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The Use of New Parameters to Optimize the Composting Process of Different Organic Wastes

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Abstract: The correct development of the composting process is essential to obtain a product of high value from organic wastes. Nowadays, some composting mixture parameters (i.e., air-filled porosity, moisture and the C/N ratio) are used to optimize the composting process, but their suitability is still debated because the literature reports contrasting results. This paper aimed to find other parameters that control the correct development of composting. The relationship between these and the compost quality was then verified. Twelve different composting mixtures were prepared using different organic wastes and bulking agents and were aerobically treated in a 300 L composter. The physico-chemical and chemical parameters of initial mixtures were analyzed, with particular regard to the total and water-extractable forms of organic C and N and their ratios and correlated with the temperature measured during composting. A positive correlation between temperature parameters during the active phase and soluble forms of N in the initial mixtures was found. A high total organic C to soluble N ratio in the composting mixtures was correlated with the low quality of the compost produced. Based on the results, a minimum content of WEN (water-extractable N) (0.4% *w/w*) or a TOC/WEN (total organic C/WEN) ratio in the range of 40–80 was recommended to ensure the correct development of the process and to produce compost of high quality.



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1. Introduction

Municipal, agro-industrial and livestock activities generate large volumes of organic wastes, whose management can produce adverse effects on the environment (soil, water and atmosphere pollution). Composting represents a suitable and environmentally friendly disposal strategy for organic waste management. Composting aerobically degrades organic wastes to compost and two main by-products, heat and carbon dioxide [1,2]. Composting is a self-heating process that proceeds through three main steps: (1) The mesophilic phase (25–40 °C), (2) the thermophilic phase (55–65 °C) and (3) the curing phase. During composting, labile organic matter is mineralized and complex recalcitrant materials tend to concentrate, increasing the organic matter stabilization in the compost [3]. Compost is a nutrient-rich organic amendment able to provide N, P, K and organic matter to the soil, also contributing to soil C sequestration [2,4].

Composting has several advantages, such as (1) sanification from pathogens and weed seeds, (2) volume and odor reduction, (3) microbial stabilization, (4) disposal cost reduction and (5) the production of organic fertilizer (compost) with an economical added value, that can be used to replace chemical fertilizers when characterized by high-quality [5,6].

Conversely, gas emissions, large area requirements and net energy consumption were reported to be the main disadvantages of composting [6–8].

The correct development of composting throughout its active and curing phase is essential to obtain high-quality compost. During the active phase, the temperature of the biomass is one of the most significant parameters for evaluating process effectiveness, as it depends on the aerobic nature of the process. In addition, compost sanitation and biodegradation rates rely on temperature changes during composting, and Cáceres et al. introduced the degree-hour concept to composting to optimize the exposure of feedstock to high and intermediate temperatures throughout the entire process [9]. An effective composting process also allows for a high degree of stabilization and maturity [10]. Stability refers to the degree of organic matter decomposition, whereas maturity refers to the removal of phytotoxic compounds from the composting feedstock. Compost stability and maturity can be assessed using different parameters, as reviewed by Bernal et al. [5]. Stability can be evaluated by using respirometric methods or by studying organic matter transformations during composting. Maturity can be assessed using physical, chemical (C/N ratio, pH, electrical conductivity, humification indices) or biological (phytotoxicity) criteria [5].

Different organic materials (e.g., agricultural and agro-industry by-products, livestock wastes, sewage sludge, organic fraction of the municipal solid wastes) can be treated through composting and they are generally mixed with bulking materials to ensure the optimal conditions for microbial growth and process development [4]. Mixture preparation is essential for balancing moisture, pH and the C/N ratio for adequate aeration and microbial growth [11]. Although mixture formulation is commonly carried out to adjust the C/N ratio and moisture in their ideal range (20–25 and 40–60%, respectively), other physical parameters (e.g., porosity, filled air space) are important in composting mixtures [12–14]. In addition, composting may be performed at low or high C/N ratios (<20 and >30) as total C and total N may include recalcitrant fractions depending on the chemical characterization of organic material [1], e.g., fiber content [15]. Puyuelo et al. demonstrated that the biodegradable C/N ratio was significantly different from the C/N ratio in several organic wastes, and pointed out that the C/N ratio used to optimize biological treatments should be defined in biodegradable terms [16]. This is in accordance with Trémier et al. who affirmed that mixture formulation must consider the biodegradability of wastes rather than other parameters [14]. As a consequence, it is often reported in the literature that composting mixtures characterized by the optimal C/N ratio did not show correct process development, resulting in a low-temperature profile and/or low-quality composts [11,17,18].

Since biochemical transformations of organic matter during composting occur in the aqueous phase, mixture formulation based on water-extractable C and N, and their ratio, appear to be appropriate. As demonstrated by Said-Pullicino et al., the water-soluble organic matter is the most accessible fraction of organic matter for the microorganisms [19], and this can be useful for monitoring the behavior of the composting process. Nevertheless, there are no studies focused on the relationship between these two parameters (water-extractable C and water-extractable N) and the evolution of the process. In this context, the present study aimed to assess chemical parameters related to C and N for the formulation of composting mixtures. The selected parameters were related to both the development of composting and to the quality (stabilization and maturity) of the produced compost.

2. Materials and Methods

2.1. Organic Materials

Table 1 shows the organic materials used in the present study and their main chemical characteristics. The composting experiments were performed in order to reuse different organic putrescible materials as the organic fraction of the municipal solid waste, pig slurry, sewage sludge and olive mill wastes. Even digestates, derived from different anaerobic trials, were used as substrates for the composting process (anaerobically digested, AFB1-contaminated chopped corn and anaerobically digested pharmaceutical wastewater). All these organic putrescible materials were co-composted with bulking agents, in order

to establish the recipe structure of starting mixtures, exposed particle surface area and porosity [14] for ensuring the optimal development of composting, and hence the quality of the final compost. In this study, the bulking agents were wood chips, (WC, particle size < 20 mm), broadleaf tree pruning (BLTP), conifer tree pruning (CTP) and chopped-tree pruning (particle size < 50 mm). The use of tree pruning at different particle sizes was considered for balancing the surface area for microbial biodegradation and, hence, to optimize the porosity [5]. Cereal straw (CS) was added as an absorbent agent, when appropriate.

Table 1. Main characteristics of raw materials and bulking agents used in the composting experiments.

Type	Acronym	Description	Moisture (%)	TOC (%)	TKN (%)	pH	Bulk Density (kg L ⁻¹)
Raw wastes	OFMSW	Organic fraction of the municipal solid wastes	69.7 ± 0.2	22.2 ± 0.1	1.3 ± 0.1	3.2 ± 0.0	0.8 ± 0.1
	PS	Pig slurry	93.4 ± 0.6	40.2 ± 0.3	3.7 ± 0.2	7.3 ± 0.1	1.0 ± 0.0
	CC	AFB1 contaminated chopped corn	14.0 ± 0.1	37.5 ± 0.0	1.5 ± 0.1	6.1 ± 0.1	0.4 ± 0.1
	AD-CC	Anaerobically digested AFB1 contaminated chopped corn	93.8 ± 0.1	21.8 ± 0.1	6.3 ± 0.3	7.5 ± 0.0	0.9 ± 0.1
	OMW	Olive mill wastes	65.1 ± 1.2	39.9 ± 0.5	0.9 ± 0.2	5.9 ± 0.0	0.8 ± 0.1
	SS	Sewage sludge	89.9 ± 0.3	18.7 ± 0.6	3.8 ± 0.1	8.0 ± 0.1	0.8 ± 0.1
	AD-PW	Anaerobically digested pharmaceutical wastewater	96.2 ± 0.2	41.4 ± 0.2	9.4 ± 0.4	7.4 ± 0.2	0.9 ± 0.0
Bulking agents	CS	Cereal straw	14.2 ± 0.0	43.2 ± 0.2	0.7 ± 0.0	6.7 ± 0.0	0.1 ± 0.0
	WC	Wood chips	57.2 ± 0.9	42.1 ± 0.2	0.6 ± 0.1	6.8 ± 0.1	0.4 ± 0.1
	BLTP	Broadleaf tree pruning	6.1 ± 0.0	34.4 ± 0.0	0.8 ± 0.1	5.7 ± 0.1	0.2 ± 0.0
	C-BLTP	Chopped broadleaf tree pruning	6.9 ± 0.0	33.9 ± 0.5	1.0 ± 0.2	6.1 ± 0.0	0.2 ± 0.0
	CTP	Conifer tree pruning	26.5 ± 0.5	39.6 ± 0.5	0.6 ± 0.1	6.8 ± 0.0	0.3 ± 0.1
	C-CTP	Chopped conifer tree pruning	24.8 ± 0.3	38.7 ± 0.7	0.7 ± 0.1	6.8 ± 0.1	0.3 ± 0.0

Mean value ± SEM, n = 3; TOC: Total organic C; TKN: Total Kjeldahl N, AFB1: Aflatoxin B1. Data are expressed on dry basis.

Raw materials and bulking and/or absorbent agents were collected in the area of Perugia (Umbria Region, Central Italy). Pig slurry, olive mill wastes and cereal straw were obtained from local farmers, whereas organic fractions of municipal solid wastes and sewage sludge were obtained from local companies that collect and treat wastes or wastewater, respectively. Digestates were obtained from pilot anaerobic digesters treating aflatoxin B1 (AFB1)-contaminated corn or pharmaceutical wastewaters. Bulking and absorbent materials were collected from a local plant nursery.

Representative samples of all organic materials were collected and portioned in two fractions. The first one was stored at 4 °C before the beginning of the experiments, while the second one was dried, crushed, sieved (<0.5 mm) and mixed for analytical determinations.

2.2. Composting Experiments

Twelve different composting mixtures were studied in this work to assess new parameters suitable for mixture formulation and composting optimization. The parameters chosen were used to explain both the progress of composting and the quality of the final compost. The five mixtures were those described in Cucina et al. [20], Cucina et al. [3] and Tacconi et al. [21], and different compositions were included to ensure that the studied parameters described in the following paragraphs could be suitable for a broad type of mixtures. Bulking and/or absorbent agents were mixed with raw materials to maximize the amount of organic waste treated and to adjust the physical properties (bulk density, air-filled porosity and moisture content), avoid water leaching and ensure optimal air circulation. The C/N ratio in the mixtures was not adjusted to the optimal values, since it was one of the parameters selected to be evaluated for its relationship with composting development and final compost quality. Based on these considerations, mixtures were prepared according to the proportions (fresh weight basis) reported in Table 2. Mixtures were placed in experimental plastic composters (0.6 m × 0.6 m × 0.8 m = 0.288 m³) at outdoor conditions. All the experiments were performed through static composting, and passive aeration was maintained thanks to the porosity of the mixture itself. The temperature was

measured once a day (same time of the day) in the center of the pile using temperature probes (Stainless Steel Temperature Probe, Vernier, Beaverton, OR, USA).

Table 2. Mixture compositions.

Mixture	Composition (% <i>w/w</i>)
1 ^a	43% OFMSW + 43% WC + 14% CC
2 ^a	40% PS + 40% WC + 12% CC + 8% CS
3	42% AD-CC + 54% WC + 4% CS
4	55% PS + 45% BLTP
5	55% PS + 45% CTP
6	55% PS + 45% C-BLTP
7	55% PS + 45% C-CTP
8	80% OMW + 20% C-BLTP
9	80% OMW + 20% C-CTP
10 ^b	70% SS + 30% WC
11 ^b	45% SS + 45% WC + 10% CS
12 ^c	50% AD-PW + 40% WC + 10% CS

^a [21]; ^b [3]; ^c [20]. OFMSW: Organic fraction of the municipal solid wastes, CC: AFB1-contaminated chopped corn, PS: Pig slurry, AD-CC: Anaerobically digested AFB1-contaminated chopped corn, OMW: Olive mill wastes, SS: Sewage sludge, AD-PW: Anaerobically digested pharmaceutical wastewater, CS: Cereal straw, WC: Wood chips, BLTP: Broadleaf tree pruning, CTP: Conifer tree pruning, C-BLTP: Chopped broadleaf tree pruning, C-CTP: Chopped conifer tree pruning.

The intensity of the composting process in the active phase was evaluated by calculating the time that the mixture temperature was higher than 55 °C ($h > 55$ °C) and the cumulative degree hours (DH), as described by Cáceres et al. [9]. The active phase of composting was considered completed when a stable temperature was reached (about 30 days). At the end of the active phase, the mixtures were sieved to 20 mm, placed in open boxes without aeration and mixed once a week for 60 days (curing phase). Representative samples of the initial mixtures and mature compost were collected and portioned in two fractions: The first one was stored at 4 °C for the determination of water-extractable C and N, while the second one was dried, crushed, sieved (<0.5 mm) and mixed for analytical determinations.

2.3. Analytical Methods

Moisture was determined on fresh samples by drying at 105 °C to constant weight. Total organic carbon (TOC), total Kjeldahl-N (TKN) and ammonium-N were analyzed according to standard methods [22]. pH and electrical conductivity were analyzed after water extraction of fresh samples (solid to water ratio of 1:10 *w/w*) using a glass electrode (pH-Meter Basic 20+, Crison Instruments, Barcelona, Spain) and a conductivity cell (Ec-Meter Basic 30+, Crison Instruments, Barcelona, Spain), respectively. Bulk density was determined following standard procedures [23], and air-filled porosity (AFP) was calculated from the wet bulk density as suggested by Albuquerque et al. [24]. Organic matter (OM) loss was calculated as described by Gigliotti et al. [25]. Humic-like substances were extracted and purified as described by Ciavatta et al. [26] and C quantification in the extracts was carried out using high-temperature combustion (805 °C, Pt catalyzed) followed by CO₂ infrared detection (Analyzer multi N/C 2100S, Analytic Jena, Überlingen, Germany). A germination assay employing cress seeds was used for the determination of potential phytotoxicity in mature compost [20].

Water-extractable organic C (WEOC) and N (WEN) were extracted from fresh samples (solid to water ratio 1:10 *w/w*) on a horizontal shaker for 24 h at room temperature. The suspensions were then centrifuged at 8000 rpm for 12 min and filtered (0.45 µm). Extracts were analyzed for organic C and N content using Pt-catalyzed, high-temperature combustion (805 °C) followed by infrared detection of CO₂ and chemiluminescence detection of NO. Water-extractable organic N (WEON) was determined by taking the difference between WEN and ammonium-N.

2.4. Statistics

Results are the arithmetic mean of three replicates, and the standard error of the mean (SEM) was reported along with the average value (Microsoft Excel Software). Bivariate correlation tests (Pearson's linear correlation coefficient) were used to study the associations between composting development and compost quality and the selected parameters of the mixtures (Microsoft Excel Software) (significance of $p < 0.01$ and $p < 0.05$, $n = 12$). Data passed the requisite tests for running a linear regression (plot of the data reveals a linear relationship, residuals are statistically independent, residuals are homoscedastic and residuals are unbiased).

3. Results and Discussion

3.1. Organic Materials Characteristics and Temperature Parameters

In the present work, the main characteristics of all raw materials used in the study (Table 1) were related to the correct development of the active phase of composting.

The correct physical structure of composting mixtures permits the oxygen availability for microorganisms and is essential for mineralization phenomena, and the oxygen availability mostly depends on the bulk density and moisture of the starting mixture [14,27]. Concerning the bulk density, the lowest values were observed, as expected, for the lignocellulosic materials (WC, BLTP, CTP, CS), and the chopped tree pruning did not increase this parameter. Among the bulking agents, the WC and the CTP were characterized by the highest values of the C/N ratio (70.2 and 66.0, respectively) and were used in the trials in a range from 20 to 58% *w/w*. In a study carried out by Barrena et al. [12], it was observed that the raw pruning wastes are also characterized by a high C/N ratio (42 and 52), and the semi-composted pruning resulted in a similar C/N ratio. All the raw wastes studied in the present work were characterized by a lower C/N ratio than the lignocellulosic materials, and this was particularly true for both digestate and sewage sludge, which showed values < 5 . It is important to know the C/N ratio because it represents an indicator of nutritional balance for microbial activity [5]. In this case, the use of a large amount of pig slurry, anaerobic digestate and sewage sludge can result in a C/N ratio lower than 25–35, which is considered the optimal value for the initial mixture, causing an excess of N per degradable C [5]. Moreover, it is important to consider the optimal value of moisture (50–65%), because water content along with the bulk density can affect the aeration and microbial O₂ supply [14]. Even in this case, the pig slurry, anaerobic digestate and sewage sludge were the organic materials that showed the highest values of moisture (>90%). This suggests that the adequate selection of bulking agents for both objectives is important to increase the degradable C and improve the physical characteristics of the starting mixtures.

To evaluate the effect of using different raw materials and bulking agents on the evolution of the composting process, temperature parameters were assessed (Figure 1, Table 3). The highest temperatures values (>55 °C) were observed in the mixtures where wood chips were used as a bulking agent (mixtures 1, 2, 3, 10, 11 and 12), and when cereal straw was added as absorbent material (mixtures 2, 3, 11 and 12). These mixtures were also characterized by the highest cumulative DH, suggesting the correct development and intensity of the composting processes, especially when about 40% of wood chips were used. During the active phase of composting, a temperature of 52–60 °C is considered the most favorable, during which mesophilic and thermophilic microorganisms firstly degrade sugars, amino acids and proteins (the mesophilic phase), and afterwards, fats, cellulose, hemicellulose and lignin (the thermophilic phase) [5,28].

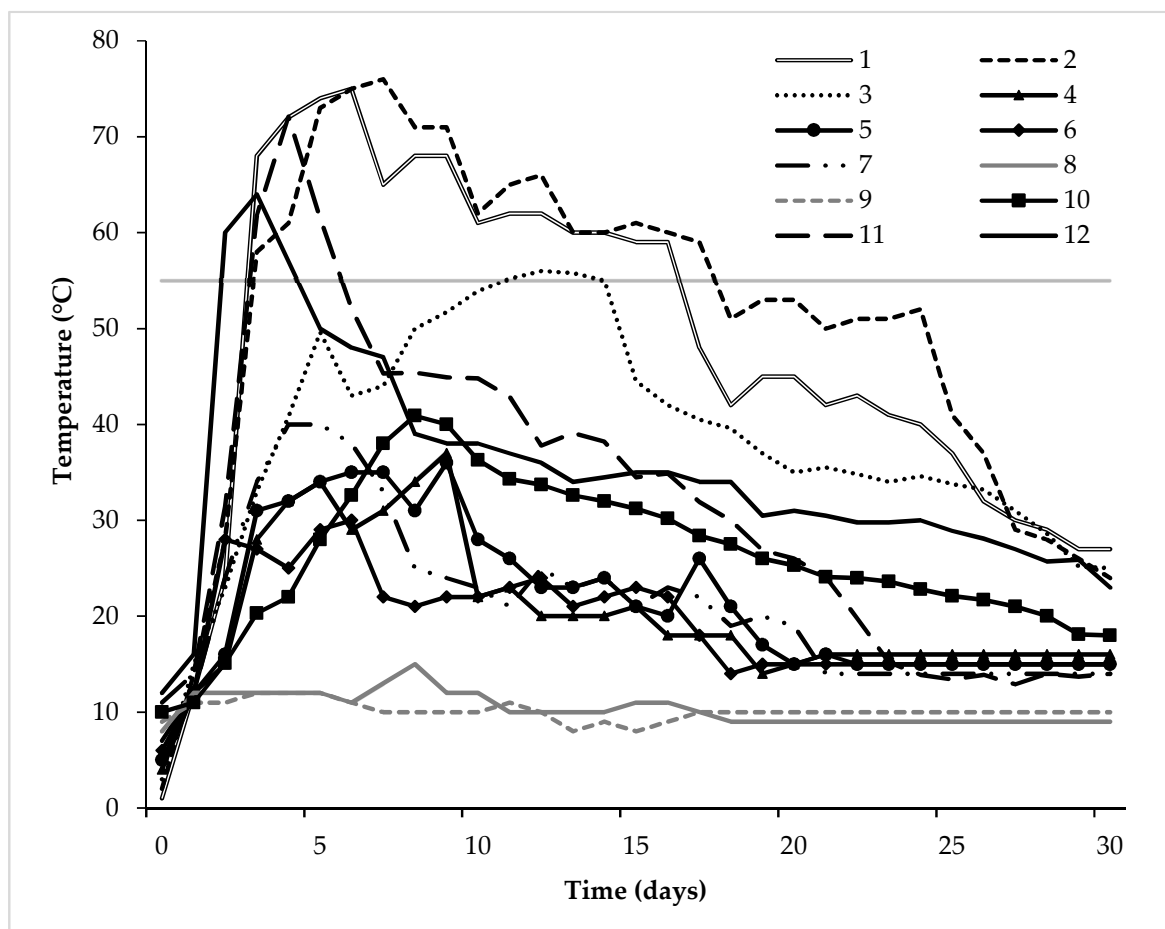


Figure 1. Temperature profiles during the active phase of composting. Horizontal line indicates 55 °C (1–2: [21]; 10–11: [3]; 12: [3]).

Table 3. Temperature parameters during composting.

Mixture	T Max	h > 55 °C	Cumulative DH
1	72	312	36,048
2	74	336	38,160
3	56	96	28,855
4	37	0	15,288
5	36	0	16,128
6	30	0	14,400
7	40	0	15,936
8	12	0	7704
9	12	0	7536
10	41	0	19,586
11	67	96	23,614
12	64	72	26,369

T max: Maximum temperature reached in the active phase; h > 55 °C: Time when the mixture temperature was higher than 55 °C; Cumulative DH: Cumulative degree hours in the active phase.

The other mixtures (from 4 to 9) were prepared with the conifer and broadleaf tree pruning, before and after being chopped, to investigate the effect of different particle sizes on the composting process. In all these mixtures, temperature values >55 °C were not achieved, and this was particularly evident in mixtures 8 and 9 where the olive mill wastes were treated with both types of chipped tree pruning. The high amount of olive mill wastes used in the mixtures resulted in an unbalanced C/N ratio with an excess of C. With this regard, Gigliotti et al. observed a low degradation of organic matter during the composting

of husks, probably due to the presence of relatively stable compounds (lipids, polyphenols and pectin) [25]. Conversely, when the pig slurry was co-composted with the tree pruning, the mixture achieved a higher temperature with respect to the olive mill wastes, with an average maximum value of 36 °C, probably due to the chemical composition of pig slurry, as discussed in the following paragraphs.

3.2. Physico-Chemical Parameters of the Mixtures and Temperature Evolution

The mixtures were analyzed for their main physico-chemical characteristics and results are reported in Table 4. Statistical analysis showed that no significant linear relationship existed between the physico-chemical parameters analyzed and the temperature parameters reported in Table 3.

Table 4. Physico-chemical characteristics of the mixtures.

Mixture	Moisture (%)	Volatile Solids (%)	pH	EC (dS m ⁻¹)	AFP (%)
1 ^a	67 ± 1	73.5 ± 0.1	3.3 ± 0.0	3.3 ± 0.1	53.7
2 ^a	55 ± 1	65.2 ± 0.0	7.3 ± 0.1	1.3 ± 0.1	53.7
3	57 ± 0	86.3 ± 0.0	6.8 ± 0.1	2.5 ± 0.2	44.7
4	57 ± 0	71.0 ± 0.3	8.4 ± 0.1	0.7 ± 0.0	70.8
5	62 ± 0	79.5 ± 0.8	8.3 ± 0.1	0.7 ± 0.0	67.2
6	70 ± 2	72.4 ± 0.7	8.3 ± 0.2	0.8 ± 0.1	56.4
7	64 ± 1	76.8 ± 0.3	8.2 ± 0.1	0.9 ± 0.0	60.9
8	60 ± 1	86.9 ± 0.4	5.9 ± 0.0	2.4 ± 0.2	34.8
9	60 ± 1	89.1 ± 1.2	6.1 ± 0.0	2.1 ± 0.1	46.5
10 ^b	68 ± 1	77.6 ± 0.8	8.2 ± 0.0	2.1 ± 0.1	47.4
11 ^b	62 ± 0	82.1 ± 0.2	7.9 ± 0.1	1.2 ± 0.1	56.4
12 ^c	65 ± 0	69.1 ± 0.3	8.2 ± 0.1	1.5 ± 0.0	51.9

^a [21]; ^b [3]; ^c [20]; Mean value ± SEM, n = 3; EC: Electrical conductivity, AFP: Air-filled porosity. Data are expressed on dry basis except for pH and EC.

The moisture values ranged from a minimum of 55 to a maximum of 68% in the mixtures where pig slurry (mixture 2) and sewage sludge were processed (mixture 10), respectively, values close to the optimal range (50–65%). This difference was attributed to a large amount of sewage sludge (70%) treated in mixture 10, and also probably to the lack of absorbent materials used, such as the cereal straw. The increase in temperature is related to a variety of factors (e.g., the percentage of porosity, as described by AFP). AFP should be in the range of 35–50% to ensure optimal air circulation [24]. Although mixture 2 showed a slightly higher value of AFP with respect to the optimal range, the correct temperature evolution was evidently not influenced (Table 3, Figure 1). These results suggest that the behavior of the active phase is not strictly dependent on the moisture and porosity of the mixture, especially if both parameters are close to the optimal values. Concerning the volatile solids, the lowest value was observed for mixture 2, suggesting that this parameter did not affect the temperature behavior during the thermophilic phase. This evidence can be supported by the results obtained for mixtures 8 and 9 that, although showing a high content of volatile solids, did not exceed the ambient temperature during the active phase. The analysis of the degradable fraction of organic matter, in terms of C and N content, might be a more suitable parameter to predict the behavior of the thermophilic phase. In a similar experiment in which husks were composted, Gigliotti et al. observed the correct behavior of the active phase to be associated with the biodegradation of labile organic compounds of organic matter, i.e., the water-soluble organic matter [25]. This fraction is easily available for microorganisms and measured in terms of WEOC and was then proposed as a good indicator of the evolution of the process [15,19,25]. This aspect is of particular relevance especially for choosing the bulking agents, which besides having the role of optimizing the physical structure of the mixture, in addition, they should also have a high concentration of low degradable fiber. In this study, the use of tree pruning did not improve the structure of mixtures, as observed by the highest AFP values when these were

not chopped and co-composted with pig slurry (mixtures 4 and 5). The attempt to use the chopped tree pruning was useful to decrease the AFP in pig slurry mixtures (mixtures 6 and 7), but the effect of reducing the particle size was more evident when olive mill wastes were treated (mixtures 8 and 9). Hence, it can be stated that the use of chopped tree pruning improves aeration, which represents a key factor for composting [5]. Even if the physical structure was optimized, mixtures 6, 7, 8 and 9 did not achieve the optimal values of temperatures for the active phase of composting. This suggests, once again, that bulk density and AFP are not the only parameters that affect the increase in temperature, but rather the chemical composition of the bulking agents also plays an important role.

With regards to the pH (Table 4), the values ranged from 3.3. to 8.4 (for mixtures 1 and 4, respectively) and these values seem to be outside the optimal range (6.5 to 8.0) for the initial stage of composting, even if a larger pH range may be allowed for ensuring the correct evolution of the process [4], as also demonstrated by our data (Figure 1, Table 4). The results of EC on the starting mixtures showed values in a range of 0.7–3.3 (for mixtures 5 and 1, respectively). By observing the EC results and the temperature profile (Figure 1) of both mixtures 5 and 1, it is possible to also suggest that this parameter did not affect the temperature evolution. It can be useful to quantify the level of soluble salts (EC determination) in order to evaluate possible phytotoxic effects on seed germination or activity of roots using the compost in the agronomic substrates [29].

3.3. Total and Soluble C and N and Temperature Evolution

As previously discussed, physico-chemical parameters of the mixtures were not significantly related to the correct progress of composting. Therefore, other parameters of the mixtures were assessed in this work and related to the temperature profiles during composting. Results of the determination of total organic C (TOC), total Kjeldahl N (TKN), ammonium-N, water-extractable organic C (WEOC), water-extractable N (WEN), water-extractable organic N (WEON) and their ratios were reported in Table 5.

Both TOC and TKN varied in large ranges (from 26.1 to 44.1% and from 0.7 to 2.1% for TOC and TKN, respectively). The large variability of these two parameters was mainly related to the use of different organic wastes. For instance, mixtures 8 and 9 showed the largest amount of TOC and the lowest amount of TKN, and it was mainly related to the large use of olive mill waste in the mixtures, which is known to be rich in organic C and poor in N [30]. Mixture 10, which was mainly composed of sewage sludge (Table 1), showed the lowest TOC and the highest TKN within the tested mixtures, values similar to the composting mixture analyzed in Şevik et al. [31]. Due to the large variability of TOC and TKN, the C/N ratio of the studied mixtures also varied from a minimum value of 14.1 (mixture 10) to a maximum value of 61.7 (mixture 8), and only two mixtures (6 and 7) showed a C/N ratio within the range recommended by Reyes-Torres et al. (25–30) [32].

Ammonium-N content in the mixtures varied according to their composition, and it was higher in the mixtures containing the organic fraction of municipal solid wastes and pig slurry with respect to the mixtures containing olive mill wastes and sewage sludge. Indeed, these last two organic wastes are usually characterized by a low concentration of ammonium-N [33]. Consequently, the TOC/ammonium-N ratio ranged from a maximum value of 882 (mixture 9) to a minimum value of 167 (mixture 1).

Table 5. Total organic C, total Kjeldahl N, ammonium-N, water-extractable C, N and organic N and their ratios in the mixtures.

Parameters	Mixtures											
	1	2	3	4	5	6	7	8	9	10	11	12
TOC (%)	33.4 ± 0.1	26.1 ± 0.1	40.5 ± 0.2	28.4 ± 0.1	31.8 ± 0.0	34.2 ± 0.0	35.9 ± 0.2	43.2 ± 0.4	44.1 ± 0.3	29.6 ± 0.6	31.2 ± 0.2	36.3 ± 0.0
TKN (%)	1.7 ± 0.1	1.2 ± 0.1	1.0 ± 0.1	1.6 ± 0.1	1.6 ± 0.0	1.3 ± 0.1	1.3 ± 0.1	0.7 ± 0.1	1.0 ± 0.2	2.1 ± 0.2	1.7 ± 0.2	1.6 ± 0.1
TOC/TKN	19.6	21.7	40.5	17.7	19.9	26.3	27.6	61.7	44.1	14.1	18.4	22.7
Amm-N (%)	0.20 ± 0.02	0.12 ± 0.01	0.10 ± 0.01	0.11 ± 0.01	0.13 ± 0.01	0.10 ± 0.00	0.12 ± 0.01	0.05 ± 0.01	0.05 ± 0.01	0.10 ± 0.02	0.06 ± 0.01	0.11 ± 0.02
TOC/Amm-N	167	217.5	405	258.2	244.6	342	299.2	864	882	296	520	330
WEOC (%)	4.00 ± 0.01	3.18 ± 0.05	4.20 ± 0.12	1.15 ± 0.05	1.24 ± 0.06	1.15 ± 0.05	1.20 ± 0.06	10.67 ± 0.24	9.36 ± 0.18	2.18 ± 0.10	2.38 ± 0.13	3.54 ± 0.04
WEOC/TKN	2.35	2.65	4.2	0.72	0.78	0.88	0.92	15.24	9.36	1.04	1.4	2.21
WEOC/Amm-N	20	26.5	42	10.5	9.5	11.5	10	213.4	187.2	21.8	39.7	32.2
WEN (%)	0.50 ± 0.04	0.43 ± 0.03	1.00 ± 0.06	0.17 ± 0.01	0.18 ± 0.01	0.18 ± 0.02	0.17 ± 0.00	0.09 ± 0.02	0.08 ± 0.01	0.25 ± 0.02	0.40 ± 0.04	0.52 ± 0.00
WEON (%)	0.30 ± 0.01	0.31 ± 0.01	0.90 ± 0.01	0.06 ± 0.00	0.05 ± 0.00	0.08 ± 0.01	0.05 ± 0.00	0.04 ± 0.01	0.03 ± 0.00	0.15 ± 0.02	0.34 ± 0.01	0.41 ± 0.02
TOC/WEN	66.8	60.7	40.5	167.1	176.7	190	211.2	480	551.3	118.4	78	69.8
WEOC/WEN	8	7.4	4.2	6.8	6.9	6.4	7.1	118.6	117	8.7	5.9	6.8
TOC/WEON	111.3	84.2	45	473.3	636	427.5	718	1080	1470	197.3	91.8	88.5
WEOC/WEON	13.3	10.3	4.7	19.2	24.8	14.4	24	266.8	312	14.5	7.0	8.6

Mean value ± SEM, n = 3; TOC: Total organic C, TKN: Total Kjeldahl N, Amm-N: Ammonium-N, WEOC: Water-extractable organic C, WEN: Water-extractable N, WEON: Water-extractable organic N. Data are expressed on dry basis.

WEOC and WEN were determined on mixtures' samples to evaluate the relationship between the soluble forms of these two elements and the evolution of composting. WEOC showed large variability between the mixtures analyzed, mainly depending on their composition. The highest values were detected for mixtures 8 and 9 (10.7 and 9.4%, respectively) and this was expected since olive mill wastes are usually rich in soluble organic matter. For instance, Gianico et al. reported that 87% of the chemical oxygen demand of olive milling residues was represented by soluble molecules [34]. Mixtures containing olive mill wastes (8 and 9) were characterized by the highest WEOC/TKN (15.2 and 9.4) and WEOC/ammonium-N (213 and 187) ratios due to their high content of soluble organic matter and low content of N. The largest amounts of WEN were detected in the mixtures containing anaerobically digested organic wastes (mixture 3 and 12). This was expected since it is well known that during anaerobic digestion, organic N is mineralized [35], leading to the formation of ammonium-N, which increases the amount of water-extractable N. With regard to WEON, mixtures 4, 5, 6, 7, 8 and 9 showed a content of WEON ten times lower than the other mixtures, and this was mainly related to the fact that even if most of the mixtures were characterized by an acceptable level of TKN (mixtures 4 to 7), it was present mainly in non-soluble forms. When the ratios between the analyzed parameters were determined (TOC/WEN, WEOC/WEN, TOC/WEON and WEOC/WEON), it was interesting to observe that the highest values of these parameters were always presented by mixtures 8 and 9 (olive mill waste mixtures). This was a consequence of the large amount of organic matter (both in total and soluble forms) and the low amount of N in the olive mill wastes. Similarly, mixtures 4, 5, 6 and 7 (pig slurry mixtures) also showed high values of TOC/WEN and TOC/WEON ratios (ranging from 167 and 211 and from 428 to 718, respectively).

The relationship between mixtures' chemical parameters shown in Table 5 and the correct development of composting in terms of temperature (Figure 1, Table 3) was then evaluated, and the results are reported in Table 6. Although the parameter $h > 55\text{ }^{\circ}\text{C}$ did not show significant correlations with the analyzed parameters, the maximum temperature and cumulative DH were found to be significantly correlated with some of the above-described parameters. The strongest and significant correlations were found between temperature parameters and WEN, WEON, TOC/WEN, TOC/ammonium-N, TOC/WEON, WEOC/WEN and WEOC/WEON ratios. Interestingly, WEN and WEON showed a positive correlation with the correct development of the active phase of composting, meaning that the presence of soluble N form positively affects the composting. Conversely, TKN was not significantly correlated with the temperature parameters. This is in accordance with Puyuelo et al. and Trémier et al. who reported that, more so than the total amount of N, it is the soluble forms of the element that should be taken into consideration to optimize composting [14,16]. This is because soluble N compounds (i.e., amino acids, small peptides and ammonium-N) are easily available forms of N that microorganisms can use for their metabolism and growth [36,37]. With respect to TOC/WEN, TOC/ammonium-N, TOC/WEON, WEOC/WEN and WEOC/WEON ratios and their relationship with temperature parameters during the active phase of composting, the correlation found in the present work was negative. This means that an elevated ratio between organic C (both in its total and soluble forms) and water-soluble N forms negatively affect the development of composting. This allows one to affirm that soluble N represents the limiting factor for the correct development of composting, with particular regard to the active phase. The TOC/TKN ratio, which is the most-used parameter for the formulation of a composting mixture, does not take into account the fact that C and N may be present in the organic wastes in recalcitrant forms that cannot be quickly degraded and mineralized by composting microorganisms. For instance, mixtures 4 to 7 in this work showed values of TOC/TKN compatible with composting, taking into account commonly used values [10,32]. Nevertheless, none of these mixtures reached $55\text{ }^{\circ}\text{C}$ during the active phase of composting and the cumulative DH was significantly lower than the other mixtures. This can be attributable to the fact that the main components of these mixtures (pig slurry

and tree pruning) are characterized by low contents of easily degradable forms of C and N [38]. The mixtures that showed the worst temperature parameters during the active phase of composting (mixture 8 and 9) were characterized by high values of TOC/WEN, WEOC/WEN, TOC/WEON and WEOC/WEON, suggesting that the presence of a large amount of organic matter, even in soluble forms, is not a sufficient parameter to predict the correct evolution of temperature.

Table 6. Linear regression between chemical parameters of the mixtures and temperature evolution during the active phase of composting.

x	T Max (y)				h > 55 °C (y)				Cumulative DH (y)			
	m	q	R ²	r	m	q	R ²	r	m	q	R ²	r
TOC	−2.1080	117.93	0.3124	n.s.	−7.8552	347.46	0.1351	n.s.	−853.54	50299	0.2379	n.s.
TKN	24.3370	11.01	0.1919	n.s.	18.7950	49.68	0.0036	n.s.	7560	10217	0.0860	n.s.
TOC/TKN	−0.9250	70.85	0.3588	0.5990 *	−2.1832	136.84	0.0623	n.s.	−336.34	30174	0.2204	n.s.
Amm-N	297.18	14.12	0.3152	n.s.	1694.8	−100.54	0.3193	n.s.	155288	4626	0.3998	0.6323 *
TOC/Amm-N	−0.0596	69.03	0.4314	0.6568 *	−0.2070	159.25	0.1623	n.s.	−26.81	31584	0.4060	0.6372 *
WEOC	−3.1730	56.78	0.2173	n.s.	−1.4462	81.33	0.0014	n.s.	−1044.3	24653	0.1093	n.s.
WEOC/TKN	−2.7408	54.62	0.3159	n.s.	−3.9539	89.75	0.0205	n.s.	−978.41	24207	0.1870	n.s.
WEOC/Amm-N	−0.1926	55.10	0.3945	0.6281 *	−0.4063	97.13	0.0547	n.s.	−75.89	24750	0.2845	n.s.
WEN	54.7250	26.06	0.4417	0.6646 *	205.60	4.55	0.1942	n.s.	27274	11324	0.5095	0.7138 **
WEON	50.6530	32.75	0.3551	0.5959 *	175.39	33.32	0.1326	n.s.	25059	14704	0.4036	0.6353 *
TOC/WEN	−0.1101	64.87	0.7358	0.8578 **	−0.3418	137.45	0.2209	n.s.	−47.13	29276	0.6263	0.7914 **
TOC/WEON	−0.0404	62.94	0.7465	0.8640 **	−0.1367	136.48	0.2667	n.s.	−17.58	28580	0.6577	0.8110 **
WEOC/WEN	−0.2100	54.31	0.5068	0.7119 **	−0.7904	95.75	0.0785	n.s.	−141.06	24327	0.3729	0.6107 *
WEOC/WEON	−0.1467	53.78	0.5403	0.7351 **	−0.3477	96.60	0.0945	n.s.	−58.98	24298	0.4054	0.6367 *

n.s.: Not significant, *: Significant at $p < 0.05$, ** significant at $p < 0.01$, $n = 12$; T max: Maximum temperature during the active phase, DH: Degree hours, TOC: Total organic C, TKN: Total Kjeldahl N, Amm-N: Ammonium-N, WEOC: Water-extractable organic C, WEN: Water-extractable N, WEON: Water-extractable organic N.

Based on the results reported in Tables 5 and 6, an attempt to propose the optimal values of chemical parameters to optimize the active phase of composting was carried out. Nevertheless, it should be highlighted that these are only preliminary results that need experimental confirmation. If the WEN is considered, its concentration should be higher than 0.4% (w/w) in order to ensure an optimal amount of easily available N. Indeed, in the present work, all the mixtures that showed the correct development of the active phase were characterized by a WEN concentration higher than 0.4% (w/w). Within the ratios studied, TOC/WEN and TOC/WEON appeared to be the most promising parameters that should be taken into consideration during the preparation of composting mixtures, since they were found to correlate significantly with the correct development of the process. With regard to the results reported in the present work, the TOC/WEN ratio should range between 40 and 80 to achieve high temperatures and cumulative DH, whereas the TOC/WEON ratio should range between 40 and 120. The proposed values should ensure a balanced ratio between C and N amounts and their availability, allowing for the quick growth of microorganisms and correct behavior of composting.

3.4. Mixture Characteristics and Their Effect on Compost Quality

The main characteristics of the compost produced from the 12 mixtures studied and the limit values for quality compost production established by Italian legislation are reported in Table 7.

TOC and TKN concentrations in the composts studied varied depending on the initial composition of the mixtures. The highest amount of TOC was found in the compost produced from olive mill wastes (mixtures 8 and 9), whereas the highest amount of TKN was found in the compost produced from sewage sludge and anaerobically digested pharmaceutical wastewater (mixtures 10, 11 and 12).

OM-loss values higher than 30–40% are usually considered an index of a correctly developed composting process [39,40]. In the present work, only five composts showed optimal values of OM-loss (composts from mixtures 1, 2, 3, 11 and 12), whereas compost from mixture 10 showed a value near to the limit (30.9%). In the other composts (4, 5, 6, 7, 8 and 9), OM-loss ranged from 5.8 to 18.2%, demonstrating that the lack of an increase in temperature in the active phase compromised the whole composting process, also

negatively affecting the decrease in moisture. Indeed, most of the composts produced from mixtures that did not undergo a proper increase in temperature during the active phase (composts from mixtures 4, 6, 8, 9 and 10) showed high moisture values (above 50% *w/w*). Conversely, when an adequate temperature profile was achieved during composting, heating caused a significant decrease in moisture, as expected at the end of composting [41].

The pH of composts showed values expected at the end of composting. Indeed, compost usually shows a sub-alkaline pH due to the mineralization of volatile fatty acids and the release of ammonium-N after proteins' hydrolysis [1,2]. Soluble salts' content (estimated from the electrical conductivity, EC) in the composts varied in relation to the composition of the initial mixtures and ranged from 0.6 dS m⁻¹ to 4.6 dS m⁻¹ (composts obtained from mixtures 9 and 10, respectively).

The compost maturity was evaluated by the determination of the content of humic and fulvic acids (HA + FA), as well as the germination index (GI). Only compost produced by the mixtures containing olive mill wastes (mixtures 8 and 9) showed low values of HA + FA and GI (6.8 and 6.3% of HA + FA, 58.7 and 54.3% of GI). Considering that these were the two mixtures showing the worst temperature profiles, these results confirmed that a proper active phase of composting is also essential for the correct behavior of the curing phase. Indeed, the maturation of compost mainly occurs during the curing phase, when the phytotoxic compounds are mineralized and the organic matter is stabilized [42].

The comparison of the results of compost characterization with the Italian limits for quality compost production showed that five of the seven composts produced by mixtures that did not show a proper temperature profile in the active phase did not comply with the legal requirements for compost commercialization (compost from mixtures 4, 6, 8, 9 and 10) [43]. Whereas composts from mixtures 4, 6 and 10 showed excessive moisture, composts from mixtures 8 and 9 (olive mill waste mixtures) did not comply with four parameters (maximum C/N and moisture, minimum content of HA + FA and minimum GI value). Considering these results, ensuring the correct development of the active phase of composting appears mandatory to obtain a high quality of the compost produced. Given that composting is an energy-consuming process, the importance of optimizing the composition of initial mixtures is clear, as is using appropriate calculation tools such as the one described in Calisti et al. [44]. If composting mixtures are not well optimized, the final compost might not comply with legal limits and should then be disposed of by incineration or landfilling, causing environmental and economic issues [45,46].

The possible relationship between the new parameters proposed to optimize composting mixtures (WEN, WEON, TOC/WEN, TOC/WEON, WEOC/WEN and WEOC/WEON) and the main physico-chemical characteristic of composts was investigated by correlation analysis and the results are reported in Table 8.

Interestingly, the parameters of initial mixtures that were correlated with the temperature parameters in the active phase of composting were also correlated to most of the selected physico-chemical characteristics of composts. Obviously, TOC and C/N of composts were positively correlated to TOC/WEN, TOC/WEON, WEOC/WEN and WEOC/WEON ratios, and the EC of compost was positively correlated to WEN and WEON in the mixtures. This latter correlation was expected since soluble N compounds (i.e., ammonium-N) increase the electrical conductivity of the water-compost extract. In this context, it must be taken into account that an excessive WEN or WEON concentration in the initial mixture can result in high EC in the composts, with potential phytotoxic effects [47].

Table 7. Main characteristics of the compost produced from the mixtures studied and limit values for quality compost production [43].

Parameters	Mixtures												Limit Value _d
	1 ^a	2 ^a	3	4	5	6	7	8	9	10 ^b	11 ^b	12 ^c	
TOC (%)	20.2 ± 0.8	21.7 ± 0.2	21.6 ± 0.2	25.4 ± 0.1	24.8 ± 0.0	23.7 ± 0.1	25.2 ± 0.0	36.2 ± 0.0	36.2 ± 0.5	30.7 ± 0.6	23.2 ± 0.3	30.4 ± 0.2	20 (min)
TKN (%)	2.2 ± 0.1	2.1 ± 0.1	1.0 ± 0.2	1.4 ± 0.1	1.3 ± 0.1	1.2 ± 0.2	1.3 ± 0.1	1.2 ± 0.0	1.3 ± 0.0	3.2 ± 0.2	2.5 ± 0.2	2.7 ± 0.2	-
TOC/TKN	9.2	10.3	21.6	18.1	19.1	19.8	19.4	30.2	27.8	9.6	9.3	11.3	25
OM-loss (%)	62.1 ± 0.3	80.2 ± 0.8	66.0 ± 0.4	17.0 ± 0.1	17.5 ± 0.0	18.2 ± 0.2	17.9 ± 0.1	6.2 ± 0.6	5.8 ± 0.5	30.9 ± 0.4	63.1 ± 1.3	38.8 ± 1.0	-
Moisture (%)	36.9 ± 0.2	31.5 ± 0.6	44.9 ± 0.5	53.7 ± 0.6	42.8 ± 0.8	55.9 ± 0.5	48.1 ± 0.0	59.2 ± 0.4	63.6 ± 0.7	63.0 ± 0.6	48.3 ± 0.4	48.1 ± 0.3	50
pH	7.3 ± 0.1	7.2 ± 0.0	7.9 ± 0.0	8.0 ± 0.1	7.8 ± 0.1	8.0 ± 0.1	7.9 ± 0.0	7.0 ± 0.0	7.2 ± 0.0	7.9 ± 0.0	8.3 ± 0.2	8.4 ± 0.1	6–8.5
EC (dS m ⁻¹)	2.3 ± 0.0	2.1 ± 0.0	3.7 ± 0.2	1.4 ± 0.2	1.4 ± 0.1	1.5 ± 0.1	1.4 ± 0.2	0.8 ± 0.1	0.6 ± 0.0	4.6 ± 0.0	1.9 ± 0.2	3.5 ± 0.2	-
HA + FA (%)	8.4 ± 0.1	9.3 ± 0.1	11.4 ± 0.2	7.7 ± 0.2	8.3 ± 0.1	8.2 ± 0.2	8.4 ± 0.2	6.8 ± 0.1	6.3 ± 0.0	8.3 ± 0.1	11.7 ± 0.1	12.1 ± 0.1	7 (min)
GI (%)	112.9 ± 1.4	103.6 ± 0.8	100.6 ± 2.0	72.9 ± 2.3	69.3 ± 1.6	70.8 ± 1.0	69.4 ± 0.5	58.7 ± 0.8	54.3 ± 1.0	78.3 ± 1.4	92.3 ± 2.3	83.7 ± 0.2	60 (min)

^a [21]; ^b [3]; ^c [20]; ^d [43]; Mean value ± SEM, n = 3; TOC: Total organic C, TKN: Total Kjeldahl N, OM: Organic matter, EC: Electrical conductivity, HA + FA: Humic and fulvic acids, GI: Germination index. Data are expressed on dry basis except for pH and EC.

Table 8. Pearson's correlation coefficients between selected parameters of the mixtures and composts studied.

		Mixture						Compost								
		WEN	WEON	TOC/WEN	TOC/WEON	WEOC/WEN	WEOC/WEON	TOC	TKN	C/N	OM-loss	Moisture	pH	EC	HA + FA	GI
Mixture	WEN	1														
	WEON	0.9880 **	1													
	TOC/WEN	-0.6910 *	-0.6199 *	1												
	TOC/WEON	-0.6798 *	-0.6798 *	0.9725 **	1											
	WEOC/WEN	n.s.	n.s.	0.9344 **	0.8438 **	1										
	WEOC/WEON	n.s.	n.s.	0.9538 **	0.8820 **	0.9940 **	1									
Compost	TOC	n.s.	n.s.	0.7624 **	0.6919 *	0.7904 **	0.7884 **	1								
	TKN	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	0.5830 *	1							
	C/N	n.s.	n.s.	0.8397 **	0.8379 **	0.7524 **	0.7647 **	0.5830 *	-0.8119 **	1						
	OM-loss	0.7599 **	0.7243 **	-0.7374 **	-0.7863 **	n.s.	n.s.	-0.6904 *	n.s.	-0.6836 *	1					
	Moisture	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	0.5823 *	n.s.	0.5777 *	n.s.	1				
	pH	n.s.	n.s.	n.s.	n.s.	-0.6650 *	-0.6507 *	n.s.	n.s.	n.s.	n.s.	n.s.	1			
	EC	0.7798 **	0.7643 **	-0.7161 **	-0.7575 **	n.s.	n.s.	n.s.	0.6419 *	-0.5848 *	n.s.	n.s.	n.s.	1		
	HA + FA	0.7351 **	0.7450 **	-0.7222 **	-0.7501 **	-0.5927 *	-0.6198 *	n.s.	n.s.	n.s.	0.6774 *	n.s.	0.6722 *	0.5898 *	1	
	GI	0.7626 **	0.6892 *	-0.7984 **	-0.8320 **	-0.6105 *	-0.6418 *	-0.7870 **	n.s.	-0.7343 **	0.9476 **	n.s.	n.s.	n.s.	0.6065 *	1

n.s.: not significant, *: significant at $p < 0.05$, **: significant at $p < 0.01$, n = 12. TOC: total organic C, TKN: total Kjeldahl N, WEOC: water extractable organic C, WEN: water extractable N, WEON: water extractable organic N, OM: organic matter, EC: electrical conductivity, HA + FA: humic and fulvic acids, GI: germination index.

OM-loss was positively correlated with WEN and WEON, demonstrating that soluble N compounds are essential to sustain microbial metabolism and, thus, composting effectiveness. Conversely, OM loss was negatively correlated with TOC/WEN and TOC/WEON ratios, confirming that high values of these ratios cannot ensure a balanced substrate for microbial growth during composting.

Moreover, the parameters commonly used to evaluate the maturity were correlated with the new parameters proposed to optimize composting mixtures. Both HA + FA and GI were positively correlated with WEN and WEON, and negatively correlated with TOC/WEN, TOC/WEON, WEOC/WEN and WEOC/WEON ratios. Considering these results, the parameters proposed to optimize composting mixtures also appear suitable to ensure the production of good-quality composts, in terms of hygienization, stabilization and maturation.

4. Conclusions

The present work aimed to assess new parameters for composting in order to better explain the process and to optimize it, in terms of correct temperature evolution and high-quality compost. Although physico-chemical (i.e., moisture, air-filled porosity) and chemical (i.e., TOC/TKN) parameters were commonly used to optimize composting mixtures, they did not ensure the correct development of the aerobic process. Conversely, a significant positive correlation was found between temperature evolution during the active phase of composting and soluble forms of N (WEN and WEON) in the starting mixtures, demonstrating that the easily available N compounds play a key role in the correct development of composting. On the contrary, high TOC/WEN, TOC/WEON, WEOC/WEN and WEOC/WEON ratios resulted in a low temperature and poor quality of the final compost.

Based on these results, it was suggested that the study of soluble forms of C and N may help to predict the mineralization rate during the active phase and the correct increase in temperature. In particular, WEN and the TOC/WEN ratio might be useful parameters to evaluate the aptitude of a starting mixture to be composted. Nevertheless, future research is needed to confirm the results reported in this work, by studying mixtures with fixed values of moisture, AFP, volatile solids, pH and C/N, in order to better understand the role of WEN and TOC/WEN ratio parameters in the degradation of organic matter during composting.

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