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Microplastics Release from Conventional Plastics during Real Open Windrow Composting

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Abstract: The recycling of bio-waste plays an important role in a circular economy as it transforms bio-waste into a valuable resource (organic fertilizer). However, even separately collected bio-waste can contain some plastic waste, which is usually separated after composting and not before it. Primary studies have confirmed the degradation of plastic during composting, but the release of microplastics from them has not been studied. This article presents a quantification and comparison of the release of microplastics from commonly used plastics during green waste composting. Microplastics were identified by Nile red staining and examination under a fluorescent microscope. Plastic degradation was assessed by weight loss calculation, scanning electron microscope (SEM), and Fourier-transform infrared spectroscopy (FTIR) analysis. On average, 17 to 52 microplastics' are released from 5-by-5 cm pieces of conventional plastics during composting. The control polylactic acid sample showed the smallest amount of released microplastics: four particles on average. The number of released microplastics depended on the polymer type and thickness of the samples. The results of the current article can be further used for the prediction of microplastic generation and setting a limit on the plastic content in bio-waste

Keywords: microplastics; composting; bio-waste; degradation; FTIR; SEM; weight loss

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1. Introduction

Bio-waste constitutes the largest fraction of municipal solid waste (MSW), accounting for 34% in Europe [1]. The proper management of bio-waste plays an important role in a circular economy and in achieving sustainable development. Aerobic and/or anaerobic treatment of bio-waste reduces the load on MSW landfills and can create additional environmental and economic benefits by turning waste into organic fertilizer and a soil improver. However, a separate collection of bio-waste is needed to produce a safe, high-quality fertilizer with a minimum content of inclusions. The importance and necessity of a separate collection of bio-waste are emphasized in European legislative and policy documents. The revised Waste Framework Directive obliges all EU member states to collect bio-waste separately or ensure recycling at the source from the end of 2023 onwards. Furthermore, the Fertilizing Products Regulation, amended in 2019, states that fertilizer products can only contain compost from separately collected bio-waste.

Currently, aerobic treatment (composting) is the most commonly used technology for managing separately collected bio-waste in Europe, delivering more than 11.8 million tons of compost per year in European countries [1]. However, the pollution of compost with plastic, especially microplastics (polymer particles < 5 mm), is a growing concern. Several authors have noted a high concentration of microplastics in compost made of separately collected municipal bio-waste [2–6]. Potential sources of microplastics include food packaging and non-degradable plastic bags that come with bio-waste due to improper

home sorting [7–9]. According to Kranert [10], bio-waste can contain between 0.9% and 12% of misplaced materials in Germany. The pre-treatment stage of composting usually does not include the separation of plastic waste, and this waste, together with bio-waste, enters the composting conditions. Screening ready-made compost can significantly reduce plastic content but cannot separate microplastics smaller than the pore size of the sieve. In addition, microplastics of <2 mm are not covered by the Fertilizing Products Regulation, and there is no established maximum permissible concentration, so their number is not monitored.

During the composting process, plastics can undergo mechanical, thermal, photooxidative, and biological degradation, which reflects in surface degradation (roughening and scratches), chain scissoring, changes in the crystallinity, a decrease in the molecular weight, oxidation (i.e., the formation of C-O, C=O, ether, aldehyde, ketone, and/or carboxylic acid products), and changes in mechanical properties (a decrease in the tensile strength and elastic modulus) [7,11]. Plastic degradation can theoretically cause the formation and release of microplastics from plastics under compost conditions, but no detailed studies have been carried out. Several studies investigated microplastic release during degradation under UV light conditions and mechanical forces [12,13] However, the release of microplastics during composting was only partially investigated by Gui [4] and Sintim [14]. Gui [4] explored the possibility of polyethene (PE) films, polypropylene (PP) boards, and expanded polystyrene foams releasing microplastics during the composting process, but the experiment was conducted under laboratory conditions. The formation of microplastics under real composting conditions has been investigated only by Sintim [14] but was focused on biodegradable mulches.

Therefore, the aim of this article is to investigate and quantify the release of microplastics from conventional plastics during real open windrow composting. Also, the article presents data on the degradation of selected plastics during the composting process and its correlation with microplastics' release. The obtained results are important for understanding the origin of microplastics in ready compost and further prediction of microplastics' generation during composting depending on the plastic content in bio-waste. In addition, confirmation and quantification of the microplastics' release can give impetus to the adoption of regulations on the plastic content in organic waste used to produce fertilizers and on the content of microplastics < 2 mm in the ready compost.

2. Materials and Methods

2.1. Samples Description

To investigate the release of microplastics during composting, high-density polyethylene (HDPE), low-density polyethylene (LDPE), recycled LDPE (R_LDPE) and corn-based polylactic acid (PLA) garbage bags, polystyrene (PS) and PP (PP_fr) food containers, and PP film packaging were purchased in local markets in Lithuania. These types of plastics were chosen due to their widespread use in Europe. Photos and descriptions of plastics used for the experiment are given in Table S1. All plastic products were cut with steel scissors into 5-by-5 cm pieces (three replicants per sample), washed with distilled water to remove the microplastic particles from cutting, and dried. Then samples were weighed and put into 8-by-12 cm steel mesh bags with a pore size of 50 µm. It should be noted that microplastics smaller than 50 µm could get into/out of the mesh bag but bags with smaller pores have less openness and less airflow, which is important for normal composting. Steel mesh bags were stapled with stainless steel staples. To prevent losing the bags in the compost heap, they were connected together and placed between two steel grids with a pore size of 5 cm (Figure S1).

The experiment was handled on a real open composting site at the Alytus Regional Waste Management Centre (Lithuania). Green waste was chosen as input material for the composting, as according to the results of the previous article [8], green compost has the lowest concentration of microplastics. Composting was carried out with natural aeration and periodic reloading of the pile. The mesh bags were placed at a 60-cm depth in the compost pile for the full period of composting from August 2021 to March 2022. Before each

reloads (usually once a week), mesh bags were taken out to avoid damage and then put back in. The pile temperature at the 60-cm depth was monitored continuously using temperature sensors, and the data are presented in Figure S2. Such a long duration of the composting process was determined by the composting method (open windrow composting), the high humidity in summer, and the low temperatures in winter. It should be noted that this way of green waste composting is widely used in Lithuania and Europe in general.

2.2. Characterization of Plastic Particles

Before the final sieving of ready compost, the mesh bags were retrieved, transported to the lab, and cleaned gently on the outside to remove adhering compost. Then the mesh bags were opened, and samples (macroplastic pieces) were removed, washed with distilled water, dried at 60 °C for two hours, and subjected to further analysis.

2.2.1. Microplastics Amount Calculation

Mesh bags were clearly washed inside with distillate water. Water after washing of macroplastic pieces and mesh bags inside were collected and treated with Fenton's reagent (except PLA) based on Masura [15] protocol to remove organic residuals and vacuum filtrated on glass fiber filters. For microplastic identification and calculation, a fluorescence microscope was used. The filters were stained with Nile Red dye solution at a concentration of 10 µg mL^{-1} as in Maes [16] and examined under a fluorescent microscope on a blue excitation scale. Fluorescence particles were photographed, and then calculated and measured in ImageJ software. Detailed descriptions and efficiency of methods used for microplastic extraction and identification were presented in the previous article [8]. The recovery rate for microplastic identification using Nile Red dye was checked using ten 0.3–0.6 mm particles of each investigated polymer and was more than 90% for all plastics. Examples of fluorescence images are presented in Figure S3.

The article presents the numerical abundance of released microplastics, since the weight of microplastics < 1 mm is insufficient for direct measurement. The mass abundance of microplastics can be measured by thermal methods, but they are destructive and do not allow further characterization of microplastics [8]. However, the approximate mass of released microplastics was calculated according to Simon [17] and Braun [3]. A detailed description of the mass calculation is described in a previous article [18].

2.2.2. Determination of Weight Losses

Weight change is the simplest way to measure plastic degradation [19]. Prior to being placed in the windrow, all samples were dried at 60 °C for two hours and weighed (W_1) with an electronic microbalance (accuracy ± 0.001 mg). After the retrieval of samples from compost and their cleaning, washing, and drying, macroplastic pieces were weighed again (W_2). Weight change (W) (expressed in %) was calculated by subtracting the weight measured at the end of the experiment (W_2) from the weight at the start of the experiment (W_1) using Formula (1) [20].

$$W = \frac{W_1 - W_2}{W_1} \times 100\% \quad (1)$$

2.2.3. Chemical Analysis

Fourier transform infrared spectroscopy (FTIR) was used as an effective tool to observe the oxidation of polymers and identify the functional groups [21]. FTIR spectra of macroplastics were collected before and after the composting using a Bruker Alpha spectrometer coupled with a single reflection diamond attenuated total reflectance (ATR) accessory. The investigated wavenumber range was 4000–500 cm^{-1}, and the resolution was 4 cm^{-1}, with 64 scans performed on each sample.

2.2.4. Surface Analysis

Scanning electron microscopy (SEM) was used to observe the surface morphology and modification after the composting process. SEM effectively images surface changes such as the formation of holes and cracks [19]. Samples were sputter-coated with gold and then attached to an adhesive carbon film for observation of the surface. The SEM micrographs were recorded using the Zeiss EVO MA10 scanning electron microscope operating under a high vacuum at 20 kV.

2.2.5. Data Analysis

Statistical analysis was performed using SPSS statistics (IBM, Armonk, NY, USA) software. The release of microplastics, weight loss, and the carbonyl index were expressed as a mean ± SD. The normality test was performed using the Shapiro–Wilk test. Kruskal–Wallis and Mann–Whitney tests were used to analyze the differences between samples. A p-value < 0.05 was used to indicate statistical significance. The relationship between the amount of microplastics released and weight loss/carbonyl index/thickness of samples was estimated using a Spearman correlation analysis.

3. Results

3.1. Microplastics Amount and Mass

Figure 1 shows the amount of microplastics released from seven types of plastic during a full cycle of open windrow composting of green waste. The graph shows both the total number of microplastics found in the mesh bags and the separate numbers more and less than 50 µm. Microplastics smaller than 50 µm could get into/out of the mesh bag due to the pore size; therefore, these are assigned to a separate group.

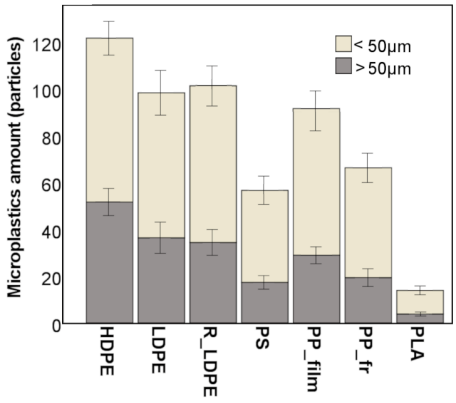

Figure 1. Microplastics amount released from macroplastics during composting. Error bars: 1 SD.

The following were the largest amounts of microplastics released from PE bags: 52 ± 6/122 ± 13 particles (>50 µm/total) from HDPE; 36 ± 7/98 ± 16 particles from LDPE and 34 ± 5/101 ± 14 particles from R_LDPE. A slightly smaller amount of microplastics was found in mesh bags with PP samples: 29 ± 4/91 ± 12 particles. However, there was no statistically significant difference between LDPE, R_LDPE, and PP film samples (p > 0.05). The hard plastic samples showed a significantly smaller amount of released microplastics: 19 ± 4/66 ± 10 particles from rigid PP and 17 ± 3/56 ± 9 particles from PS. The smallest amount of microplastics was found in the mesh bags with the PLA samples: 4 ± 1/15 ± 3 particles. Most of the microplastics found were <50 µm. A correlation analysis between the amount of released microplastics and the thickness of initial conventional macroplastic samples showed a strong negative correlation (r = −0.77 for microplastics > 50 µm, and r = −0.83 for all microplastics).

The calculated mass of released microplastics is shown in Figure 2. The highest mass released from HDPE samples was 89 ± 13 µg or 0.44 ± 0.045% from the initial mass of samples. The mass of released microplastics from LDPE and R_LDPE was half as much:

42 ± 3 µg and 37 ± 5 µg, respectively. Film PP release was 22 ± 3 µg, while rigid PP was 13.5 ± 2.5 µg. In PS bags, the mass of microplastics was 10 ± 0.5 µg. The lowest mass released from PLA samples was 3 ± 0.2 µg. A strong positive correlation was found between the mass of released microplastics and their amount (r = 0.92 for microplastics > 50 µm, and r = 0.85 for all microplastics).

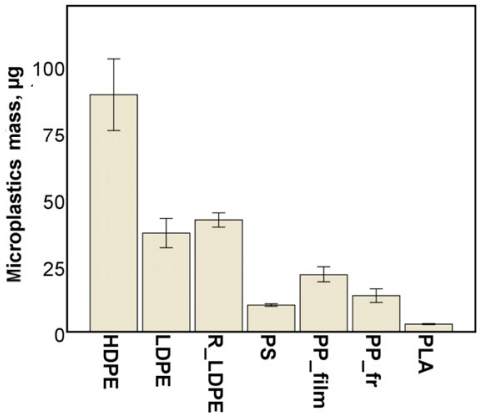

Figure 2. Mass of released microplastics.

3.2. Macroplastic Degradation

Visual observation of the macroplastics after washing and drying showed no significant changes for conventional plastic other than a yellowing of the R_LDPE, the PS, the film, and the rigid PP and a visible crack on the PS. Photos of samples before and after composting are presented in Table S2. Film samples were still transparent and flat. However, the PLA sample decomposed, and no pieces of the sample were visible.

3.2.1. Weight Loss

Weight loss in plastic samples is shown in Figure 3. In the present study, the highest weight loss was found in HDPE samples: 1.9 ± 0.33%. Weight loss in other films was significantly lower: 1.31 ± 0.17% for recycled LDPE; 0.64 ± 0.13% for LDPE, and 0.83 ± 0.13% for PP. Such a large difference may be due to the thickness of the samples. A strong negative correlation was observed between the weight loss and the thickness of the samples (r = −0.94). Hard plastics lost the least weight: PP lost 0.23 ± 0.05% and PS − 0.1 ± 0.03%. PLA was almost completely decomposed, and the weight loss was >99%, which meets the EU standards for compostable bags. The correlation analysis between weight loss and the amount of released microplastics showed a positive strong correlation (r = 0.85 for microplastics > 50 µm and r = 0.84 for all found microplastics).

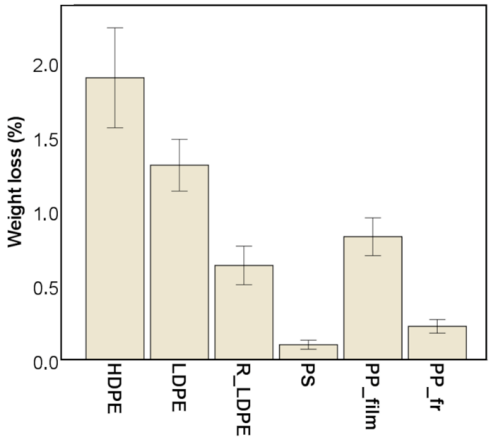

Figure 3. Weight loss in macroplastics after composting. Error bars: 1 SD.

3.2.2. FTIR

The FTIR spectra of plastics confirmed their oxidation during composting. Examples of spectra before and after composting are presented in Figure 4. A comparison of spectra showed the growth in the hydroxyl group stretching vibration region (3100–3500 cm^{-1}) and a slight increase in the carbonyl group stretching vibration region (1650–1850 cm^{-1}) for all plastics, indicating the formation of functional groups. In addition, the higher absorbance value at wavenumbers 2850 and 2922 for PS and wavenumbers 2949, 2917, and 2838 for rigid PP was noticed, possibly representing an increase in the C-H bond intensity, explained by chain scissoring [7]. HDPE also demonstrated the formation of a new peak at 1034 cm^{-1}, which can be reliable to a C-H deformation [22], and at 1639 cm^{-1}, which can reflect changes in the alkene C=C peak circa [21].

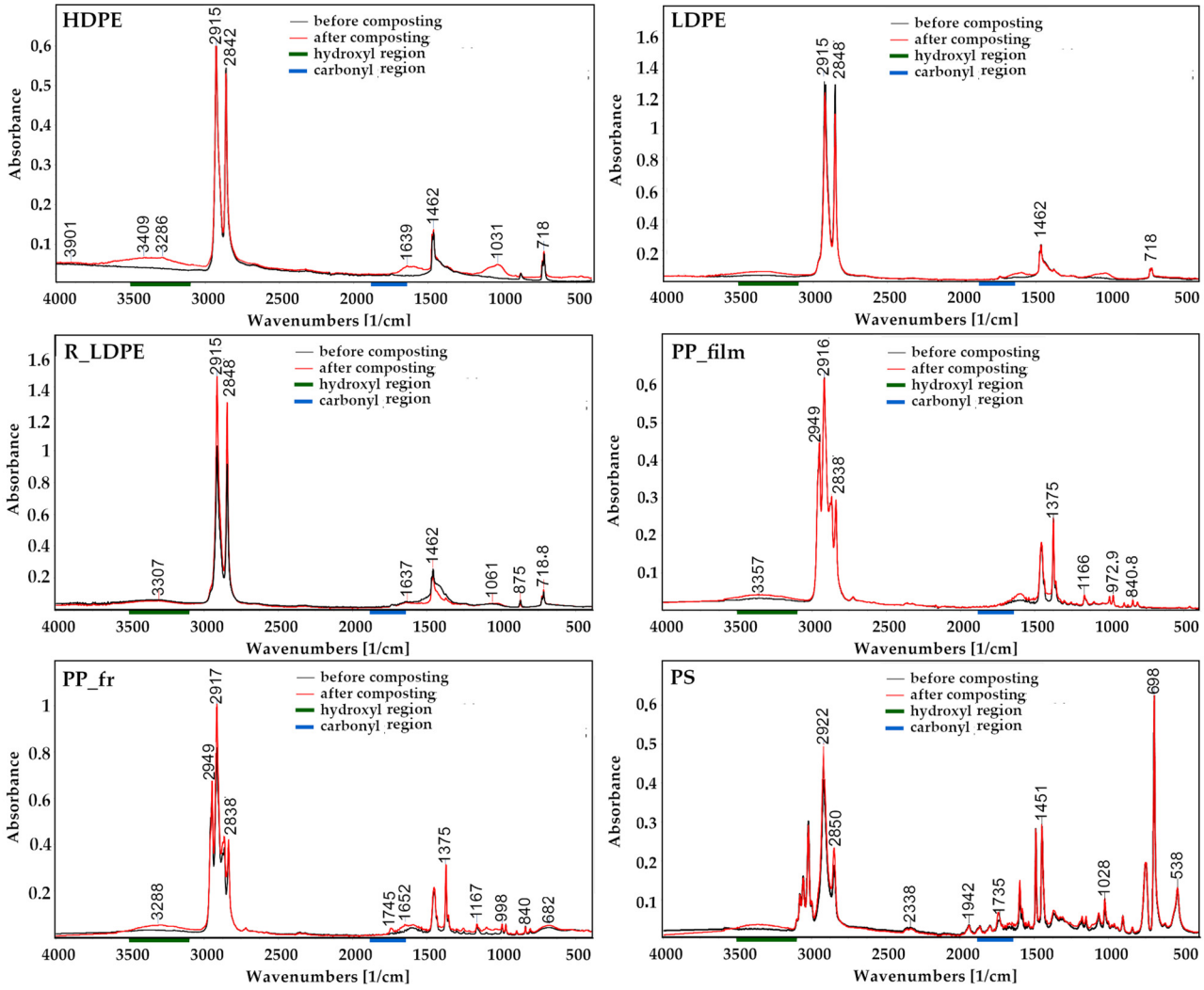

Figure 4. FTIR spectra for studied polymer type before and after composting.

3.2.3. SEM

SEM images reflected many microscopic changes on the macroplastic surface. Composting causes the creation of cracks, scratches, plowing, erosion, and holes on all samples. Some examples of SEM images for samples are presented in Figure 5. Changes in the surface of the film samples mainly included cracks and holes. At that time, observation of the surface of rigid plastics mainly revealed cracks, scratches, and plowing. It should be noted that the holes on the surface of all macroplastics were mainly <10 μm in diameter. Such surface changes as micro-holes can indicate a microbial attack during composting.

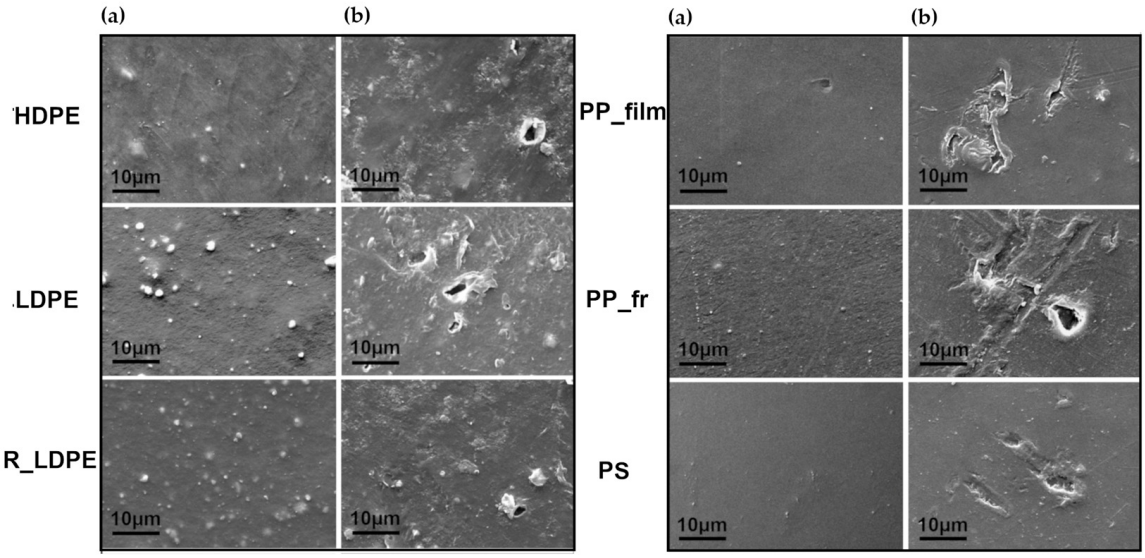

Figure 5. SEM images of the microplastic surface before (**a**) and after (**b**) composting.

4. Discussion

4.1. Microplastics' Release

Organic waste is a valuable resource, but to close the organic loop and extract the maximum benefit in the form of high-quality and pollutant-free fertilizer, it must be properly collected and processed. Improper sorting and the use of conventional garbage bags result in non-compostable plastic waste being mixed with bio-waste, which does not separate at the pre-treatment stage. The results of this work confirm that in the process of composting, microplastics can release from conventional non-compostable plastics. It was found that 17 to 52 microplastic particles > 50 mm or 56 to 122 microplastics in total were released from 5 cm^2 conventional plastic samples. The mass of released microplastics varied from 10 ± 0.5 µg to 89 ± 13 µg.

The amount of microplastics depended on the type of plastic and its thickness. PE samples released a large amount of microplastics than PP and PS samples. It can be explained by the structural stability of PP, which is a crucial property in determining its slow biodegradation [23]. Moreover, PP has more resistance to stress cracking than polyethylene [23,24]. Biodegradation of PS is even more difficult due to its unique structure, with its linear carbon backbone and alternating backbone atoms attached to phenyl moieties [25,26]. As for the difference between LDPE and HDPE, according to the literature, LDPE is more susceptible to degradation due to the branched structure, and tertiary carbon atoms at branching sites are more readily attacked [27]. However, in the current investigation, HDPE was characterized by the largest amount of released microplastic, which can be caused by the difference in sample thickness. The correlation analysis shows a strong relationship between sample thickness and the amount of released microplastics. The difference is also visible between film and rigid PP: 29 ± 4/91 ± 12 particles were released from film PP, and from rigid PP, 19 ± 4/66 ± 10 particles were released.

Gui [4] also confirmed the release of microplastics from rigid PP and film PE but found smaller amounts, on average five and nine particles, respectively. It can be connected with experiment conditions because, in the current article, the experiment was conducted in real conditions for seven months and the pore size of mech bags was 50 µm, compared with 35 days in lab conditions and 180 µm in Gui [4].

Microplastic amounts in the article were presented in two variations: total found and >50 µm. Initially, the work focused on microplastics > 50 µm based on the pore size of a steel mesh bag. However, when the microplastics were quantified, a significant amount of microplastics < 50 µm was noted, so it was decided to present them as a separate group

as well. It should be noted that microplastics < 50 µm could come from outside the mesh bag and do not belong to macroplastic samples—one of the limitations of this study.

4.2. Macroplastic Degradation

Weight loss and chemical and surface changes confirm the degradation of macroplastics during composting. In the current article, weight loss was observed for all samples. The highest weight loss was observed for HDPE: 1.9%, but a calculated average mass of released microplastics for this plastic was lower—0.44%. This means that only a small part of the weight loss is associated with the release of microplastics, the rest is most likely caused by biodegradation and the use of plastics as a source of carbon and energy by microorganisms [27]. The film samples showed a greater weight loss than the rigid samples, which correlated with the amount of released microplastics from conventional macroplastic samples ($r = -0.76$ for >50 µm and -0.88 for all found microplastics). In addition, a strong relationship between mass loss and sample thickness was noted. The importance of considering the thickness as a significant variable in the degradation test was also pointed out by Ruggero [22]. Ruggero [22] also investigated weight loss LDPE after 60 days of composting and found that LDPE lost an average of 0.72%, which is close to the values found in this work—0.64%. Vijaya and Reddy [28] studied the degradation of LDPE and HDPE during composting in soil mixed with MSW. According to their results, HDPE samples lost on average 0.47% after six months and 1.34% after eight months; and LDPE lost 0.91% and 3.2%, respectively.

SEM analysis revealed many cracks and holes on the surface of macroplastics, which may also be evidence of the release of microplastics [19]. However, all the holes found were up to 10 µm, indicating the need for a more detailed study of the release of microplastics < 50 µm, since, according to preliminary data, these microplastics make up the majority of released microplastics. Alassali [7] also confirmed that composting causes the generation of cracks and scratches and minor surface roughening on the surface of LDPE. Singh [11] studied LDPE degradation in natural compost and noticed surface erosion, exfoliation of the surface, and perforation of the matrix. However, Ruggero [22] did not observe particular visual changes on the surface of LDPE after 60 days of composting.

The analysis of FTIR spectra revealed the chemical changes of macroplastics, showing that the effect of composting is not limited to mechanical degradation. The degradation of macroplastics was confirmed by chain scissoring and the formation of new function groups in carbonyl and hydroxyl groups stretching vibration regions. The presence of carbonyl groups is one of the main characteristics of chemical aging [13,29].

4.3. Recommendations

The demand for compost is high in Europe, and organic waste is an important substrate for the production of compost. However, an important aspect remains, namely, to reduce the risk of introducing pollutants, especially microplastics, into agricultural land through such compost. Prevention of bio-waste contamination with plastics prior to treatment is the most effective and efficient approach, as removing plastic contamination from bio-waste during treatment is both expensive and limited in its effect [30].

An effective way to limit plastic pollution could be to ban the use of conventional plastic bags for disposing of bio-waste. According to the results, corn-based PLA bags are a good alternative to conventional plastic bags. PLA samples were characterized by the lowest amount of released microplastics. In addition, more than 99% of the mass decomposed during the composting period, which meets the standards of compostable plastics established by EN 13432. The degradation of PLA under compost conditions was also mentioned by Ainani [31], but it should be taken into account that at temperatures below 60 °C, the degradation rate reduces. Therefore, PLA bags can be safely composted only in an industrial composting environment. Besides, it is necessary to increase the awareness of the population through information campaigns about the correct collection

and sorting of organic waste. In addition, compost site workers can manually remove conventional plastic bags and plastic items before composting.

5. Conclusions

The study investigated the release of microplastics from the surfaces of seven types of commonly used plastics during real open windrow composting. It was found that, on average, 17–52 microplastics > 50 μm or 56–122 total were released from conventional 5-by-5 cm plastic samples. The amount of released microplastics depended on the type of plastic and its thickness ($r = -0.83$). HDPE with the smallest thickness was characterized by the largest amount of released microplastics. The mass of released microplastics varied from 10 ± 0.5 μg (PS) to 89 ± 13 μg (HDPE).

The degradation of macroplastics was confirmed by weight loss, SEM, and FTIR analyses. The highest weight loss was also found for HDPE samples: $1.9 \pm 0.33\%$, while the calculated mass of released microplastics from them was $0.44 \pm 0.045\%$ of the initial mass. The rest loss was more likely caused by biodegradation and the use of plastics as a source of carbon and energy by microorganisms Correlation analysis revealed a strong relationship between the amount of released microplastics and weight loss. SEM images showed holes, cracks, scratches, and erosion on all samples, all of which can also confirm the release of microplastics. The FTIR analysis revealed signs of a chemical change in the samples, namely the formation of new functional groups in the carbonyl and hydroxyl groups stretching vibration region, a consequence of polymer oxidation.

The corn-based PLA sample taken as a control decomposed with a weight loss of more than 99% and showed the least amount of released microplastics, namely four particles on average. This means that PLA bags can be a good alternative for the collection of bio-waste which further be industrially composted.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/su15010758/s1, Table S1: Information on plastic particles used in the experiment; Table S2: Plastic samples before and after composting; Figure S1: Plastic samples are enclosed in steel mesh bags before being buried in the compost pile (a) between steel grids (b); Figure S2: Temperature during composting; Figure S3: Examples of fluorescence image of (a) HDPE; (b) LDPE; (c) R_LDPE; (d) PS; (e) PP_film; (f) PP_fr.

Author Contributions: Conceptualization, A.S. and G.D.; Methodology, A.S.; Software, A.S.; Validation, A.S. and G.D.; Formal Analysis, A.S.; Investigation, A.S., R.K. and J.C.; Resources, A.S., R.K. and J.C.; Data Curation, A.S.; Writing—Original Draft Preparation, A.S.; Writing—Review and Editing, G.D., R.K. and J.C.; Visualization, A.S.; Supervision, G.D. All authors have read and agreed to the published version of the manuscript.

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