005. This study aims to expand the knowledge on MPs extraction by applying a method specifically developed for the separation of MPs in marine sediments to soil samples [31]. The suitability of the method for soil was evaluated by (i) analyzing the material coming out of the device and the material left within it and (ii) characterizing the resulting MPs using FTIR photoacoustic spectroscopy (FTIR-PAS). The points listed above were necessary since soils and sediments, albeit being similar mineral-organic matrices, may have different chemical-physical interactions with MPs, such as aggregation state, redox potential, pH, and particle size distribution.

The standard operating procedures for soil chemical-physical and biological analyses are usually performed on fractions less than 2 mm; thus, the coarser material (skeleton) is either discarded or analyzed separately. However, it must be considered that compost may be a source of environmental plastics of various sizes (i.e., MPs; size less than 5 mm [9]) that must not be overlooked, even if limitations on MP content are currently present in the European Community [10]. In the present case, it was considered that MPs were present both below and above 2 mm. Therefore, two MPs-recovery modes were implemented: on the whole soil (Whole Soil mode, WSm) and on the fine earth (Fine Earth mode, FEm).

2. Materials and Methods

2.1. Study Site and Sampling

We were interested in studying a real case where urban compost—and therefore MPs—have been applied regularly in the past. Therefore, an agricultural farm located near Florence (Tuscany, Italy), where urban solid compost has been applied in large quantities since 2005, was selected. The compost was supplied by a waste treatment company. For privacy reasons, detailed information about the farm and the company cannot be disclosed. We focused on the upper 40 cm of soil, which normally undergoes tillage operations. Table 1 lists the sample abbreviations and their corresponding descriptions.

Table 1. Sample characterization: particle size distribution, % of particles greater than 34 μm, and organic carbon and nitrogen content of the soil samples from which MPs were extracted and characterized. Wheat (*Triticum aestivum* L., cv. Verna) was the crop on A and B fields, while sunflower (*Helianthus annuus* L.) was on C, D, and E. Compost from urban waste was applied since 2005.

Sample Name	Sampling Depth cm	Surface Area ha	Sand	Silt	Clay	>34 μm	OC_{Tot}	N_{Tot}	pH
			%						
A-20	0–20	~2	48.2	37.0	14.8	45.0	3.3	0.79	7.85
A-40	20–40	~2	47.2	36.8	16.0	44.8	3.2	0.79	7.60
B-20	0–20	~2	41.8	36.0	22.2	53.1	3.1	0.76	7.59
B-40	20–40	~2	51.0	30.1	18.9	42.3	3.5	0.74	7.84
C-20	0–20	~6	24.4	44.7	30.9	63.8	6.0	0.76	7.39
C-40	20–40	~6	29.4	47.2	23.4	46.6	6.9	0.73	7.62
D-20	0–20	~2	24.4	55.1	20.5	63.7	3.4	0.75	7.82
E-20	0–20	~2	42.7	38.0	19.3	48.2	4.0	0.79	7.42

In May 2018, five different areas of the farm, named A, B, C, D, and E, were sampled as follows: in each area, four sites located about ten meters apart from each other were excavated, and a composite sample was constituted (~2 kg). Where an anoxic grey layer was found (sites A, B, and C), two depths were sampled (0–20 and 20–40 cm); otherwise, only the top layer was sampled (sites D and E). At the time of sampling, the differentiation between the two horizons was thought to be important from a pedological point of view, due to the possibility of influencing MPs recovery.

2.2. Chemical and Physical Analysis of Soil

Soil was characterized on fine earth, i.e., on fractions passed through a 2 mm sieve and dried at room temperature.

Briefly, the particle size distribution was measured by densimetry in the range of 2–50 μm [42] and by wet sieving for sizes above 50 μm. Total C and N were measured by dry combustion using a NA 1500 CHNS Analyzer (Carlo Erba, Cornaredo, Italy). The procedure with 1M HCl [43] was used to remove carbonates to distinguish organic from inorganic C. The pH was measured at a 1.0:2.5 (*w*:*v*) ratio. Dry weight was measured on sample aliquots of fine earth, which were kept at 105 °C until a constant weight was attained. These aliquots were then discarded and not used for MPs extraction.

2.3. Microplastics Extraction and Characterization

The MPs' extraction protocol is summarized in Figure 1.

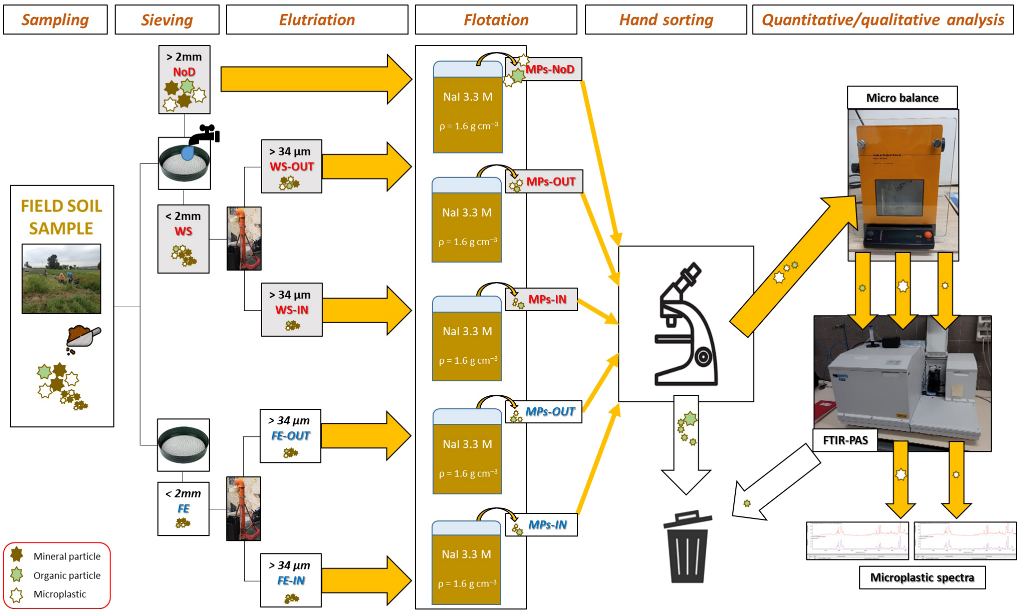

Figure 1. Soil sample treatment from the field to microplastic identification and quantification. The white and brown icons represent MP pieces and soil particles, respectively. Fem and WSm indicate Fine Earth and Whole Soil, respectively. The soil material (120–150 g) introduced into the elutriation device [31] was collected IN and OUT. NoD indicates a material greater than 2 mm that was not introduced into the device. The density of the suspension in the device was about 1.01 g cm^{-3}. Water flow (1 L min^{-1}) and air flow (14–15 L min^{-1}) were regulated to obtain a pulsating-dripping flow. The MPs obtained by flotation were hand-sorted under an optical microscope based on their shape, refractivity, and bright colors; pieces with evident vegetal structures were discarded. Some of the non-discarded pieces, mostly the black-brown ones, were discarded after inspecting their FTIR-PAS spectra, where sharp peaks typical of MPs polymers were absent (see Figure S3).

Each composite sample from the field (~2 kg) was split into two batches labeled WSm (Whole Soil mode) and FEm (Fine Earth mode). The FEm batch was dry sieved at 2 mm, as customarily done in soil analysis, while the WSm batch was used as such. For each sample (A, B, C, etc.), air-dried aliquots of about 100 g and 120 g were weighed and recorded for FEm and WSm, respectively. This step was performed to obtain approximately 100 g of fine earth in the two modes. Then, each FEm aliquot was dispersed in water and introduced into the device described below and processed within it, while each WSm aliquot was wet sieved at 2 mm. The coarser fraction (skeleton and MPs, also called No Device material, NoD) was stored for subsequent MPs separation by flotation, and the finer fraction was recovered as a suspension/sludge and then processed in the device.

Elutriation and flotation. A device was built to extract MPs from soil samples by adapting the method pioneered by Claessens et al. [31] for marine sediments. It was based on the principle of elutriation, a process that separates lighter and smaller particles from other particles using an upward stream of air and water. A PVC pipe (105 cm long with an inner diameter of 12 cm) was fitted with a 34 µm mesh screen at the bottom (The Wire Company, London, UK), a value close to that used by Claessens et al. [31], and supported by a rim just over the air stone (Figure S1). An upward water flow was then created by forcing water from the bottom of the device, where aeration was provided through an air stone to allow the separation of plastic from soil particles. The combination of water/air flow separated the lighter particles—including MPs—from the heavier soil particles in each soil sample, and the rising water transported them to the top of the device, where they eventually flowed over the edge of the PVC tube and were retained on an external 34 µm sieve. The upper part of the device was protected by a cover (aluminum foil) to prevent contamination of each sample by airborne particles or fibers. Water flow (1 L min^{-1}) and air flow (14–15 L min^{-1}) were regulated to obtain a pulsating-dripping flow out of the device (Figure S1). The process was continued until almost clear water dripped out of the device (about 45–60 min). The material—soil and MPs—collected on both internal and external 34 µm sieves and on the 2 mm sieve—named IN, OUT, and NoD—separately underwent a flotation procedure in NaI solution (3.3 M, d = 1.6 g cm^{-3}) as follows: each sample was transferred to a glass centrifuge tube, 40 mL of a NaI solution was added, and the mixture was centrifuged for 20 min at 103 g. After centrifugation, the top layer containing MPs was vacuum-filtered over a paper filter (8 µm membrane Whatman®, grade 40; Merck KGaA, Darmstadt, Germany). The flotation-centrifugation-filtration procedure was repeated three times for the same sample. To prevent contamination, each filter was placed in a closed glass Petri dish and oven-dried at 30 °C for three days. Finally, the material (MPs and biological debris) on each filter was removed and stored in closed glass vessels for further analysis.

Microscope separation, piece counting, and mass quantification. Microplastic and vegetable pieces obtained from flotation were observed using an optical dissection microscope (Bi 6X, Officine Galileo, Milan, Italy). Unequivocal vegetable pieces were removed, and the resulting pieces were subjected to FTIR-PAS analysis.

Identification using FTIR-PAS spectra. The MPs pieces obtained from the microscope separation were analyzed using an FTIR Spectrum 3 spectrometer (Perkin Elmer, Monza, Italy) equipped with an MTEC 300 photoacoustic detector (MTEC, Ames, IA, USA). A spectrum (256 scans) was acquired for each MP piece, without any further manipulation. During spectral acquisition, the sample was purged with He (1 L min^{-1}) to reduce IR absorption interference from air $CO_{2,}$ and H_2O [44]. Scans were conducted in the wavenumber range from 4000 to 400 cm^{-1} at a resolution of 8 cm^{-1}. A reference spectrum, obtained using black carbon, which absorbs throughout the range of IR frequencies, was subtracted as the background from each sample spectrum. Spectrum 10 software (Thermo Fisher Scientific, Waltham, MA, USA) was used to compare the spectra recorded for soil MPs against (i) those of plastic polymers contained in the library and (ii) those purposely acquired under the same operative conditions on pristine plastic materials, such as those listed in Table 2. In addition to the most common plastic polymers, the library also contains natural materials such as wood and natural fibers [45].

Table 2. Microplastics identified by FTIR-PAS: acronyms used in this work.

Acronym	Name
ABS	acrylonitrile butadiene styrene
AHR	aromatic hydrocarburic resin
AR	alkydic resin
CE	cellophane
EPM	polyethylenpropylene
PE	polyethylene
PEPD	polyethylene propylene diene
PET	polyethylene terephthalate
PEVC	polyethylene-vinyl chloride
PP	polypropylene
PS	polystyrene
PSVC	polystyrene vinyliden chloride
PVC	polyvinyl chloride
PVTB	polyvinyl toluene butadiene

The spectra were also visually inspected to reveal false attributions and detect non-plastic pieces (see Figure S3). After this inspection, non-plastic pieces were discarded, and the number of MP pieces and their total weight were recorded using a Sartorius 4503 microbalance ($\pm 10^{-6}$ g sensitivity).

2.4. Extraction Efficiency

A composite soil sample was used to estimate the extraction efficiency of the devices. It was obtained by mixing three subsamples collected from an adjacent farm field where MP-contaminated compost had never been spread. The uncontaminated soil mass (about 10 kg) was sieved at 2 mm.

Since the most common MPs found in the samples from A, B, C, D, and E were polyethylene (PE), polypropylene (PP), and polystyrene (PS), five uncontaminated soil aliquots (100 g each) were added with 5 PP + 5 PE + 5 PS pieces, a total of 15 pieces for each soil aliquot.

The added MP pieces were about 1 mm wide and were obtained by laser-cutting plastic bags (PE and PP) and styrofoam dishes (PS). Before cutting, the composition of the bags and dishes was checked using FTIR spectroscopy. Each 100 g aliquot of soil added with MP underwent drying/wetting cycles for one month. MP extraction was then performed as described for the contaminated samples.

2.5. Data Treatment

The masses of all soil samples were standardized to a constant weight, which was chosen to be 100 g of dry matter of fine earth. To estimate the mass and number of pieces retrievable in 1 ha, a soil depth of 40 cm and a bulk density of 1.3×10^3 kg m^{-3} were assumed.

The literature reports MPs quantification either as mass [46] or count [47,48], or dimension [49]. Here, MPs were quantified through mass, which was fitted using a linear model, and counts, which were fitted using a general linear model, family Poisson, with log as a model link function. The high dimensionality of the data—different types of MPs recovered in each soil sample—was reduced by Multiple Correspondence Analysis (MCA) [50,51], a technique commonly used to analyze surveys. Each row in the dataset represents a single soil sample, while each column is a categorical variable, i.e., one of the identified MPs. Each cell could take only two possible states: presence (Y), when the sample contained one or more pieces of that MP polymer, or absence (N) when that polymer was not found in the sample. Data analysis was performed using the statistical software R v4.3.1 [52] with suitable libraries [53–56]. The procedures for reproducible research were accomplished using Sweave [57].

3. Results

3.1. Chemical and Physical Characteristics of Soil

The soils under study were typical of the Italian inland hilly areas and were characterized by an alkaline pH due to the presence of $CaCO_3$. They were loams and silty-loams with remarkably high OC content, even exceptional, in samples C-20 and C-40 (Table 1). Regarding the particle size distribution, samples C-20, C-40, and D-20 contained less sand than the others.

3.2. Extraction Efficiency

All five extractions yielded 14 pieces OUT of the device and 1 piece IN it (general recovery rate of 93%).

3.3. Repartition of Soil Particles in and out of the Device

Figure 2 shows the percentage of material larger than 34 µm recovered through the extraction procedure, where obtained refers to the percentages recovered IN and OUT of the device, while expected refers to the percentage calculated by log-linear interpolation of the data obtained by Bouyoucos' method on a semi-continuous basis (Table 1 in text and Figure S2).

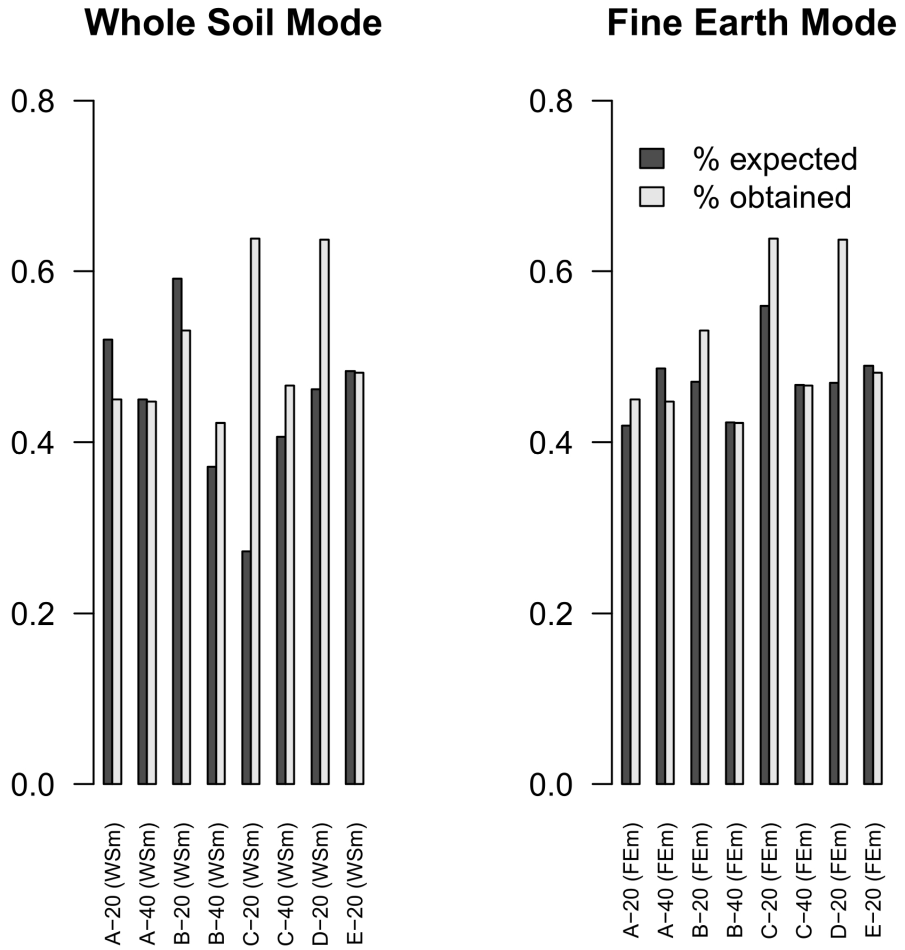

Figure 2. The recovery performance of the device shown in Figure S1 for soil particles larger than 34 µm. The expected percentage refers to the particle size distribution obtained by densimetry [42], while the obtained percentage refers to the sum of the soil material IN and OUT of the device. The data were standardized to 100 g of fine earth oven-dried at 105 °C.

In WSm, four samples, namely A-20, A-40, B-20, and E-20, showed either complete recovery or some loss of the material—soil and MPs—while A-40, B-40, C-40, and E-20 exhibited the same behavior in FEm. In other words, the procedure appeared to recover a greater amount of material than that initially inserted.

3.4. Quantification of MPs: Masses and Counts

Figure 3 shows the MPs masses recovered IN and OUT of the device; the NoD ones are obviously present in WSm only. The figures used to plot the graphs are listed in Table S1.

Considering the difference in IN−OUT within each sample, 12 out of 16 samples showed values greater than zero (Table 1), indicating that the device was ineffective in concentrating MPs. In addition, the largest amount of MPs was recovered IN the device for FEm, while for WSm, the largest aliquot of MPs was found in the samples not processed within the device (NoD; white bars in Figure 3). Although their repartition in the device was different, the total amount of MPs found for WSm and FEm was not statistically different at $p = 0.98$ (c) panel in Figure 3). The mean mass concentration of recovered MPs in all samples without considering the device (IN, OUT, NoD) was estimated to be 50.5 and 51 mg kg^{-1} for WSm and FEm, respectively (263 and 265 kg ha^{-1}). Regarding WSm, it must be noticed that about 87% of the MPs mass was recovered in the skeleton (particles > 2 mm; NoD).

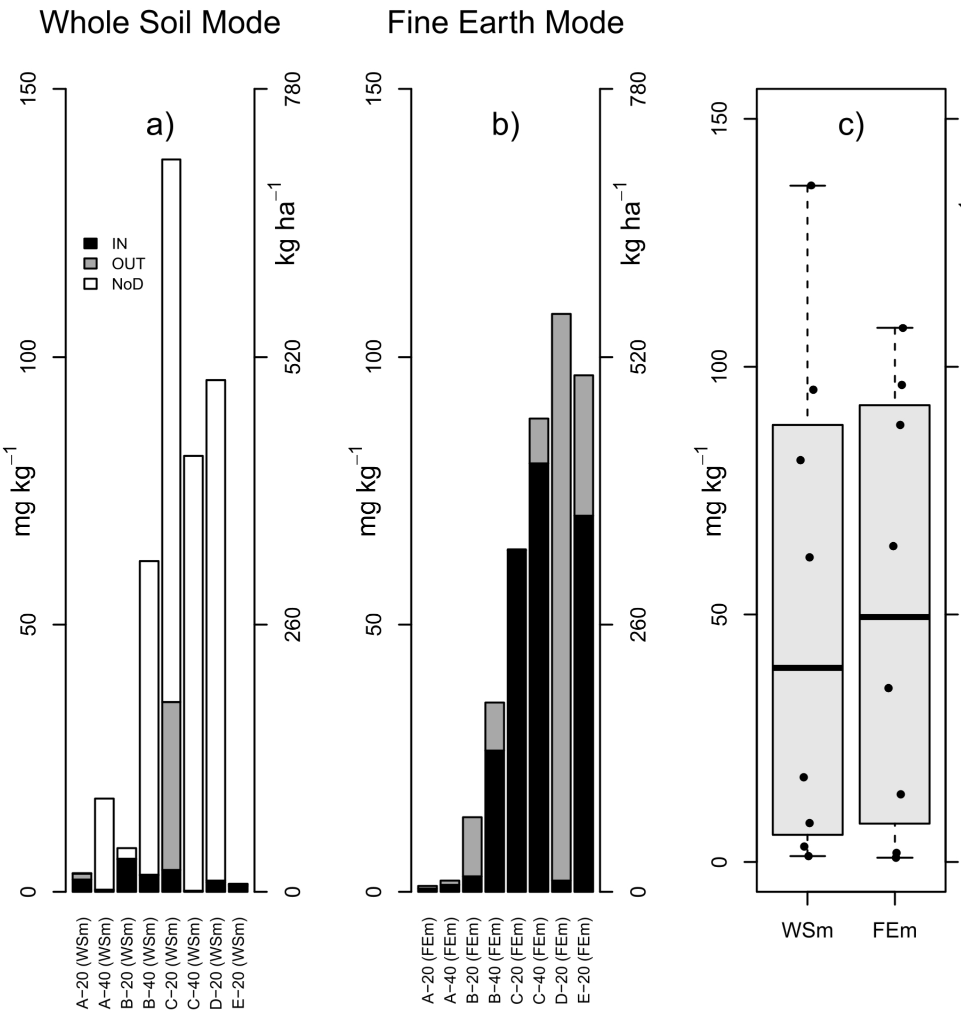

Figure 3. Concentration of total MPs recovered from the elutriation procedure. IN, OUT, and NoD refer to the protocol shown in Figure 1. Panel (**c**) shows the data from panels (**a**,**b**) as dots, which are summarized by boxplots, where the central line and whiskers indicate the median and the most extreme data point lying no more than 1.5 times the interquartile range from the box, respectively. The amounts were standardized to 100 g of fine earth oven-dried at 105 °C.

The number of recovered plastic pieces is illustrated in Figure 4.

In WSm and FEm, the mean number of total pieces per kg was 45 and 96.3, respectively, with a significant difference at $p < 10^{-3}$. Under visual inspection, most of the pieces separated by WSm were undoubtedly larger than those obtained by FEm: in fact, the mean weight of a single MP piece (total MP mass/nr of pieces) was 2.9 mg and 1.3 mg for WSm and FEm, respectively.

The number of plastic pieces grouped by type of polymer is shown in Figure 5. The total figures were 17.6 pieces kg^{-1} and 38.6 pieces kg^{-1} (91.520×10^3 pieces ha^{-1} and 200.720×10^3 pieces ha^{-1}) for WSm and FEm, respectively.

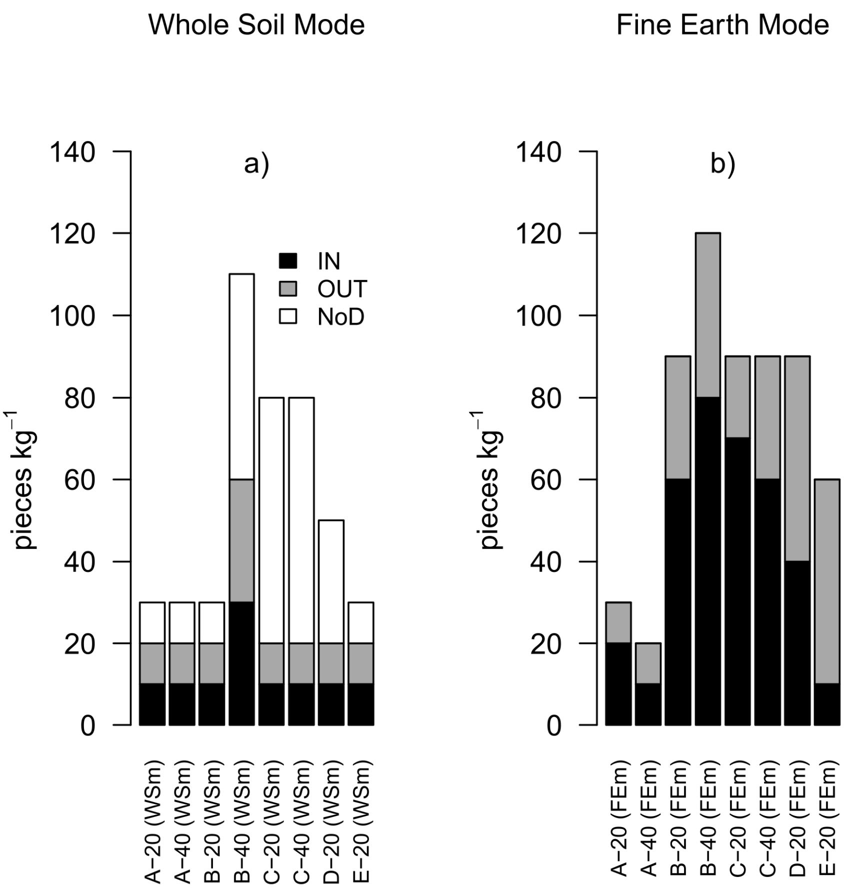

Figure 4. The number of MPs pieces recovered from the elutriation procedure using whole soil (**a**) and fine earth (**b**). IN, OUT, and NoD refer to the protocol shown in Figure 1.

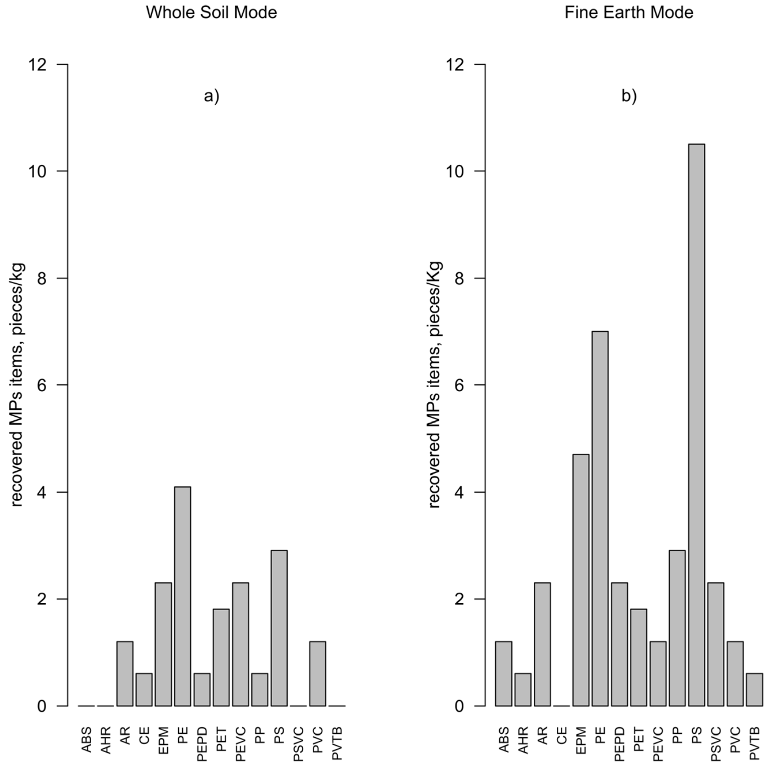

Figure 5. Number of MP pieces, sorted by type of polymer (see acronyms in Table 2), in whole soil (**a**) and fine earth (**b**). Counts are standardized to 100 g of fine earth oven-dried at 105 °C.

3.5. Microplastic Characterization

The recovered 14 MPs polymers are listed in Table 2.

The composition/variety of recovered MPs polymers (Figure 5) was, to some extent, more complex in FEm, where only CE was missing, while ABS, AHR, PSWC, and PVTB were absent in WSm.

Figure 6 shows the type of MP polymers found in each sample, with the y-axis representing the frequency of polymer occurrence. PS was widely distributed throughout the samples, while CE, PVTB, and AHR were recovered in only one sample. Samples from WSm showed: (i) a wider range of MPs types for sizes greater than 2 mm (NoD); (ii) almost no MPs OUT of the device; and (iii) a slightly lesser range in MPs types than FEm.

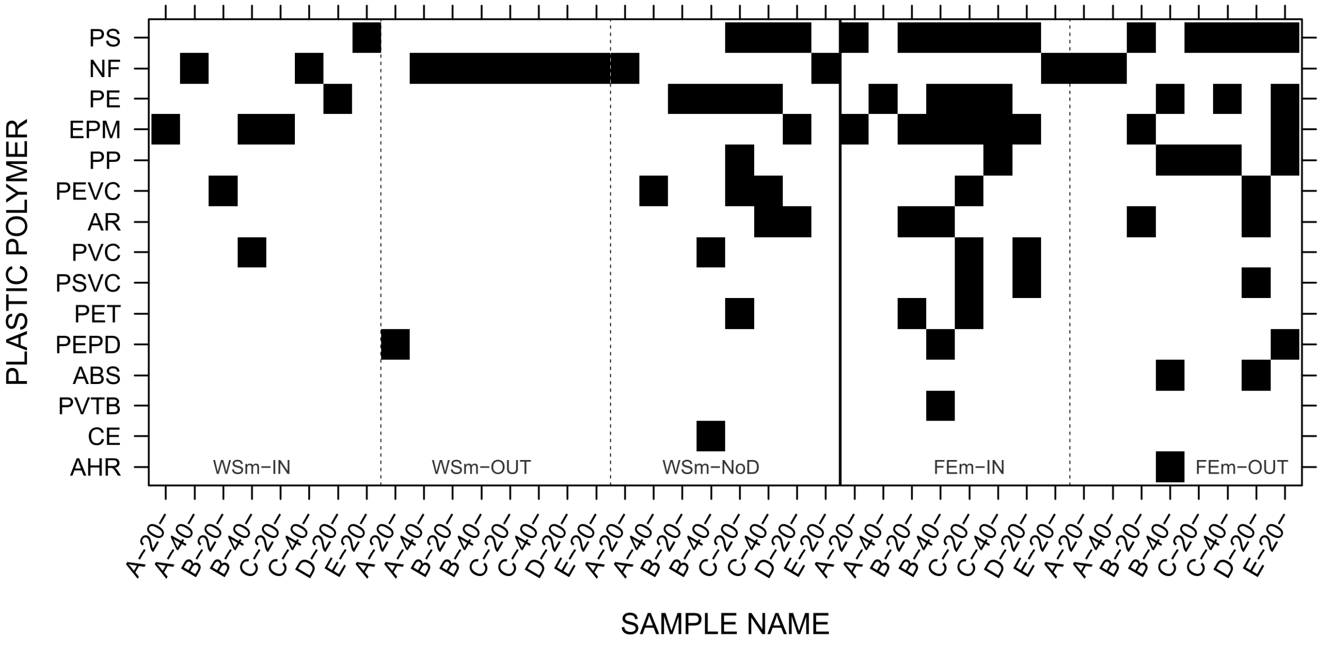

Figure 6. The type of MP polymers (see Table 2 for their acronyms) found in each sample for both the WSm and FEm modes and each device outlet (IN, OUT, NoD). Black squares indicate the presence of MP polymers. Note that there is also a category named NF, which stands for plastic Not Found.

Multiple Correspondence Analysis (MCA). The differences in MPs' composition across device compartments and operating modes were assessed using MCA. The results are presented in Figure 7. The first two dimensions explained about 37% of the total variability, a common value for MCA. The confidence ellipses did not significantly separate the assortment of MPs recovered from the three compartments of the device. In contrast, the two operating modes were separated, indicating a different assortment of MPs. The MCA confirmed the two empirical indications shown in Figure 6: (i) the device did not discriminate among the MPs types, and (ii) the variety of MPs changed between FEm and WSm.

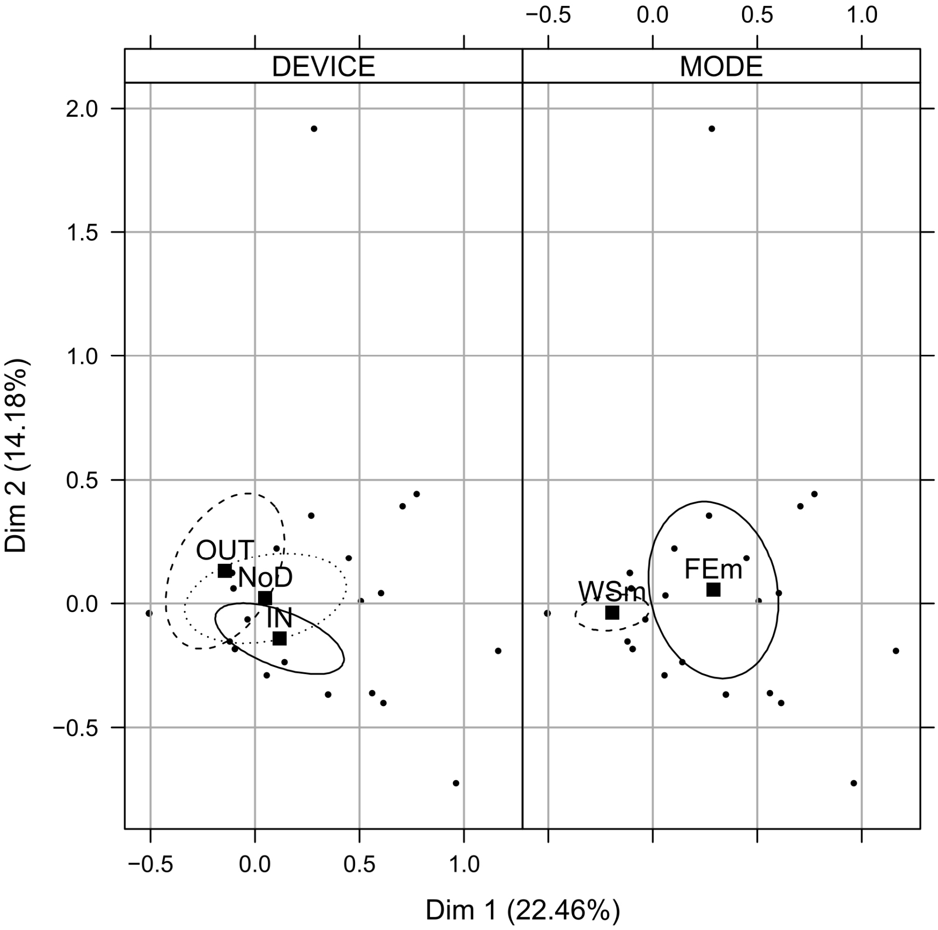

Figure 7. Plot of the first two dimensions resulting from the MCA analysis of MPs related to device compartments (**left panel**) and operating mode (**right panel**). The confidence level for the ellipses was 95%. IN, OUT, and NoD refer to where the MPs were recovered, while operating modes WSm and FEm refer to soil material greater than and less than 2 mm, respectively. For further details of the procedure, refer to Figure 1.

4. Discussion

Recently, interest in MPs contamination in the environment has increased, and the presence of MPs has been reported worldwide in many ecosystems [58]. The majority of works found in the recent literature have focused on MPs in marine environments, with only a few related to terrestrial agro-ecosystems [25,59]. In such environments, the use of urban compost, containing 14 to 895 MP pieces per kg [6], has become widespread, and the relative scarcity of studies on agro-ecosystems implies that no acknowledged methods for MPs extraction and analysis in soil are available. Therefore, we evaluated the performance of an apparatus previously used for MP recovery in aquatic sediments [60] on agricultural soil amended with solid urban compost since 2005.

The use of the device in soil showed an unexpected and, at first sight, inexplicable gain of solid material (soil + MPs), as shown in Figure 2. The device has one internal and one external sieve, which retains particles greater than 34 μm. In Figure 2, the gray bars indicate the amounts of material retained by the device, while the black bars indicate the amount greater than 34 μm as measured by densimetry. An ideal device would return identical values for both bars; a less ideal but more common device would have some losses, thus obtaining lower values for the gray bars. Unexpectedly, Figure 2 shows some gray bars higher than the corresponding black bars, indicating a gain in material. This unexpected behavior could be explained by the different particle size distributions in the samples introduced into the device compared to the distribution measured using the densimetric method. This method purposely destroys the soil aggregates both with the dispersed $Na_6(PO_3)_6$ and by applying kinetic energy via overnight agitation. In contrast, the samples used to separate the MPs were only wetted immediately before being introduced into the device, thus undergoing less physical and chemical destruction. In other words, the densimetric method yielded a particle size distribution shifted towards finer dimensions, while the distribution of the samples introduced into the device shifted towards coarser dimensions.

The extraction efficiencies for the PE, PP, and PS particles were 93%. Regarding the MP type left IN the device, three pieces were made of PS, while the other two were made of PP and PE. This relative abundance of PS pieces left in the device was most probably due to their mass, i.e., the thickness of the plastic sheets from where they were cut, being PS sheets thicker (2/10 mm) than PE and PP (1/10 mm).

Taking into account the MP pieces recovered by elutriation (Figure 3), it should be noted that an unexpectedly large number of pieces remained in the device compared to the values obtained for the extraction efficiency. One month of wetting/drying cycles was supposed to be able to re-create or get closer to the field conditions. Evidently, the laser-cut PE, PP, and PS pieces behaved differently in elutriation compared to the corresponding polymers that endured aging in the soil since 2005. The long time spent in the soil has certainly altered the chemical-physical properties of the MPs [61]; thus, they were possibly associated with either mineral particles or organo-mineral aggregates, and their wettability was modified by scratches and chemical degradation [62].

The association with mineral particles would increase the density of MP pieces, thus hampering their rising out of the device. Some preparative steps of the sample, such as dispersion/agitation in $Na_6(PO_3)_6$ solution or other physical treatment (ultrasonication) performed before elutriation, would break the soil aggregates, thus recasting the soil in a looser sediment-like material on which the device was already successfully tested. However, these treatments would result in some alteration of MPs, as has been reported for ultrasonication [28,63,64]. However, in the present case, the extent to which MPs remained trapped in heavier soil aggregates was unknown; a balance between alteration and the amount extracted must be achieved. The contrasting results obtained with the commonly accepted methodology for extraction efficiency—i.e., the use of fresh MPs, again underlined the need to operate close to the real situation, as suggested by Wang et al. [41].

The standard procedure for soil analyses considered only fine earth, that is, particles smaller than 2 mm. Since the upper size limit of MPs was established to be 5 mm [65], it was considered critical to select or ignore the skeleton. By omitting the coarser fraction larger than 2 mm, a potential loss of an important part of the MPs would have occurred. Unexpectedly, the mean number of total pieces was larger in the FEm mode than in the WSm mode (Figure 5). This behavior can be explained by the different amounts of initial WSm and FEm used in the sample preparation. According to the standard practice in soil analysis, the initial FEm soil batches (~1 kg) were dry-sieved to obtain the less-than-2 mm fraction (~700 g), from which 100 g were drawn for elutriation, while the procedure for WSm consisted of taking approximately 120 g directly from the initial batch (~1 kg) that had been wet-sieved, thus 100 g were elutriated. Therefore, the FEm sample was more homogeneous, and the larger initial amount increased the probability of finding more MP pieces in the material passing through the 2 mm sieve. The variability of the WSm samples could also explain the larger variability in the recovery performance compared to that observed in FEm (Figure 2). On the other hand, the presence of stones reduced the probability of finding MP pieces in the sample, since the stones' mass corresponded to a volume in which MPs could not be present. Furthermore, the unavoidable friction experienced during sieving could break the large aggregates into smaller ones, thus increasing the number of fine particles of soil and plastics passing below. The different initial sample sizes between FEm and WSm also affected the different assortments of polymers recovered in the two modes (Figures 5 and 7, right panel). This problem can be mitigated or solved by sampling the soil by volume, at least where a significant percentage of the skeleton is present. Specifically, skeleton-rich soils require larger sample volumes. Conversely, when the skeleton is negligible, smaller composite volumes should be sampled and analyzed as whole soil to capture a more accurate representation of the MP distribution, especially for meso- and macroplastic particles. This sampling scheme should result in a closer representation of MP across diverse soil contexts.

However, the order of MPs quantity, disregarding whether they were found either IN or OUT of the device, is comparable with those found in arable soils with other methods [58], taking into account mass [66] and counts [47,64,67]. As previously reported [47,64,67], the most abundant particles were PS, PE, EPM, and PP, probably because they are the most demanded plastics in the world [68].

5. Conclusions

Microplastics present in Italian agricultural soil, amended with urban compost since 2005, were extracted using a device based on elutriation, a principle widely used in sediment processing. The microplastics were then counted, weighed, and characterized using FTIR-PAS analysis.

This extraction approach, which has not been attempted before on soils, was generally successful since both the masses and numbers of plastic pieces recovered (greater than 34 µm) were of the same order of magnitude as those found in soils by many authors who have used other extraction procedures. Polystyrene, polyethylene, and polypropylene were the main polymers, i.e., the materials of which most household appliances are made.

Elutriation, the principle on which the present extraction device is based, offers a viable way to remove the predominant soil mineral particles and concentrate MPs in a smaller volume for further processing.

The different properties of fresh MP—those used in the efficiency tests—resulted in an erroneous estimation of the efficiency, as a higher-than-expected number of MPs was found in the device. Elutriation offers several advantages and some limitations compared to more traditional density separation or flotation methods for MP extraction from soils. Among the advantages of elutriation are (i) the gentle separation of particles based on size and density without chemicals, reducing the risk of damaging MP particles, and (ii) the removal of mineral soil particles, as the upward flow helps separate lighter MPs from heavier mineral particles, improving their recovery in heterogeneous soil matrices. However, elutriation has some limitations, including (i) incomplete release of MPs from aggregates since MPs trapped within water-stable aggregates may remain inaccessible and (ii) the need for specific equipment and time. In contrast, density separation and flotation typically rely on salt solutions to exploit the density differences between plastics and soil minerals. Although these methods are widely used and effective for a broad size range, they can introduce chemical contaminants or cause partial degradation of some polymers, and require various washing steps. Overall, elutriation provides a promising complementary or alternative approach, especially when combined with sample pre-treatments, to improve MPs recovery from soil. Our study demonstrates the feasibility of this approach but also underscores the need for further optimization.

Although the methodology yielded positive outcomes, it highlighted some relevant aspects: (i) the presence of water-stable soil aggregates incorporating MPs, thus hampering their extraction, and (ii) the inclusion of the skeleton that allowed the retrieval of large pieces of MPs otherwise discarded, but it also brought in stones that occupied a significant volume inaccessible to MPs.

While aspect (i) underlines the need to operate as close to the real situation as possible, some soil-specific problems could be either mitigated or solved: (ii) by introducing a pre-treatment to break or loosen the soil aggregates by chemical dispersion and/or ultrasonication, thus releasing the plastics they might contain; and (iii) by sampling the whole soil by volume rather than by weight.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/agronomy15071736/s1. Figure S1: Device, adapted from Claessens et al. [31], used to extract/concentrate microplastics. To avoid the collection of airborne particles, the upper part of the device, i.e., its outlet and 34 µm sieve, was sealed from the air with aluminum foil during the elutriation of each sample. Figure S2: Particle size distribution of sample A-20. Particles larger than 50 µm were separated by wet sieving, while smaller particles were measured using densimetry. Figure S3: Three examples of FTIR-PAS spectra of MP pieces recovered from some soil samples by elutriation and subsequent flotation in the NaI solution (3.3 M, d = 1.6 g cm^{-3}). Table S1: Plastic, 34 µm < diameter < 2 mm, dry matter at 105 °C, mg kg^{-1} of soil sieved at 2 mm. IN, OUT, and NoD refer to plastic in, out, and not introduced into the device, respectively.

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