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Research Article

A Selective Thermal-Alkaline Hydrolysis Method for the Separation of Compostable and Conventional Plastics in Compost

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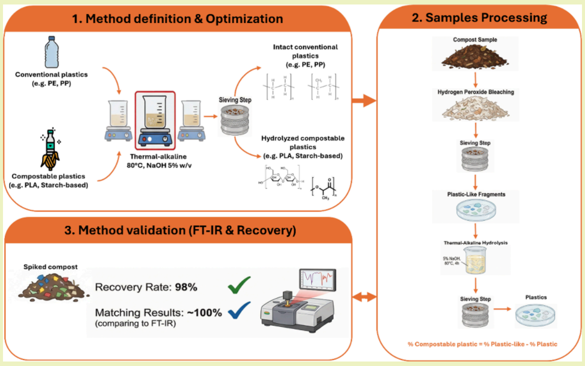
ABSTRACT: Compostable plastics, unlike conventional plastics, can be treated as biowaste and are valid components of the organic fraction of municipal solid waste. However, distinguishing between plastics and compostable plastics remains a significant challenge, particularly in composting facilities due to the lack of suitable analytical methods. This distinction is crucial for verifying the complete degradation of compostable plastics during composting and accurately assessing their organic recycling rates. In this context, this study aimed to develop an analytical method to distinguish plastic and compostable plastic residues in compost based on their different susceptibility to thermal-alkaline hydrolysis. First, optimal conditions to selectively hydrolyze starch-based and poly(lactic acid)-based compostable plastics without affecting plastics were established (80 °C, 5% w/v NaOH, 2 h). Subsequently, after isolating impurities (e.g., lithoids, metals, glass, and plastic-like materials) through hydrogen peroxide bleaching, the method was employed to distinguish plastic and compostable plastic residues in compost samples, validating the results comparing the method with attenuated total reflectance Fourier transform infrared spectroscopy. Recovery tests confirmed the method's reliability across a 0−1% w/w concentration range, with a recovery efficiency of 98%. Beyond assessing composting performance and compost quality, this method may also serve as a foundation for updating official methods referenced in national and European fertilizer regulations.



KEYWORDS: *alkaline hydrolysis, analytical method, biodegradable bioplastics, circular economy, recycling, waste management*

1. INTRODUCTION

The increasing global production and consumption of plastics have led to a pervasive environmental challenge, with plastic contamination now ubiquitous across various ecosystems, from oceans to agricultural soils.[1] A significant pathway for plastic entry into the environment is through biowaste management streams, particularly via compost derived from the organic fraction of municipal solid waste (OFMSW).[2,3] Despite regulations and efforts to promote source separation, conventional plastics (e.g., polyethylene, polypropylene, and polystyrene) frequently contaminate OFMSW, subsequently finding their way into compost products.[4] When compost is applied to agricultural lands, it introduces plastic residues into the soil, posing long-term ecological risks due to the recalcitrant nature of these materials.[3,5] The persistence of conventional plastics in soil for hundreds to thousands of years raises concerns about their accumulation, potential fragmentation into microplastics and nanoplastics, and subsequent impacts on soil health, biodiversity, and even food safety through plant uptake.[6,7]

In response to the environmental concerns associated with conventional plastics, compostable plastics have emerged as a promising alternative, offering a pathway toward a more sustainable circular economy.[8] Compostable plastics, specifically designed to degrade in industrial composting facilities, are increasingly being introduced into the market for applications such as packaging, food-service ware, and bags for biowaste collection.[9] Their intended end-of-life scenario is composting, where they are converted into carbon dioxide, water, and biomass under specific conditions, thereby theoretically preventing their accumulation in the environment.[10] Notably, compostable plastics can be assimilated to biowaste representing biodegradable polymers, like many other degradable polymers characterizing the OFMSW. As consequence of that, they act as a slow-degrading polymer in soil providing energy to soil microorganisms.[11] Conversely, conventional plastics are considered undesirable contaminants in composting plants and in compost.

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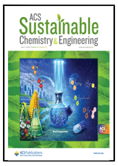

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However, the widespread adoption of compostable plastics and their effective management within existing waste infrastructure are not without challenges because it is very hard to distinguish between compostable plastic and plastic residues in the final compost. Considering that the sieve mesh commonly used to refine compost in industrial plants ranges from 5 to 10 mm, fragments of plastic-like materials in this size range can be readily present in the final compost. European Regulation 1009/2019 on marketable fertilizer products (including compost) does not differentiate between plastics and compostable plastics when defining limits for impurities >2 mm in compost, treating all plastic-like materials as contaminants.[12] This can lead to an overestimation of plastic contamination, potentially hindering the market acceptance of compost products and discouraging the use of compostable plastics, even when they have been designed as compostable. Furthermore, the lack of reliable analytical tools limits the ability to accurately track the fate of compostable plastics during composting, making it difficult to assess their organic recycling rate and to optimize composting conditions.[13]

Existing methods for plastic analysis in environmental samples typically involve density-based separation, visual inspection, and spectroscopic techniques like Fourier transform infrared (FT-IR) spectroscopy or Raman spectroscopy (Table S1).[14] While FT-IR spectroscopy is a powerful tool for identifying polymer types based on their unique spectral fingerprints, it is often labor-intensive, particularly when dealing with small or fragmented particles that need to be separated from organic matrices requiring significant sample preparation.[15] Furthermore, direct quantification of plastic and compostable plastic residues within heterogeneous matrices like compost using spectroscopic methods can be challenging due to matrix interferences (e.g., the presence of compost residues) and the difficulty of analyzing large numbers of individual very small particles.[16] Other techniques, such as pyrolysis−gas chromatography−mass spectrometry (Py-GC/MS), offer high sensitivity and specificity for polymer identification but are often complex and costly and require specialized equipment and skilled personnel, making them less suitable for routine analysis or large-scale screening.[17] Given these limitations, there is a clear and urgent need for simplified, cost-effective, and accurate methods for distinguishing and quantifying plastic and compostable plastic residues in complex organic matrices like compost. In this context, this study proposed a novel selective thermal-alkaline hydrolysis method for the efficient separation and quantification of plastics and compostable plastics in compost samples. Scientific literature consistently reported that compostable plastics, contrarily to plastic, dissolve as the consequence of alkaline hydrolysis at both high pH and temperatures (>55 °C),[18,19] suggesting adopting the selective hydrolysis under alkaline conditions as a simple method to recognize compostable plastic from plastic.

This work aimed to (i) optimize the thermal-alkaline conditions to achieve selective hydrolysis of various types of compostable plastics while ensuring the stability and integrity of conventional plastics, (ii) to validate the proposed method by comparing its quantitative and qualitative results against those obtained from a control method (i.e., FT-IR spectroscopy), (iii) to assess the recovery efficiency of the method, and (iv) to evaluate the current levels of plastic and compostable plastic residues in five composts sampled from facilities treating OFMSW.

2. EXPERIMENTAL SECTION

2.1. Materials and Compost Sampling and Preparation

To use materials that effectively can be retrieved in OFMSW compost samples, commercial plastic and compostable plastic materials available at Italian supermarkets were employed in the method development and validation. Prior to the experiments, the compostable plastics and plastics used to both define the optimal hydrolysis conditions (Section 2.2) and conduct the recovery tests (Section 2.4) were identified via attenuated total reflectance (ATR) FT-IR spectroscopy (4500 series portable FT-IR spectrometer, Agilent, Santa Clara, United States). Polymer identification was performed considering both spectral matching with reference libraries (Agilent Poly 8 Library and an in-house database; match factor >85%, see also Section 2.3) and the presence of characteristic diagnostic absorption bands reported in the literature (Table S2), ensuring reliable discrimination among conventional plastics and compostable plastics. After identification, samples were cut into pieces of approximately 0.5 × 0.5 cm in size. This controlled size was selected to ensure reproducibility of the thermal-alkaline treatment and to represent fragments within the typical size range of impurities found in compost, i.e., smaller than 10 mm (the most common mesh size used in compost sieving) but larger than 2 mm (the threshold for impurities in compost according to European Regulation 1009/2019). 2 mm is also the threshold set by the international standardized test for defining a plastic material disintegrated under composting conditions (ISO 20200:2023). It should be noted that this size specification (0.5 cm × 0.5 cm) applies only to the reference materials used in the optimization and validation steps. In contrast, plastic and compostable plastic fragments recovered from compost samples (Section 3.2) and used in the optimization step (Section 2.2) were not modified and therefore exhibited irregular shapes and variable dimensions, representative of real environmental samples.

The compostable plastic items used included starch-based (SB) commercial shopping bags ($n = 3$), SB bags for the sorted collection of OFMSW (Naturabiomat GmbH, Schwaz, Austria), and PLA-based cutlery, plates, and cups (Bibo Italia spa, Turin, Italy) available in Italian supermarkets. All items were labeled as compostable by TÜV Austria and the Italian Consortium of Composters (CIC), according to standard UNI EN 13432:2002. According to the literature, commercial SB compostable plastics employed in this study were mainly composed of starch and polyester components, i.e., poly(butylene adipate-*co*-butylene terephthalate, PBAT) (Figure S1B),[10,18] as highlighted by the diagnostic peaks at 3400−3200 and 1080−1020 cm^{-1} (O−H and C−O stretching, assigned to starch component) and 1750−1730 cm^{-1} (C═O stretching, assigned to the polyester component) (Table S2). PLA-based compostable plastics showed the typical absorption bands of PLA (Figure S1B and Table S2), with diagnostic peaks assigned at 1760−1750 cm^{-1} (C═O stretching), 1180−1080 cm^{-1} (C−O−C stretching), and 2995−2945 cm^{-1} (CH_3 stretching).

Conventional plastic items were selected to be representative of the most common plastics found in compost produced starting from OFMSW:[4] LDPE from waste collection bags (film, 0.1 mm thickness), HDPE from vessels used for food storage (film, 0.5 mm thickness), PP from food packaging (film, 0.1 mm thickness), PVC from vessels used for food storage (rigid, 0.3 mm thickness), PET from water bottles (film, 0.2 mm thickness), PS from single-use glasses (Bibo Italia spa, Turin, Italy) (rigid, 0.2 mm thickness), and PS from food containers (expanded, 10 kg m^{-3} density). Conventional plastics were also identified prior and after experiment by identifying their diagnostic peaks (Table S2) and matching the FT-IR spectra with libraries (match factor >85%).

Representative compost samples (about 10 kg) were collected from five plants treating OFMWS in Italy (Table S3), representing the most representative technologies for OFMSW treatment, i.e., composting and anaerobic digestion. Of the five plants, three operated aerobic composting of OFMSW mixed with green waste (GW), while the other two performed anaerobic digestion of OFMSW and GW, followed by digestate composting. In all plants, compost samples were collected after the final sieving, whose mesh varied in the range from 8 to 10 mm. Before the experiments, the compost samples were characterized for total solids (TS), volatile solids (VS), total organic carbon (TOC), organic matter, total nitrogen (N), C/N, pH, and electrical conductivity (EC). The results are reported in Table 1.

Table 1. Characteristics of Composts from OFMSW Used in Method Validation[a]

parameter	unit	#1	#2	#3	#4	#5
total solids	%	63 (0)	79 (0)	83 (0)	71 (2)	67 (2)
volatile solids	%	75 (0)	74 (1)	57 (0)	64 (2)	64 (0)
pH	pH unit	6.2 (0.1)	7.6 (0)	9.3 (0)	8.2 (0.1)	8 (0)
electrical conductivity	dS m^{-1}	6.6 (0.4)	6.9 (0.5)	3.8 (0.1)	8.7 (0.1)	8.1 (0.1)
total organic C	%	36 (0)	36 (0)	28 (1)	23 (1)	22 (0)
organic matter	%	73 (1)	72 (0)	56 (1)	46 (0)	44 (1)
total N	%	2.3 (0.1)	2.7 (0.2)	2.1 (0)	1.8 (0.2)	1.8 (0)
C/N		15.6	13.3	13.3	12.8	12.2

[a] Mean value (SD), $n = 3$. Data are expressed on a dry weight basis except for total solids, pH, and electrical conductivity.

2.2. Definition of the Optimal Method Conditions

Following the scheme shown in Figure 1, the initial experiments were designed to determine the optimal conditions for selectively hydrolyzing compostable plastics and enabling their removal from the solid phase while leaving conventional plastics intact.

All tests were conducted in 100 mL glass flasks using samples of plastics and compostable plastics prepared as described in Section 2. The experimental design also included aged samples of plastics (LDPE, HDPE, PP, PET, PVC, and PS) and compostable plastics (PLA-based and SB-shoppers) recovered from the composts used for method validation (Section 2). All samples were predried in a ventilated oven at 45 °C until a constant weight was reached. Each material was tested in five replicates, resulting in a total of 110 flasks per each thermal-alkaline treatment: (7 plastics + 7 compostable bioplastics + 6 aged plastics + 2 aged bioplastics) × 5 replicates.

Based on literature evidence, all test conditions were preceded by a preliminary thermal treatment in deionized water (about 0.5 g of the sample in 100 mL of water resulting in a sample concentration of 0.5% w/v) at 80 °C for 2 h (100 rpm stirring). This step was essential to promote the glass transition of compostable plastics to the amorphous structures, which are known to be more prone to hydrolysis.[11]

After 2 h, NaOH (reagent grade, ≥98%, anhydrous pellets; Merck KGaA, Darmstadt, Germany) was added to achieve the target concentrations (T1: 0%, T2: 1.25%, T3: 2.5%, T4: 5%, and T5: 10% w/v), as reported in Table S4. Following the NaOH addition, the samples were subjected to an additional 2 h treatment under the same temperature and stirring conditions (80 °C, 100 rpm).

After the thermal-alkaline treatment, the contents of each flask were sieved using a 2 mm mesh steel sieve. A 2 mm mesh was selected, as both European and Italian regulations on marketable fertilizers set 2 mm as the size threshold for plastic-like residues in compost.[12,20] The remaining materials were recovered and dried in a ventilated oven at 45 °C until a constant weight was achieved.

2.3. Validation of the Method through Comparison with FT-IR Spectroscopy

Once the optimal conditions for selectively hydrolyzing compostable plastics were established (80 °C, 5% w/v NaOH), the method was validated using five compost samples (three replicates for each compost) collected from composting plants in Italy that treat the OFMSW (Section 2.1, Table S3), as outlined in Figure 1.

Before applying the selective hydrolysis method, a preliminary separation of impurities (i.e., lithoids, glass, metals, and plastic-like materials, including plastics and compostable plastics) from the compost samples was performed. This was done using the method proposed by ANPA (see Section 2.5).[21] Briefly, impurities (plastic-like materials, metals, glass >2 mm, and lithoids >5 mm) were determined by H_2O_2 30% v/v digestion to remove organic matter, starting from a 100 g dry weight (dw) of compost samples. Digestion was carried out at 45 °C until reaction ceased (i.e., no gas development, about 24 h), by adding small aliquots (10−20 mL) of 30% v/v H_2O_2 to avoid excessive foaming and potential sample losses. Treated samples were then sieved by a 2 mm mesh sieve, washed with deionized water, dried at 45 °C until a constant weight was achieved and manually separated. Once isolated, the plastic and compostable plastic residues recovered from the compost were first analyzed by using ATR FT-IR spectroscopy (4500 series portable FT-IR spectrometer, Agilent, Santa Clara, United States) and matching the spectra against the Agilent Poly 8 Library and an in-house database. Plastic and compostable plastic residue spectra were then labeled if they met a minimum match threshold of 85% when compared to the libraries and following identification of diagnostic peaks (Table S2). After their identification and counting, plastic and compostable plastic fragments weights were determined using an analytical balance (±0.0001 g).

Subsequently, all of the recovered plastic and compostable plastic fragments from each compost replicate were recombined and subjected to the selective hydrolysis method. The fragments were placed in a 100 mL glass flask, and deionized water was added (100 mL). The flasks were heated to 80 °C and stirred at 100 rpm for 2 h. After this, NaOH (reagent grade, ≥98%, anhydrous pellets; Merck KGaA, Darmstadt, Germany) was added to reach the target concentration of 5% w/v, and the mixture was left for an additional 2 h under the same temperature and stirring conditions. Following the thermal-alkaline treatment, the content of the flasks was sieved through a 2 mm mesh steel sieve. The retained residues were recovered, dried in a ventilated oven at 45 °C until constant weight was achieved, and then weighed, theoretically providing the weight of conventional plastic residues in the compost. All recovered fragments were again identified via ATR FT-IR spectroscopy and counted.

Finally, the results of the two analytical approaches, i.e., ATR FT-IR spectroscopy and selective hydrolysis, were compared to validate the method both quantitatively (percentage of plastic and compostable plastic residues in compost, expressed on a dry weight basis) and qualitatively (number and type of residue).

2.4. Validation of the Method through Recovery Tests

The selective hydrolysis method developed in this study was further validated through recovery tests using plastics and compostable plastics (Section 2.1). Fifteen compost samples of about 500 g (three for each compost used in the method validation, Section 2.3) were artificially spiked with different combinations of plastics and compostable plastics at concentrations ranging from 0 to 1% w/w (on a dry weight basis), as detailed in Table S5.

The recovery efficiency of the method was assessed by applying the same procedure described in Section 2 to each spiked compost sample, i.e., impurity separation followed by selective hydrolysis of compostable plastics under thermal-alkaline conditions. The results obtained from the recovery tests (expressed as the percentage of plastics and compostable plastics in the compost) were then compared to the theoretical concentrations, which were calculated based on the known spiked amounts and the experimental data from the method validation using unspiked compost samples (Section 2).

2.5. Analytical Methods

A detailed description of the analytical methods used for compost characterization, impurity separation, and ATR FT-IR spectroscopy procedures is provided in Table S6.

2.6. Statistics and Calculations

All the reported results are mean values of three replicates (five for the optimization of selective hydrolysis conditions). Mean values and

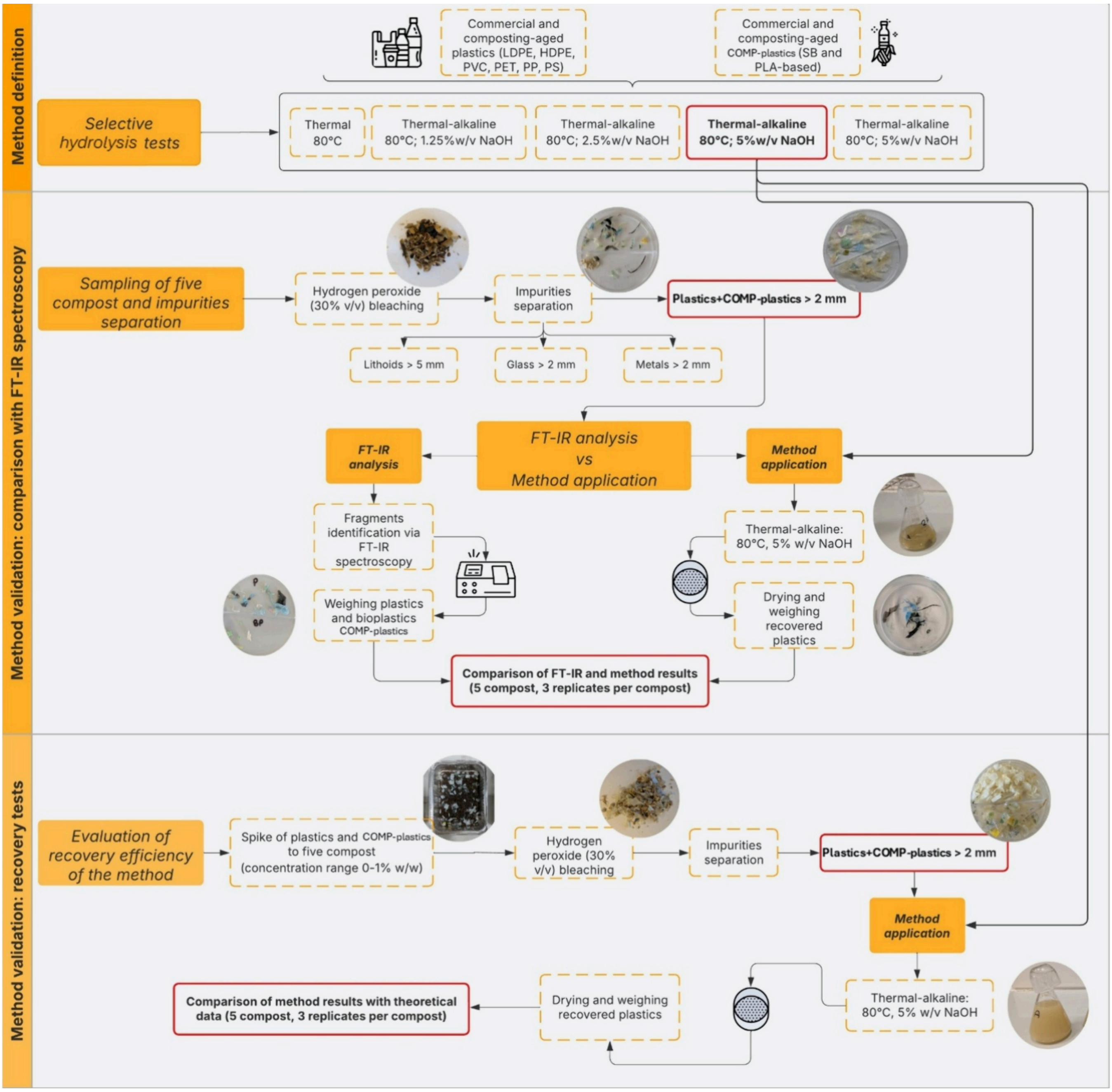

Figure 1. Experimental design for method optimization and validation. COMP-plastics: compostable plastics.

standard deviation were calculated by using XLSTAT for Microsoft Excel (Addinsoft, XLSTAT statistical and data analysis solution, 2024, version 2024.1). Determination of significant differences among the parameters analyzed over time at a level of significance of $P < 0.001$ was carried out by analysis of variance (ANOVA) and Tukey's test, after verifying the normality and the homoscedasticity of the data by graphical analysis of residuals (XLSTAT for Microsoft Excel, Addinsoft, XLSTAT statistical and data analysis solution, 2024, version 2024.1).

The weight loss (%) of plastic and compostable plastic samples after the hydrolysis tests under different conditions (Section 2.2) was calculated by applying the following equation (eq 1):

$$\text{weight loss (\%)} = \frac{m_0 - m_T}{m_0} \times 100 \tag{1}$$

where m_0 was the sample weight before thermal-alkaline treatment and m_T was the sample weight after thermal-alkaline treatment.

The recovery efficiency of the method was calculated by applying the following equation (eq 2):

$$\text{recovery efficiency (\%)} = \frac{m_{\text{experimental}}}{m_{\text{theoretical}}} \times 100 \tag{2}$$

where $m_{\text{experimental}}$ was the total weight of plastics and compostable plastics as determined in the recovery tests and $m_{\text{theoretical}}$ was the total weight of plastics and compostable plastics as calculated considering the known spiked concentration and the experimental results from this work.

Despite the balance readability of 0.0001 g, a practical resolution of 0.001 g was adopted to account for sample handling and replicate variability. Accordingly, the minimum quantifiable concentration of plastic-like materials in compost was estimated as 0.001% (w/w) based on the 100 g sample size.

Table 2. Weight Loss of Plastics and Compostable Plastics Samples under Different Thermal-Alkaline Hydrolysis Tests[a]

	material	T1	T2	T3	T4	T5
plastics	LDPE	0.04 (0.13)	0.11 (0.21)	0.05 (0.36)	0.01 (0.02)	0 (0.77)
	HDPE	0.13 (0.05)	0.09 (0.08)	0.14 (1.4)	0.63 (0.98)	1.4 (2.3)
	PP	0 (2.3)	0.04 (0.1)	0 (0.32)	0 (1.5)	1.1 (1.9)
	PET	0.02 (0.1)	0 (0)	0.15 (0.19)	0.61 (0.9)	0.87 (1.8)
	PVC	0 (0.11)	0.31 (0.58)	0 (0.16)	0 (0.14)	0.06 (0.27)
	PS	0 (0.2)	0 (0.1)	0.13 (0.32)	0.05 (0.15)	0 (0.16)
	PS-expanded	0 (0.3)	0 (0.06)	0.31 (0.68)	0 (1.47)	0 (3.1)
aged plastics	LDPE	0.05 (0.11)	0.12 (0.19)	0 (0.11)	0.18 (0.31)	0.41 (1.2)
	HDPE	0.08 (0.21)	0.21 (0.61)	0 (0.56)	0 (0.14)	0.51 (0.94)
	PP	0 (0.14)	0.07 (0.23)	0.29 (0.59)	0 (0.21)	0 (0.11)
	PET	0 (0.14)	0 (0.02)	0.39 (0.88)	0.31 (1.2)	0.15 (0.33)
	PVC	0 (1.1)	0 (0.03)	0.74 (2.31)	0.11 (0.13)	0.41 (0.98)
	PS	0.12 (0.35)	0 (0)	0 (0.01)	0 (0.21)	0 (0.46)
compostable plastics	PLA-cutlery	0.32 (0.23)	31.5 (5.6)	50 (0.3)	100 (0)	100 (0)
	PLA-dish	0.05 (0.02)	59 (3.8)	53.9 (2.9)	100 (0)	100 (0)
	PLA-glass	0.19 (0)	88.3 (0.9)	84.6 (0.9)	100 (0)	100 (0)
	SB-shopper1	8.4 (1.7)	15.9 (2)	21.6 3	100 (0)	100 (0)
	SB-shopper2	4.5 (1.2)	19.6 (4.1)	29.1 (5.3)	100 (0)	100 (0)
	SB-shopper3	4.5 3	24.1 3	25.9 (2.4)	100 (0)	100 (0)
	SB-sorted collection	1.9 (1.4)	21 (2.9)	27.2 (1.1)	100 (0)	100 (0)
aged compostable plastics	PLA	0.98 (0.19)	84 (2.3)	89.9 (2.6)	100 (0)	100 (0)
	SB-shopper	1.1 (0.3)	36.9 (2)	45.3 (3.5)	100 (0)	100 (0)

[a] Mean value (SD), $n = 5$. T1: thermal (80°C, 4 h); T2: thermal-alkaline (80°C, 1.25 w/v NaOH, 4 h); T3: thermal-alkaline (80°C, 2.5 w/v NaOH, 4 h); T4: thermal-alkaline (80°C, 5 w/v NaOH, 4 h); T5: thermal-alkaline (80°C, 10 w/v NaOH, 4 h).

3. RESULTS AND DISCUSSION

3.1. Optimization of Method Conditions for Selective Hydrolysis of Compostable Plastics

To identify the optimal conditions for the selective hydrolysis of compostable plastics while preserving the integrity of traditional fossil-based plastics, various plastic and compostable plastic samples were subjected to a range of thermal and thermally alkaline hydrolysis tests, and their weight loss was quantified (Table 2). Traditional plastics, including LDPE, HDPE, PP, PET, PVC, and PS, exhibited negligible weight loss across all tested conditions, ranging from T1 to T5. For most traditional plastic types, the mean weight loss remained below 1% even at the highest NaOH concentrations (T4 and T5). For instance, virgin LDPE showed a maximum mean weight loss of 0.11 ± 0.21% in T2, while it was 0 ± 0.77% in T5. Similarly, aged LDPE showed a maximum weight loss of 0.41 ± 1.2% in T5. No significant trend of increased weight loss with an increasing NaOH concentration was observed for traditional plastics, indicating their resistance to the alkaline conditions employed, as confirmed by the FT-IR analysis of the samples recovered after treatment (Figure S1a), which indicated the absence of any qualitative modifications of plastics. Indeed, polyolefins (LDPE, HDPE, and PP) were consistently identified by the presence of intense C−H stretching bands at ~2915 and 2848 cm^{-1} and bending vibrations in the range of 1470−1375 cm^{-1}, which remained unchanged after thermal-alkaline treatment, confirming their chemical stability under the tested conditions (Table S2 and Figure S1A). This consistent lack of weight loss for both virgin and aged materials suggested that the aging process (i.e., composting) did not compromise their chemical stability under alkaline conditions, in accordance with the literature.[22] The inherent resistance of plastics was attributed to their chemical structures, which are typically composed of robust carbon−carbon backbones (in polyolefins such as LDPE, HDPE, PP, and PS) or strong covalent bonds (in PVC, where chlorine atoms are directly bonded to the carbon chain) that are not readily attacked by nucleophilic species such as hydroxide ions under these conditions.

In contrast to plastics, compostable plastics demonstrated significant and concentration-dependent weight loss under thermal-alkaline conditions. The difference in weight loss following thermal-alkaline hydrolysis between traditional plastics and compostable plastics was statistically significant across all alkaline treatments ($P < 0.001$) (T2−T5).

PLA-based plastics showed remarkable sensitivity to NaOH. Even at the lowest NaOH concentration (T2: 1.25% w/v), PLA-cutlery, PLA-dish, and PLA-glass exhibited substantial weight losses of 31.5 ± 5.6%, 59.0 ± 3.8%, and 88.3 ± 0.9%, respectively. This trend continued to increase with higher NaOH concentrations, reaching complete (100 ± 0%) weight loss following thermal-alkaline hydrolysis for all three PLA products at T4 (5% w/v NaOH) and T5 (10% w/v NaOH). PLA-based materials were characterized by a strong ester carbonyl band at ~1750 cm^{-1} and C−O−C stretching vibrations between 1180 and 1080 cm^{-1} (Figure S1B and Table S2). The susceptibility of these bonds to alkaline hydrolysis likely explains the complete dissolution observed under T4 conditions, i.e., in the presence of a strong base like NaOH, the ester bonds of PLA undergo alkaline hydrolysis, yielding a carboxylate and an alcohol. Over time, as more ester bonds are cleaved, the polymer chains break down into smaller oligomers and eventually lactic acid monomers.[23] The elevated temperature (80 °C) likely accelerated the hydrolytic process by increasing reaction kinetics as well as promoting the glass transition of crystalline structures into amorphous ones, promoting their complete removal from the solid phase.

SB compostable plastics also showed significant weight loss as the NaOH concentration increased. At T2, weight losses

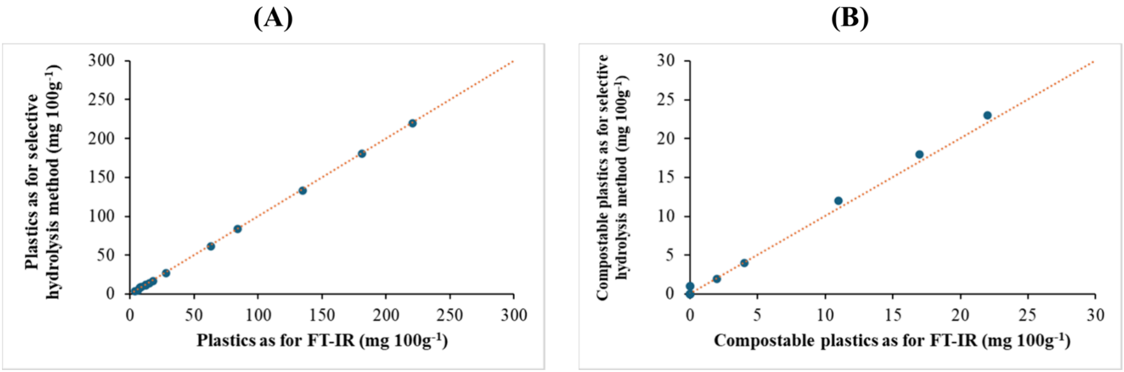

Figure 2. Comparison of FT-IR and selective thermal−alkaline hydrolysis methods for plastic and compostable plastic residue quantification in compost. (A) Plastic and (B) compostable plastic mass determined by the two methods (*n* = 15). The orange line represents the 1:1 relationship. The close agreement indicates equivalent performance of both analytical approaches.

ranged from 15.9 ± 2% (SB-shopper1) to 24.1 ± 3% (SB-shopper3). By T3 (2.5% w/v NaOH), weight loss values for SB-based materials were between 21.6 ± 3% and 29.1 ± 5.3%. All SB compostable plastics achieved 100 ± 0% weight loss following thermal-alkaline hydrolysis at T4 and T5 (5% and 10% w/v NaOH, respectively). The observed behavior of SB compostable plastics in alkaline conditions was likely due (i) to the hydrolysis of the ester linkages in the polyester component of SB compostable plastics, i.e., poly(butylene adipate-*co*-butylene terephthalate, PBAT),[10,18] and (ii) to a process known as peeling reaction for the polysaccharide component. During the peeling reaction, the reducing end of a polysaccharide can undergo a series of reactions leading to chain scission and degradation under alkaline conditions.[24] These concurring factors may have enabled the total removal of SB compostable plastics from the solid phase (<2 mm).

The aged SB-shopper and PLA-based samples demonstrated hydrolysis patterns consistent with, or even slightly more accelerated than, some virgin compostable plastics at lower NaOH concentrations. Aged PLA residues exhibited high susceptibility to alkaline degradation, with 84 ± 2.3% weight loss at T2 and 89.9 ± 2.6% at T3, culminating in complete (100 ± 0%) weight loss at T4 and T5. The aged SB-shopper residue showed 36.9 ± 2% weight loss at T2 and 45.3 ± 3.5% at T3, reaching 100 ± 0% weight loss at T4 and T5. This suggested that the aging process (i.e., composting), which may involve some degree of predegradation, might slightly facilitate their subsequent alkaline hydrolysis by creating more accessible end-groups or reducing crystallinity.[25] This suggested that the efficiency of the selective thermal-alkaline hydrolysis method defined in this work is likely maintained even for wasted or aged compostable plastics.

Overall, the T4 conditions tested in this study (5% w/v NaOH at 80 °C for 4 h) were found to selectively hydrolyze various types of compostable plastics while leaving traditional plastics intact. In addition, the magnitude of weight loss observed for compostable plastics (up to 100% weight loss) was significantly higher than the measurement uncertainty associated with the analytical balance, ensuring that the reported trends were statistically robust. Therefore, these conditions were selected for use in the subsequent experiments (Sections 3 and 3.3). Although the T5 conditions produced similar results to T4, they were not considered further, as they require more chemicals, leading to increased costs and hazard.

3.2. Method Application: Plastic and Compostable Plastic Residue Separation in Compost Samples

3.2.1. FT-IR Spectroscopy vs Selective Thermal-Alkaline Hydrolysis Method: Quantitative Insights.

The selective thermal-alkaline hydrolysis method proposed in this study was first validated by comparing its results with those obtained using a control identification method, i.e., FT-IR spectroscopy (Figure 1). Quantitative results of the separation of compostable plastics and plastics following their recovery from the compost matrix and using both methods are presented in Table S7, while Figure 2 reports the comparison of the results from both methods.

By assigning diagnostic peaks and matching the spectra with libraries (Table S2), FT-IR spectroscopy enabled the identification of compostable plastic fragments in all compost samples except compost #1. On average, 9, 2, 1, and 1 compostable plastic fragments were recovered from 100 g of dry weight (dw) of compost #2, #3, #4, and #5, respectively (Table S7). These corresponded to concentrations of 0.017% dw (compost #2), 0.002% dw (compost #3), and 0.001% dw (compost #4). In compost #5, the concentration of compostable plastics was below the minimum quantifiable concentration (i.e., 0.001% dw) (Table S7). Plastic fragments were significantly more abundant than compostable plastics across the compost samples, ranging from 8 fragments (compost #4) to 42 fragments (compost #1) per 100 g of compost, corresponding to concentrations between 0.01% and 0.16% dw. The low residual concentrations of compostable plastics observed in compost likely result from suboptimal process conditions leading to incomplete disintegration and biodegradation, such as localized anaerobic microenvironments or inadequate frequency/intensity of turning and aeration, rather than from any intrinsic limitations in the biodegradation of compostable materials. This interpretation is supported by the fact that compostable plastic items entering industrial composting streams are, in principle, compliant with established compostability certification standards (UNI EN 13432:2002).

The thermal-alkaline treatment selectively hydrolyzed only compostable plastics fragments, as confirmed by FT-IR analysis of the fragments recovered after the application of the method, all of which were identified as plastics (Tables S2, S7, and S8 and Figure S2). Notably, the number of plastic fragments recovered after the selective thermal-alkaline hydrolysis treatment matched exactly the number recovered directly from the compost in all analyzed samples. Supporting the

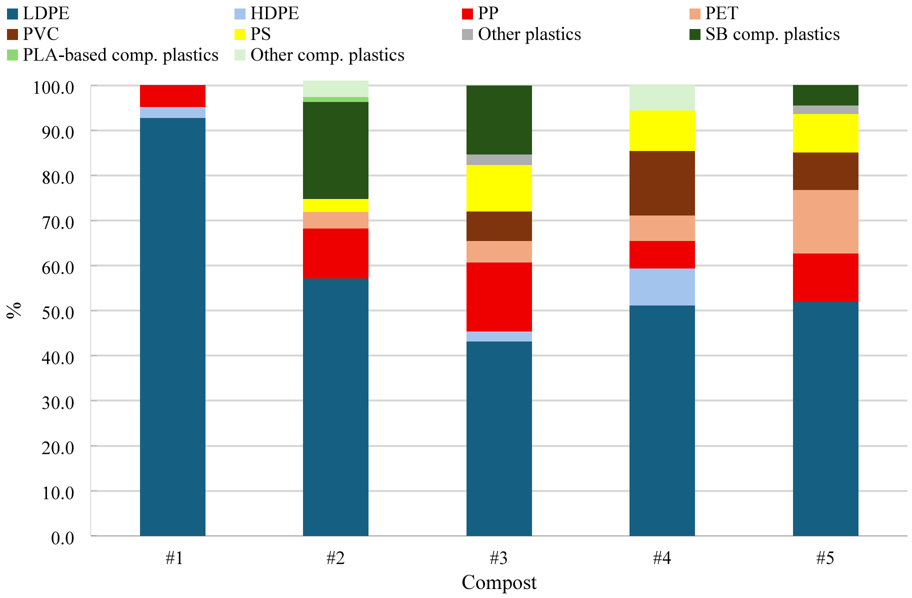

Figure 3. Classification of plastic and compostable plastic residues in composts according to FT-IR spectroscopy carried out on fragments recovered after hydrogen peroxide bleaching (% of total fragments). Comp. plastics: compostable plastics.

method's high selectivity, the mass of plastic fragments recovered after the thermal-alkaline treatment was in agreement with the values obtained via FT-IR identification, with discrepancies lower than 5% in all samples (Figure 2).

Finally, the concentrations of compostable plastic residues determined using the proposed method closely matched those obtained via FT-IR analysis: 0.017% (FT-IR) vs 0.018% (proposed method) in compost #2, 0.002% vs 0.002% in compost #3, and 0.001% vs 0.001% in compost #4 (Figure 2).

3.2.2. FT-IR Spectroscopy vs Selective Thermal-Alkaline Hydrolysis Method: Qualitative Insights.

Beyond quantitative recovery, assessing the composition of plastic and compostable plastic residues may provide critical insight into the types and origins of contamination within compost derived from OFMSW. Figure 3 presents the classification of plastic and compostable plastic fragments identified through FT-IR spectroscopy (i.e., matching spectra with libraries at a minimum threshold of 85% and identifying diagnostic peaks) (Table S2), expressed as the percentage of total fragments recovered from each compost sample following hydrogen peroxide bleaching. In addition, Tables S7 and S8 report the results of the count of plastic and compostable plastic fragments isolated and identified in the five composts (three replicates each). It should be noted that FT-IR analysis provides tentative polymer identification, particularly for plastics extracted from complex matrices such as compost, where biofilms and organic contaminants can produce atypical spectra (Figure S3). To improve the reliability of polymer assignments, a minimum match threshold of 85% was applied in this study, considerably higher than the 70% typically accepted in the literature.[26]

Across all composts, LDPE was the predominant plastic, particularly in compost #1, where it accounted for over 90% of the fragments. LDPE also remained the most abundant material in the other samples. This dominance likely reflects the widespread use of LDPE in household packaging, such as shopping bags and flexible food wraps,[27] which may escape source separation or are mistakenly disposed of with biowaste despite not being compostable.

Other conventional plastics, including PP, PS, PET, PVC, and HDPE, were identified in varying proportions in compost #2 through #5. These materials originate predominantly from consumer product packaging, food containers, disposable tableware, and other single-use items commonly present in municipal waste streams.[27] These findings are fully consistent with the literature,[4,15] where PE, PS, PP, PET, and PVC have been identified as the main synthetic polymers contaminating compost derived from OFMSW, with PE being the most abundant. Interestingly, the same polymers have also been found to be the primary constituents of plastic contamination in garden composts.[16]

As mentioned in Section 3.2.1, compostable plastics were detected in composts #2–#5, with SB compostable plastics representing the majority, accounting for up to 25% and 20% of total fragments in composts #2 and #3, respectively. The presence of SB compostable plastic residues in compost derived from biowaste was also recently reported in the literature.[15] These materials are commonly used in certified compostable bags, food-service items, and packaging marketed for biowaste disposal and thus represent an increasing fraction of OFMSW.[25] In this study, SB compostable plastic residues were characterized by an FT-IR spectrum where the absorption bands identifying the starch component of the bioplastic (i.e., 3400–3200 and 1080–1020 cm^{-1}) showed reduced intensity with respect to unaged SB compostable plastics (Table S2 and Figures S1B and S3). This was in accordance with the literature dealing with SB compostable plastic degradation during anaerobic digestion and composting, where the preferential consumption of starch by microorganisms is frequently reported.[10,28]

Notably, only one compost (i.e., compost #2) showed detectable residues of PLA-based compostable plastics, possibly indicating either a lower consumer awareness regarding the correct disposal of PLA-based items into the OFMSW stream or a higher degree of biodegradability and disintegrability of PLA during the composting process. The FT-IR spectra of PLA-based compostable plastic residues found in compost #2 were characterized by the diagnostic absorption peaks typically ascribed to PLA (i.e., 1760−1750, 1180−1080, and 2995−2945 cm^{-1} (Table S2)), confirming that, differently from SB compostable plastics, PLA biodegradation and disintegration during composting occur without significant variations in its chemical composition.[11]

After the application of the selective thermal-alkaline hydrolysis method, visual inspection (Figure S2) and FT-IR analyses (Figure S3) of the residual fragments confirmed the hydrolysis of compostable plastics and the resistance of plastic residues to the alkaline treatment. Interestingly, the differences in the FT-IR spectra of plastic residues pre- and post-NaOH treatment might be ascribed to the removal of organic impurities from the plastic fragments. This hypothesis was supported by the reduction of the absorbance intensity at wavelengths usually assigned to organic matter, i.e. carbohydrates (3400−3200 and 1050 cm^{-1}) and fats (3000−2850 and 1750 cm^{-1}), in the spectrum of plastic residues after NaOH treatment (Figure S3). Furthermore, when washed with a strong anionic surfactant like sodium lauryl sulfate (SLS) (1% w/v, 60 °C, 2 h under continuous stirring), plastic fragments showed a spectrum close to the spectrum after NaOH treatment, confirming the organic nature of the impurities detected on plastic fragments recovered from compost samples.

3.3. Recovery Efficiency

Recovery tests were employed to assess how much of a known amount of analyte (i.e., plastics and compostable plastics) can be accurately retrieved by the analytical method. A high recovery efficiency indicates that the method not only is precise but also minimizes matrix effects or losses during the analytical procedure.

Figure 4 reports the comparison between theoretical and experimental results of plastic and compostable plastic determination in spiked compost samples, while detailed results are presented in Table S9. The theoretical total percentage of plastics and compostable plastics varied across the compost and replicates, ranging from 0.06% (compost #4, replicate 1, and compost #5, replicate 1) to 0.84% (compost #1, replicates 1 and 3). The experimental data consistently showed values very close to the theoretical data for both plastic and compostable plastic percentages, as well as the total percentage. The overall mean recovery efficiency across all replicates and composts was 98 ± 2%, indicating high accuracy and precision of the method. Furthermore, these results proved that the procedure did not lead to substantial losses of either material during the experimental procedure (i.e., washing, drying, or weighing steps). The accuracy of the method was further underscored by the close agreement between the theoretical and experimental concentrations of both plastic and compostable plastic fractions across all samples. This accuracy might be crucial for the method's application in real-world scenarios, where accurate quantification of these materials is necessary for compliance with regulations or for assessing the effectiveness of composting processes. For instance, the ability to accurately quantify even low theoretical percentages of compostable plastics (e.g., 0.05% in compost #3, replicate 3, Table S9) demonstrated the method's sensitivity for detecting trace amounts.

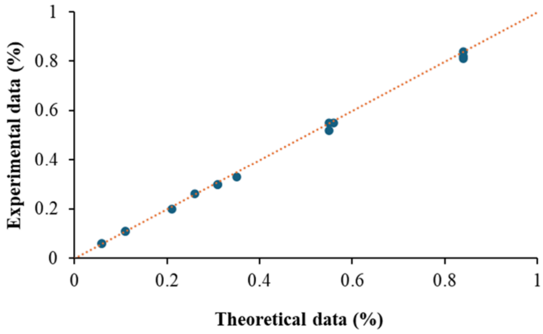

Figure 4. Validation of the method through recovery tests: comparison between theoretical and experimental (plastic + compostable plastics) contents (n = 15). The orange line represents the 1:1 relationship. Data closely following the 1:1 line demonstrate high recovery accuracy for both material types.

Summarizing, recovery tests confirmed the method's reliability across a 0−1% w/w concentration range, with a recovery efficiency of 98%.

3.4. Impurities in the Analyzed Composts

Table 3 reports the impurity (i.e., lithoids, glass, metals, and plastic-like materials) contents of five different compost samples produced from OFMSW, expressed as the percentage on a dw basis, along with relevant limit values.[12,20] Although compostable plastics represent a valid constituent of OFMSW, they have been included in this counting since plastic-like materials are currently not discriminated by relevant legislations.

Four out of five compost samples consistently met the regulatory limits for various impurities, while compost #3 exceeded the glass limit. Specifically, lithoid materials (>5 mm) ranged from 0% (compost #4 and #5) to 1% (compost #3), with all samples falling below the 5% limit. The glass content ranged from 0.03% (compost #2) to 0.95% (compost #3). Metal impurities were either absent or present in very low concentrations (up to 0.04% in compost #2), meeting the 0.3% limit. As previously reported in Section 3.2, compostable plastics were detected in small amounts in compost, and only compost #2 showed a concentration of compostable plastic residues >0.01% (i.e., 0.02%). The plastic residue content (>2 mm) across all compost samples ranged from 0.01% (compost #4 and #5) to 0.16% (compost #1). These values were below the current EU regulatory limit of 0.3%. The total impurity content, encompassing all measured fractions (>2 mm), ranged from 0.1% (compost #2 and #4) to 1% (compost #3), with four out of five samples adhering to the 0.5% limit, except for compost #3.

While the plastic concentrations obtained in this study were below the current normative limits, it is imperative to consider the long-term implications of even these seemingly low levels when compost is regularly applied to agricultural soils. First, the current EU Fertilizers Regulation 1009/2019 sets a limit of 0.3% for plastics, with future restrictions planned, lowering the limit to 0.25% starting from 2026 and further restrictions from 2029.[12] Furthermore, considering that plastics are expected to degrade in the soil environment in hundreds to thousands of years,[6] the threat of plastic residue accumulation from compost applications should be carefully evaluated, even when considering low concentrations of plastic residues as those found in this study. To illustrate the potential impact, a hypothetical scenario in which compost is applied at a typical rate of 20 Mg dw ha^{-1} year^{-1} was considered. With this compost application rate, even at the lowest observed plastic concentration of 0.01% (compost #4 and #5), this translates to

Table 3. Impurities in the Five Composts from OFMSW and Comparison with Limit Values Established by the EU Fertilizers Regulation 1009/2019[e]

	>5 mm	>2 mm					
compost	lithoids (%)	glass (%)	metals (%)	plastics (%)	compostable plastics (%)	total (%)	total-compostable plastics (%)
#1	0.75 (0.29)	0.1 (0.08)	0 (0)	0.16 (0.02)	0 (0)	0.26 (0.07)	0.26 (0.07)
#2	0.28 (0.04)	0.03 (0)	0.04 (0.06)	0.04 (0.02)	0.02 (0)	0.11 (0.03)	0.09 (0.02)
#3	1 (0.24)	0.95 (0.3)	0 (0)	0.05 (0.01)	0 (0)	1 (0.35)	1 (0.35)
#4	0 (0)	0.1 (0.02)	0 (0)	0.01 (0.01)	0 (0)	0.1 (0.03)	0.1 (0.03)
#5	0 (0)	0.14 (0.06)	0 (0)	0.01 (0)	0 (0)	0.15 (0.06)	0.15 (0.06)
limit values[a]	5[b]	0.3	0.3	0.3 (0.25[c])	n.a.[d]	0.5	n.a.

[a] EU Fertilizers Regulation 1009/2019. [b] Italian Legislative Decree 75/2010. [c] Starting from 2026, the plastic impurity limit is lowered to 0.25%; starting from 2029, this limit will be subjected to further restrictions. [d] Not applicable. [e] Mean value (SD), *n* = 3. Data are expressed on a dry weight basis.

2 kg of plastics being introduced into the soil per hectare annually. Considering the highest observed concentration of plastics of 0.16% (compost #1), this figure rises to 32 kg of plastics per hectare per year. In one hundred years of compost application, this would result in 200−3,200 kg of plastics being introduced and accumulated into the soil. Considering the results of this study (Table S7), it can also be estimated that annually, up to 830,000 fragments of plastics can be introduced per hectare of soil, and this value increases to more than 80 million fragments of plastics accumulated in one hundred years of compost application to the soil.

The long-term environmental consequences of such accumulation are not yet fully understood but are growing areas of concern. Microplastics in soil can alter soil physical properties, affect water retention, influence nutrient cycling, and potentially impact soil microbial communities and fauna.[29] Moreover, there is increasing evidence of the uptake of microplastics by plants, raising concerns about their potential entry into the food chain.[7] In this regard, it is conceivable that in future evaluations, policymakers will elect to further reduce the permissible plastic contamination threshold in compost after 2029. Consequently, the capacity to reliably differentiate between plastic and compostable plastic residues will become increasingly critical. Overall, despite meeting existing regulatory limits, the results of this study underscore the necessity of advancing beyond mere conformity toward a more sustainable and precautionary approach to plastic contamination in compost. In this context, the adoption of environmentally friendly alternatives, such as certified compostable plastics, represents a strategic pathway. Although elevated inputs of compostable plastics in the OFMSW stream require well-performing composting plants to avoid compostable plastic residues in compost, the intrinsic biodegradability of these materials allows them to biodegrade in soil over relatively short temporal scales (i.e., months to a few years), in stark contrast to conventional plastics, which may persist for centuries.[6,30] Furthermore, the biodegradation of compostable plastic residues is expected to contribute to the soil carbon cycle, potentially stimulating microbial activity and enhancing biochemical functioning of soils under certain conditions (i.e., climate and soil type).[31] Finally, from an environmental risk perspective, the concerns associated with the uptake and accumulation of microplastic particles are considerably mitigated in the case of microplastics derived from compostable plastics. Recent evidence indicates that the latter exhibits markedly lower ecological risks if compared to conventional microplastics, primarily due to their reduced residence time in the natural environments.[10,32]

3.5. Regulatory and Practical Implications of the Proposed Method

The analytical method developed in this study may represent a critical advancement for the monitoring and management of plastic contaminants in compost, particularly considering the increasingly stringent regulatory thresholds. As mentioned in Section 3.3, from 2026, European regulations will further reduce the allowable concentration of visible plastic contaminants in compost.[12] This tightening of thresholds places substantial pressure on composting facilities to ensure the absence of plastic residues in the final products. At the facility level, although the developed method requires several hours of reaction and drying to constant weight, it remains significantly simpler, more cost-effective, and more accessible than techniques such as spectroscopic analysis or pyrolysis−GC/MS. Therefore, despite the time requirement, the method would be feasible for industrial-scale monitoring, where high-cost instrumentation and skilled personnel are impractical.

Technically, although the method was developed using compostable plastic samples, it may also be applicable to biobased compostable plastics (i.e., bioplastics), as the biological origin of the raw material is not expected to interfere with thermal-alkaline hydrolysis.

From a regulatory standpoint, the method provides a scientific basis for revising existing compost quality standards and analytical protocols, which currently classify all visible plastic residues as contaminants, regardless of their biodegradability. Incorporating such a method into official testing frameworks would allow for a more accurate and fair assessment of compost contamination, preventing misclassification of biodegradable materials as pollutants. This, in turn, would support compost producers in complying with contamination limits while promoting the use of certified compostable plastics as part of sustainable waste management strategies.

Moreover, the method may potentially be applied to other complex organic matrices, such as digestate from the anaerobic digestion of OFMSW, extending its relevance beyond composting. As anaerobic digestion is increasingly integrated into municipal biowaste treatment chains,[13] especially within urban circular bioeconomy contexts, reliable differentiation of compostable plastics becomes equally important to ensure digestate quality and compliance with land application criteria.

A further and potentially transformative implication of this method lies in its utility for establishing facility-level mass balance frameworks for compostable plastics. Current approaches to assessing the fate of compostable plastics in industrial composting are constrained by the lack of analytical tools capable of tracking biodegradable polymers through heterogeneous waste matrices. The method proposed in this study could enable the quantitative tracking of compostable plastics through pretreatment, biological treatment, and post-treatment phases, by adapting its principles to more complex organic substrates. This could support more accurate assessments of degradation efficiency, facilitate benchmarking across technologies or facilities, and help identify process bottlenecks, such as the frequent removal and unintended disposal of compostable plastics alongside conventional plastics during mechanical pretreatment. Such practices undermine the environmental rationale for using compostable plastics and contribute negatively to the organic recovery rates. Implementing this method within composting facility management could enhance transparency, traceability, and performance monitoring in biowaste systems, ultimately supporting the transition to more sustainable materials and waste treatment paradigms.

4. CONCLUSIONS

This study established a selective thermal-alkaline hydrolysis protocol as a robust analytical framework for the differentiation of conventional plastics and compostable plastics in compost. The method's selectivity, high recovery efficiency, and consistency with spectroscopic results addressed a key analytical limitation in the current monitoring of plastic contaminants in biowaste streams.

Beyond its immediate analytical performance, the method has significant implications for both regulatory enforcement and operational practice. As composting facilities processing the OFMSW are increasingly subject to stringent compost quality criteria, the capacity to distinguish between persistent plastic pollutants and biodegradable alternatives is essential to enable fair compliance assessments. In this context, the method offers a foundation for updating official analytical protocols and aligning regulatory frameworks with the evolving landscape of compostable plastic use and disposal. Furthermore, the approach presents opportunities for integration into standardized monitoring frameworks and quality assurance protocols across biowaste recovery infrastructures.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acssuschemeng.6c01629.

> Literature review for compostable plastic and plastic separation; FT-IR diagnostic peaks for polymer identification; characteristics of the composting plants; tested conditions for method optimization; experimental design of recovery tests; detailed analytical methods; compostable plastic and plastic separation in compost; number of fragments retrieved in compost; results of recovery tests; FT-IR spectra and visual inspection of selected samples from method optimization; visual inspection of selected samples from method validation; FT-IR spectra of selected samples from method validation (PDF)

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M.C.: conceptualization, methodologies, validation, formal analysis, investigation, data curation, writing-original draft, supervision, and project administration. F.A.: writing-review and editing.

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■ ABBREVIATIONS

dw, dry weight; HDPE, high-density polyethylene; FT-IR, Fourier transform infrared spectroscopy; GW, green waste; LDPE, low-density polyethylene; OFMSW, organic fraction of municipal solid waste; PET, polyethylene terephthalate; PLA, polylactic acid; PP, polypropylene; PS, polystyrene; PVC, polyvinyl chloride; SB, starch-based; v/v, volume/volume; w/v, weight/volume

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