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# Fertilizing potential and CO<sub>2</sub> emissions following the utilization of fresh and composted food-waste anaerobic digestates

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## ARTICLE INFO

# ABSTRACT

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Keywords: Organic waste Phosphorus fractionation Relative phosphorus efficiency Ryegrass Wet and dry-batch anaerobic digestion, and composting are common technologies in food waste (FW) management, resulting in different outputs. However, the effects of composting on carbon dioxide (CO<sub>2</sub>) emissions, nitrogen (N) and phosphorus (P) fertilizing capacity in view of closing nutrient cycle are still poorly investigated. In this work, two FW anaerobic digestates from the wet (D<sub>1</sub>) and dry-batch process (D<sub>2</sub>), and their respective composts (C<sub>1</sub> and C<sub>2</sub>) were tested in a soil incubation (84 days at 25 °C) to assess CO<sub>2</sub> emissions in comparison with a mixed (animal slurry/energy crop) digestate (BD) and a reference municipal solid waste compost (MSWC). The same products were also tested for the relative P efficiency (RPE) in soil, in comparison with a chemical-P source (30 mg P kg<sup>-1</sup>). Lastly, the apparent recovery fraction of N (N-ARF) from the five organic products was determined in a pot test with ryegrass (84 days; 300 kg available N ha<sup>-1</sup>), compared to a chemical fertilizer (NPK). Composting strongly reduced net-CO<sub>2</sub> emissions compared to the two digestates (625 vs. 2850 mg CO<sub>2</sub> kg<sup>-1</sup> soil). Oppositely, composting very modestly influenced RPE that ranged around 100–90% in D<sub>1</sub> and C<sub>1</sub>, and ≈30% in D<sub>2</sub> and C<sub>2</sub>. Moreover, composting did not significantly reduce N-ARF that ranked in descending order as follows: NPK (77.5%)>D<sub>1</sub>=BD (17.7%)≥C<sub>1</sub> (14.7%)>MSWC (3.6%)>D<sub>2</sub> (1.2%)>C<sub>2</sub> (-3.1%). Composting was shown a reliable strategy for FW digestate management, as it reduces potential CO<sub>2</sub> emission without affecting these products' N- and P-fertilizing capacity.

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# **1. Introduction**

Ever increasing production of food waste (FW) following the EU policies about the organic waste (OW) recycling strongly promotes anaerobic digestion (AD) as a reliable technology for its proper management, this representing at the same time a source of renewable energy (Nicholson et al., 2017). In this frame the AD of OW is seen as a valuable strategy to comply with the reduction of landfilled/incinerated waste, in turn reducing the whole amount of greenhouse gases (GHG) emitted from this waste stream, being this considered a win-win strategy (Knoop et al., 2018; Kothari et al., 2010; Slorach et al., 2019).

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In addition to this it is widely recognized that anaerobic digestates (Ads) are rich of nutrients potentially available for crop growth, in this context these have been widely utilized as fertilizers by direct spreading to the soils (Nicholson et al., 2017). In this framework the partial/total substitution of chemical fertilizer with Ads can help to reduce the whole amount of GHG emitted from the agricultural compartment. However, as reported by Evangelisti et al. (2014) and by Slorach et al. (2019), many drawbacks are associated with the direct soil distribution of anaerobic digestates such as the emission of ammonia (NH<sub>3</sub>), beside emission of both carbon dioxide (CO<sub>2</sub>) and nitrous oxide (N<sub>2</sub>O). In this light to reduce these problems AD is often coupled with composting to obtain stable products to be safely used in soils (Alburquerque et al., 2012a, 2012b; Grigatti et al., 2014; Nicholson et al., 2017).

In this framework, many researches in the latest years have been investigating the fertilizing potential (mainly nitrogen driven) and the GHG emission (especially  $CO_2$ ) from the effluents originating from the AD of various substrates (animal slurries, sewage sludge, agricultural and agro-industrial waste), and from their composts, following their addition to the soil (Grigatti et al., 2011; Alburquerque et al., 2012a, 2012b; Verdi et al., 2019). However as reported by Nicholson et al. (2017), fewer are the researches investigating the  $CO_2$  emission and the fertilizing capacity and the GHG emission from fresh and composted ADs obtained from food waste.

Abbreviations: AD, anaerobic digestion; Ads, anaerobic digestates; BD, digestate from the wet anaerobic digestion of animal slurry and energy crops;  $C_1$ , compost from  $D_1$ ;  $C_2$ , compost from  $D_2$ ;  $D_1$ , digestate from the wet digestion of food-waste;  $D_2$ , digestate from the dry-batch digestions of food-waste; FW, food waste; MSWC, municipal solid waste compost; N-ARF, nitrogen apparent recovery fraction; OW, organic waste; P-chem, chemical P source  $[Ca(H_2PO_4)_2]$ ; RPE, relative phosphorus efficiency; SCE, sequential chemical extraction; TS, total solids; VS, volatile solids

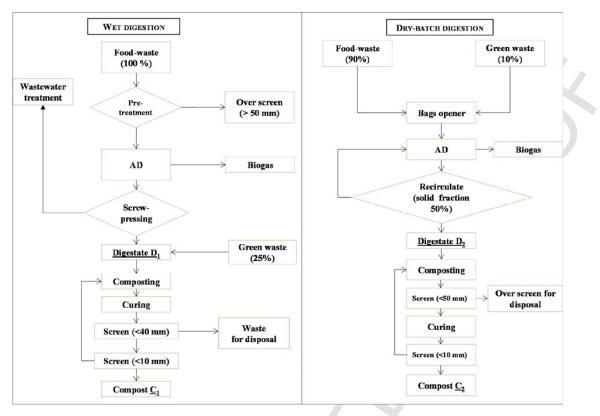


Fig. 1. Schematic representation of the wet and dry-batch anaerobic digestion process of the food waste.

# Table 1 Main characteristics of the tested products.

Product	pН	TS	VS	С	N	C:N	NH <sup>+</sup> <sub>4</sub> -N	NO <sup>-</sup> 3-N	OUR	Р	Ca	Fe	Al	Mg	K	S	Ca:P
		(%)		(mgg	<sup>-1</sup> )		$(mg kg^{-1})$		$(\text{mmol O}_2 \text{ kg}^{-1} \text{ VS h}^{-1})$	(mgg	g <sup>-1</sup> )						
D <sub>1</sub>	8.4	24.8	58.8	313	35	8.9	234	59	54	9.3	43	8	10	7	4.9	4.4	5
$D_2$	8.9	34.0	50.1	302	16	18.9	244	26	64	5.1	72	6	8	5	5.3	3.5	14
$\overline{C_1}$	7.3	63.0	39.0	242	25	9.7	181	207	3	6.7	40	12	11	11	5.3	3.2	6
$C_2$	10.0	76.0	42.5	255	18	14.2	199	40	12	7.8	88	10	9	7	2.9	3.9	11
BD	8.4	5.05	68.3	515	43	11.9	200	50	55	6.2	11	1	0.4	5	26	3.9	2
MSWC	8.4	88.6	43.6	222	13	17.1	101	23	2	4.3	46	15	15	6	5.0	3.1	11

TS: total solids; VS: volatile solids; C: total carbon: N: total nitrogen  $D_1$ : digestate from the wet digestion of bio-waste;  $D_2$ : digestate from the dry-batch digestion of bio-waste;  $C_1$ : compost from  $D_1$ ;  $C_2$ : compost from  $D_2$ ; BD: digestate from the wet AD of animal slurry and energy crops; MSWC: reference compost. VS, C, N, P, Ca, Fe, Al, Mg, K and S content are expressed on TS basis.

# Table 2

Heavy metal content in the compared products  $(mg kg^{-1})$ .

Product	Element					
	Cd	Cr	Ni	Cu	Zn	Pb
D <sub>1</sub>	0.70	45	24	64	201	55
$D_2$	0.33	30	12	52	116	20
$\overline{C_1}$	0.97	101	58	92	175	48
C <sub>2</sub>	1.00	91	16	119	205	43
BD	0.39	7	7	55	192	2
MSWC	1.20	57	27	125	224	72

Values are means of two replicates of organic samples; data are expressed on TS; mean deviation<5%. D<sub>1</sub>: digestate from the wet digestion of bio-waste; D<sub>2</sub>: digestate from the dry-batch digestion of bio-waste; C<sub>1</sub>: compost from D<sub>1</sub>; C<sub>2</sub>: compost from D<sub>2</sub>; BD: digestate from the wet AD of animal slurry and energy crops; MSWC: reference compost.

More specifically, few information is available to rate whether aerobic stabilization can affect the potential  $CO_2$  emission and the nutritional (N and P) capacity for plants, following the composting of digestates obtained from food waste, being this issue the main concern for a successful environmental sustainability of this waste management strategy (Nicholson et al., 2017; Slorach et al., 2019).

As previously said, the food-waste production/collection is continuously increasing in the EU countries (Nicholson et al., 2017). In this framework the most widely employed technologies for the anaerobic digestion of food -waste are the "wet" and "dry-batch" digestion (Luning et al., 2003; Angelonidi and Smith, 2015). Featuring a respective low and high total solids environment: 10–15% total solids (TS) is a common range for wet digestion, and 25–40% total solids for dry-batch digestion. In the wet process, food waste is slurried with the addition of water and mixed in tank digesters that are typically used in this process. Conversely, in the dry-batch process the

# Table 3 Phosphorus removed in the sequential extraction of the tested products.

Extractant/fraction	D <sub>1</sub>		$D_2$		$C_1$		$C_2$		BD		MSWC	
	$(mgg^{-1})$	(%)	$(mg g^{-1})$	(%)	$(mgg^{-1})$	(%)	$(mgg^{-1})$	(%)	$(mgg^{-1})$	(%)	$(mgg^{-1})$	(%)
H <sub>2</sub> O-P	0.70	8	0.59	12	0.36	5	0.36	5	2.7	44	0.16	4
NaHCO <sub>3</sub> -P	3.18	34	0.62	12	2.13	32	0.63	8	2.5	40	0.54	13
NaOH-P	0.81	9	0.08	2	0.60	9	0.15	2	0.8	13	0.58	14
HCl-P	3.47	37	3.45	68	2.43	36	5.83	75	0.9	15	2.82	66
Residual-P (H <sub>2</sub> SO <sub>4</sub> )	0.01	0.1	0.01	0.2	0.01	0.1	0.01	0.1	0.1	2	0.57	13
$\Sigma P_i$	8.16		4.74		5.53		6.96		6.90		4.09	
Recovery (% P <sub>tot</sub> )		88		93		82		89		111		109

Values are means of two replicates of organic samples; data are expressed on TS; mean deviation <5%.  $D_1$ : digestate from the wet digestion of bio-waste;  $D_2$ : digestate from the dry-batch digestion of bio-waste;  $C_1$ : compost from  $D_1$ ;  $C_2$ : compost from  $D_2$ ; BD: digestate from the wet AD of animal slurry and energy crops; MSWC: reference compost.

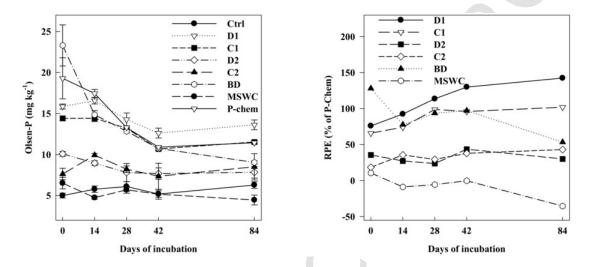


Fig. 2. Olsen-P in soil (left) and relative P efficiency (RPE) (right) during the soil incubation of the compared products.  $D_1$ : digestate from the wet digestion of food waste;  $D_2$ : digestate from the dry-batch digestions of food waste;  $C_1$ : compost from  $D_1$ ;  $C_2$ : compost from  $D_2$ ; BD: digestate from the wet AD of animal slurry and energy crops; MSWC: reference compost. P-chem: Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub>.

mixing of food-waste with structural material (green waste) is generally required. Compared with wet AD, the dry-batch process shows higher volumetric organic loading rate, lower water and energy requirements, and contained nutrient run off during the storage and field spreading of residues (Pavan et al., 2000; Kuroshima et al., 2001). The dry-batch process also results in a lower production of leachate and easier management of digested residues that can be further treated by composting or used as organic fertilizer (ten Brunmeler, 2000). Owing to all these reasons, this technology is rapidly diffusing in EU (De Baere and Mattheeuws, 2013; Angelonidi and Smith, 2015). However, the adoption of either wet or dry-batch AD technology implies the utilization of a different feeding approach: pulped food waste for the former, and a mixture of food waste and structural material (green waste) for the latter. This is likely to reflect in a different fertilizing capacity of the respective digestates and related composts.

As previously said, the re-utilization of recycled OM for agricultural purposes can have many benefits for the soil and can partially substitute chemical fertilizers. However, there is a still a poor knowledge of the fertilizing capacity and environmental risks from the utilization this type of products (Nkoa, 2014). All the analysis (such as life cycle analysis; LCA) aimed to assess the environmental impact of AD treatment of OW in comparison to other management strategies (land-filling/incineration) are based on both the assessment of the direct reduced GHG emission of Ads (in comparison to the other strategies such as land-filling/incineration) and the reduced emission derived from the partial/total substitution of mineral fertilizers (Evangelisti et al., 2014; Nicholson et al., 2017). In this context the deep knowledge of the potential GHG emission (especially  $CO_2$ ), beside the fertilizing capacity of fresh and composted food-waste digestates appear to be strategic.

It is well recognized that field application of stabilized organic products (e.g., compost) is a winning strategy matching the improvement in soil fertility with the nutrient availability increase (Svensson et al., 2004; Odlare et al., 2008; Gunnarsson et al., 2010). Stable composts are very resistant to decomposition and, therefore, significantly contribute to carbon (C) storage in soil (Whalen et al., 2008). Conversely, there is no general consensus about this issue following field application of anaerobically digested by-products.

As previously said the agricultural utilization of Ads represents a promising solution aimed to the nutrient cycle closure, however, many are the potential problems arising from their use in agricultural soils. Due to the economic constraints which require to keep the maximum bio-gas production, anaerobic digestates may have often significant amounts of easily degradable C. This can reflect in a significant potential  $CO_2$  emission once these products are distributed to the soils (Kalnina et al., 2018). However AD is a relatively recent technology applied to the food-waste management; in this light, there is a lack of information about soil behavior of anaerobically digested food wastes and their homologous composts (Nicholson et al., 2017), especially those originating from the dry-batch process that is a more recent approach in this field.

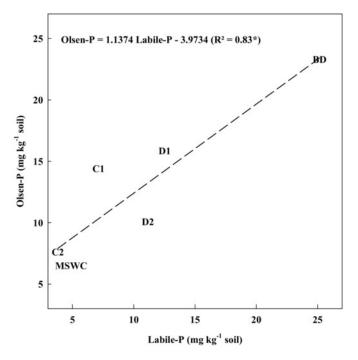


Fig. 3. Relationship between the Labile-P (from sequential chemical extraction) and Olsen-P determined in soil at day 0.  $D_1$ : digestate from the wet digestion of food waste;  $D_2$ : digestate from the dry-batch digestions of food waste;  $C_1$ : compost from  $D_1$ ;  $C_2$ : compost from  $D_2$ ; BD: digestate from the wet AD of animal slurry and energy crops; MSWC: reference compost.

In the literature, the N fertilizing capacity of the Ads from various feedstock (animal slurries, energy crop, sewage sludge, agro-industrial sludge) is recognized to be intermediate between chemical fertilizers and manures, attaining an average 40–60% fertilizing capacity of a chemical source (Grigatti et al., 2011; Nkoa, 2014; WRAP, 2016). Conversely, few information is available about the food-waste Ads and the related composts (Grigatti et al., 2014). On the contrary the P fertilizing potential from Ads is generally considered in the same range or higher with respect to chemical P fertilizers (Bachmann et al., 2016; Grigatti et al., 2015). In this framework composting is generally recognized to decrease the P potentially available for plant nutrition being this phenomenon mainly related to the formation of Ca-P scarcely soluble compounds in the presence of available Ca (Frossard et al., 2002; Eneji et al., 2003). To study these aspects chemical investigation coupled to soil incubation and pot test in vivo are successfully applied (De Boer, 2008; Gunnarsson et al., 2010; Grigatti et al., 2011; Grigatti et al., 2015). The potential P availability from recycled OW can be investigated using sequential chemical extraction (SCE) that provides a reliable picture of the theoretical short-, medium-, and long-term P release (Dou et al., 2000). In addition to P chemical investigation, soil incubation tests are commonly used to determine the P release from organic sources under simulated field conditions. These tests enable reliable information to be obtained concerning initial P availability and subsequent course over time depending on P fixation by soil components. Additionally, the comparison with a soluble mineral P source allows the relative P efficiency (RPE) to be assessed (Leytem and Westermann, 2005; De La Fuente et al., 2013). The utilization of microcosms for soil incubation operating under controlled conditions (moisture, temperature) allows gas emission (CO<sub>2</sub>) to be accurately measured via infra-red photoacoustic technology (Cayuela et al., 2010).

Besides, the plant test in pots grown in a controlled environment offers a valuable insight into the apparent N utilization efficiency of the tested products. The utilization of fast growing species as ryegrass fosters a multiple harvest approach giving the opportunity to describe the N utilization kinetics in the time frame of a growing season (Gunnarsson et al., 2010).

Given this premise, the present study was aimed at investigating the impact of aerobic stabilization on the potential  $CO_2$  emission and the N and P nutritional capacity for plant, following the composting of digestates obtained from food waste.

To do this, we compared the dry-batch and wet anaerobic digestate from food waste together with their composted homologous in a soil incubation experiment to assess  $CO_2$  emissions. These were compared with a reference anaerobic digestate (from animal slurries/energy crops), a reference compost unrelated to AD, a chemical fertilizer and an unamended control. The same treatments were used in a soil incubation test to assess the release of Olsen-P and the RPE in comparison with a soluble chemical P source. These treatments were

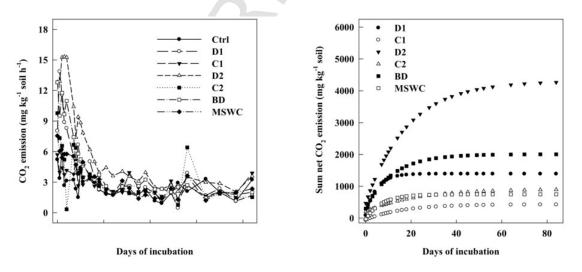


Fig. 4. Instantaneous  $CO_2$  emissions (left) and cumulated net  $CO_2$  emitted during the soil incubation of the compared products.  $D_1$ : digestate from the wet digestion of food waste;  $D_2$ : digestate from the dry-batch digestions of food waste;  $C_1$ : compost from  $D_1$ ;  $C_2$ : compost from  $D_2$ ; BD: digestate from the wet AD of animal slurry and energy crops; MSWC: reference compost.

#### Table 4 Equation parameters and regression coefficients $(R^2)$ of the fitted functions in the compared organic products in the C incubation experiment.

Treatment	Equation parameters						
	Y <sub>0</sub>	C (%)	k (day <sup>-1</sup> )				
D <sub>1</sub>	1.3952	20.6570	0.2093				
$D_2$	3.5114	28.8322	0.0546				
C <sub>1</sub>	-0.8864	7.1392	0.0625				
$C_2$	2.0247	7.0718	0.0455				
ВĎ	3.5400	20.2136	0.0847				
MSWC	-0.4690	11.4502	0.1419				

D<sub>1</sub>: digestate from the wet digestion of bio-waste; D<sub>2</sub>: digestate from the dry-batch digestion of bio-waste; C<sub>1</sub>: compost from D<sub>1</sub>; C<sub>2</sub>: compost from D<sub>2</sub>; BD: digestate from the wet AD of animal slurry and energy crops; MSWC: reference compost. Fitted function model:  $C_m = Y_0 + C_0$  ( $1 - e^{-kt}$ ), where  $Y_0$  is the intercept,  $C_m$  represents the percentage of carbon mineralized at time t,  $C_0$  is the potentially mineralizable carbon, k is the mineralization rate (day<sup>-1</sup>), t is the time (day).

Table 5

also compared in a pot test to determine the apparent N recovery on ryegrass subjected to multiple cuts over 84 days.

## 2. Materials and methods

# 2.1. Organic products

Four organic products derived from food waste were focused in this work: an anaerobic digestate was collected after 15 days of (thermophilic) wet digestion of (100%) pulped food waste (D<sub>1</sub>); a compost was obtained after 110 day of composting of (screw-pressed) D<sub>1</sub> following the addition of green waste (25% w/w) (C<sub>1</sub>) (Fig. 1); another anaerobic digestate was collected after 28 days of (mesophilic) dry-batch digestion of a mixture of food waste and green waste (90/10, w/w) (D<sub>2</sub>). The respective compost, was obtained after 62 days of D<sub>2</sub> processing without the addition of extra green waste, being the structural material derived from recirculation of the solid fraction (50% w/ w) (C<sub>2</sub>) (Fig. 1). In addition to these, an anaerobic digestate from animal slurry and energy crops collected from a local bio-gas plant after 30 days of wet digestion at 37°C (BD), and a municipal solid waste compost (MSWC) from food waste, sewage sludge and green waste were used as organic references in the soil and pot test.

Dry biomass (DW). nitrogen (N) content and uptake in ryegrass shoots at three successive harvests (days 28-84) and root at final harvest (day 84).

Treatments	Days after s	owing						
	Shoot					Root	Total (shoot+root)	
	28	56	84	Mean (0–84)	Sum (0–84)	84	84	
DW (g pot <sup>-1</sup> )								
Control	1.42	0.74	0.26	0.81 b	2.42 d	1.73 ab	4.15 c	
D <sub>1</sub>	1.70	1.33	0.29	1.11 ab	3.32 b	1.54 ab	4.85 b	
$D_2$	1.55	0.89	0.22	0.89 b	2.66 d	1.40 c	4.05 c	
C <sub>1</sub>	1.74	1.13	0.43	1.10 ab	3.29 b	1.67 ab	4.96 b	
C <sub>2</sub>	1.49	0.81	0.23	0.85 b	2.54 d	1.30 c	3.84 c	
BD	1.67	1.04	0.48	1.06 ab	3.19 bc	2.07 ab	5.26 b	
MSWC	1.57	0.88	0.31	0.92 b	2.76 cd	1.41 c	4.18 c	
NPK	1.84	1.74	1.03	1.54 a	4.61 a	2.50 a	7.12 a	
Mean	1.62 a	1.07 b	0.41 c					
N content (mg $g^{-1}$ )								
Control	31.2	12.9	12.1	18.7 c	-	9.3 b	-	
D <sub>1</sub>	38.8	15.7	14.9	23.1 b	-	11.6 ab	-	
$D_2$	27.8	13.4	19.3	20.2 bc	-	11.7 ab	-	
C <sub>1</sub>	35.2	14.0	13.8	21.0 bc	-	11.2 ab	-	
$C_2$	25.6	12.0	16.5	18.0 c	-	11.9 ab	-	
BD	37.1	15.7	13.1	22.0 b	-	10.9 ab	_	
MSWC	27.7	13.7	14.6	18.7 c	-	14.3 a	_	
NPK	59.6	35.1	22.5	39.1 a	-	11.7 ab	_	
Mean	35.4 а	16.6 b	15.9 b					
N uptake (mg pot <sup>-1</sup> )								
Control	44.2	9.5	3.2	19.0 b	56.9 c	16.2 b	73 c	
D <sub>1</sub>	65.0	20.8	4.4	30.1 ab	90.2 b	17.5 b	108 b	
$D_2$	43.0	-11.9	4.2	19.7 b	59.1 c	16.4 b	75 c	
$\tilde{C_1}$	61.1	15.7	5.9	27.5 ab	82.6 b	18.6 b	101 b	
$C_2$	38.1	9.7	3.9	17.2 b	51.7 c	15.5 b	67 c	
BD	61.9	16.2	6.3	28.1 ab	84.4 b	22.1 ab	107 b	
MSWC	43.4	12.2	4.4	20.0 b	60.1 c	19.9 b	80 c	
NPK	109.9	61.1	22.1	64.3 a	193.0 a	28.9 a	222 a	
Mean	58.3 a	19.6 b	6.8 c					

Control: unamended soil; D<sub>1</sub>: digestate from the wet digestion of bio-waste; D<sub>2</sub>: digestate from the dry-batch digestion of bio-waste; C<sub>1</sub>: compost from D<sub>1</sub>; C<sub>2</sub>: compost from D<sub>2</sub>; BD: effluent from the wet AD of animal slurry and energy crops; MSWC: reference compost. NPK: chemical reference (NH<sub>4</sub>NO<sub>3</sub>+KH<sub>2</sub>PO<sub>4</sub>). A one-way ANOVA was applied to harvest, cumulated harvests, and root data; in each column and for each trait. Different letter intervals indicate statistically different mean data according to SNK test (p < 0.05).

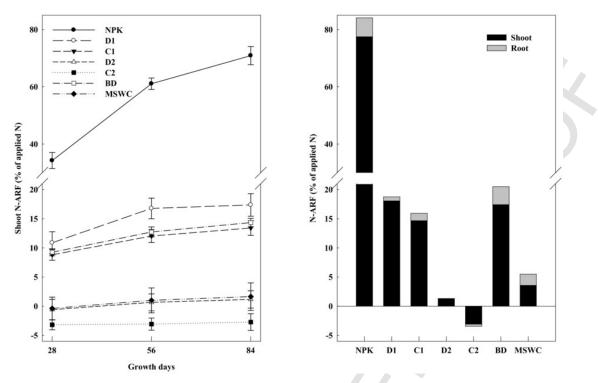


Fig. 5. Cumulative nitrogen apparent recovery fraction (N-ARF) of ryegrass shoots in three subsequent harvests during pot trial (left), and cumulated N-ARF in plant biomass (shoots and roots) at final harvest (right). Error bars, SE (n=3). NPK: chemical fertilizer; D<sub>1</sub>: digestate from the wet digestion of food waste; D<sub>2</sub>: digestate from the dry-batch digestions of food waste; C<sub>1</sub>: compost from D<sub>1</sub>; C<sub>2</sub>: compost from D<sub>2</sub>; BD: digestate from the wet AD of animal slurry and energy crops; MSWC: reference compost.

On the fresh products, we measured pH, electrical conductivity (EC), total solids (TS) and volatile solids (VS),  $\text{NH}^+_4$ -N and  $\text{NO}^-_3$ -N, and the oxygen uptake rate (OUR, Grigatti et al., 2007). The tested products were then freeze-dried and ball milled for further analytical determined via elemental analyzer (EA 1110, Thermo Electron, Germany). The total nutrients and trace elements were determined using ICP-OES (Inductively Coupled Plasma-Optical Emission spectrometry; Spectro Arcos, Ametek, Berwin, PA, USA) on  $\approx 250 \text{ mg}$  samples after microwave assisted digestion (37% HCl and 65% HNO<sub>3</sub>). The main product characteristics are summarized in Table 1.

## 2.2. Phosphorous fractionation

The organic products were submitted to P fractionation via sequential chemical extraction (SCE) according to the method of Dou et al. (2000). Freeze-dried and ball-milled products were extracted for 24 h with deionized water (d-H<sub>2</sub>O) in an end-over-end shaker, and then centrifuged. The supernatants were passed through a Whatman #42 filter, and the recovered pellets were extracted with 0.5 M NaHCO<sub>3</sub> (pH 8.5) for 24 h. The same procedure was repeated with 0.1 N NaOH and 1 N HCl. The residual pellets were treated with a mixture of 96% H<sub>2</sub>SO<sub>4</sub> and 30% H<sub>2</sub>O<sub>2</sub> via hot acid digestion at 360 °C. Inorganic P (P<sub>i</sub>) in the extracts was determined via the molybdenum blue method of Murphy and Riley (1962). The P recovered in each fraction [water extractable P (H<sub>2</sub>O-P), bicarbonate extractable P (NaHCO<sub>3</sub>-P), alkali extractable P (NaOH-P), acid extractable P (HCl-P), Residual-P] was calculated as follows:

$$P_{i \text{ fraction } x}(\%) = \frac{P_{i \text{ fraction } x}}{P_{tot \ OW}} \times 100,$$

where  $P_{i:fraction x}$  is the inorganic P determined in each fraction (H<sub>2</sub>O, NaHCO<sub>3</sub>, NaOH, HCl, Residual), and P<sub>tot OW</sub> is the total P determined in the different organic waste via ICP after microwave-assisted acid digestion.

The total recovery was calculated as the sum of all fractions  $(H_2O-P+NaHCO_3-P+NaOH-P+HCl-P+Residual-P)$  by using the following equation:

$$Tot P_{recovery}(\%) = \frac{\sum_{H2O}^{\text{Residual}} P_{i;o \text{ fraction } x}}{P_{tot OW}} \times 100,$$

where  $\sum_{H2O}^{\text{Residual}} P_{i;o\ fraction\ x}$  represents the sum of the single P recovery values (P<sub>i</sub>) in each fraction (H<sub>2</sub>O-P+NaHCO<sub>3</sub>-P+NaOH-P+HCl-P+Residual-P), and P<sub>tot OW</sub> is the total P determined in the different products via ICP after microwave-assisted acid digestion.

#### 2.3. Soil incubation

The soil used for the incubation and pot trial was collected from the top layer of a field in the Po Valley (Bologna, Italy). It was air dried and crushed at 2 mm, before the following determinations: pH (H<sub>2</sub>O 1:2.5), 7.90; particle-size distribution (mgkg<sup>-1</sup>), 184 sand, 425 silt, 391 clay; total CaCO<sub>3</sub>, 85 gkg<sup>-1</sup>; total organic carbon (TOC),  $10.2 gkg^{-1}$ ; total Kjeldahl nitrogen (TKN),  $1.60 gkg^{-1}$ ; C:N, 8.3; exchangeable K,  $330 \text{ mg kg}^{-1}$  as K<sub>2</sub>O, CEC 27.2 cmol kg<sup>-1</sup>. The total (extractable in aqua regia+HF) Al, Fe and P were 35,661, 22,224 and  $808 \text{ mg kg}^{-1}$  respectively. The NH<sub>4</sub><sup>+</sup>-oxalate (pH, 3) extractable Al and Fe were 764 and 2158 mg kg<sup>-1</sup>, respectively, while the Na dithionite-citrate extractable Al and Fe were 281 and 2462 mg kg<sup>-1</sup>, respectively. The Olsen-P was detected at 4.96 mg kg<sup>-1</sup> (as P). Before the incubation, the soil, previously conditioned as above described, was pre-incubated for four weeks at 25 °C (60% water holding capacity in the dark).

For the assessment of the Olsen-P in soil, the organic products (D<sub>1</sub>, C<sub>1</sub>, D<sub>2</sub>, C<sub>2</sub>), the organic references (MSWC and BD) and the chemical P source  $[Ca(H_2PO_4)_2; P-chem]$  were added to the pre-incubated soil and mixed thoroughly to achieve 30 mg P kg<sup>-1</sup>. Unamended soil was also compared (Control). A completely randomized design with three replicates was adopted. During incubation (25 °C; 84 days), the moisture content was monitored by weighing and addition of deionized water twice a week. The Olsen-P value was determined using the molybdenum blue method at Days 0, 14, 28, 56 and 84. The Olsen-P data were used to calculate the relative phosphorus efficiency (RPE), which was used to normalise the P data obtained in the organic treatments to P-Chem, as proposed by Leytem and Westermann (2005).

# 2.4. CO<sub>2</sub> measurements

For the CO<sub>2</sub> measurements the organic products (D<sub>1</sub>, C<sub>1</sub>, D<sub>2</sub>, C<sub>2</sub>) and the organic references (MSWC and BD) were added and mixed thoroughly with the pre-incubated soil to achieve 300 kg available N ha<sup>-1</sup> (i.e., 750 kg N ha<sup>-1</sup> at 40% of N availability; considering a ploughing layer of 30 cm and a density of 1.3, §Table 1S).

The vessels with soil (250 g) were introduced into 2.51 politetrafluoroetilene (PTFE) vessels that were closed with an air tight lid provided with an inspection hole. During the incubation, the fluxes of CO<sub>2</sub> were measured 28 times by using photo-acoustic infrared analyzer (1302 Photoacustic field gas-monitor; Innova Airtech Instruments A/S, Ballerup Denmark). The photo-acoustic gas analyzed was connected to 2.51 vessels via Teflon tubes and needle through septa to measure the concentration of CO<sub>2</sub> in the head-space of the air sealed jars. The concentration of CO<sub>2</sub> was measured after closing the vessels for 2h during the first two weeks, thereafter closure times was increased to 3h till the end of the experiment. The fluxes (CO<sub>2</sub>) were measured 5 times per week during the first two weeks, then two times per week in the following five weeks, and eventually once a week until the end. The fluxes were measured at 1, 2, 3, 4, 7, 8, 9, 10, 11, 14, 16, 18, 20, 21, 24, 28, 31, 35, 38, 42, 45, 49, 52, 56, 64, 70, 77, and 84 days of incubation.

Fluxes (mg of CO<sub>2</sub> kg<sup>-1</sup> soil h<sup>-1</sup>) were calculated assuming a linear increase of gas concentrations between consecutive measuring, as suggested by Cayuela et al. (2010). The total amount of gas emitted (mg of CO<sub>2</sub> kg<sup>-1</sup> soil) was calculated as sum of the total daily gas emission obtained by linear interpolation of instantaneous emissions between consecutive sampling times (Straathof et al., 2014; Fungo et al., 2019). Mineralized C (% of added C) was calculated on the basis of total CO<sub>2</sub> emissions, deducing the emissions from the control as reported by Grigatti et al. (2014). The C mineralization kinetics were analyzed by Sigma Plot 10 software (Systat Software Inc., Chicago, IL, USA). The kinetics fitted a single exponential model with the following equation:

$$C_m = Y_0 + C_0 (1 - e^{-kt})$$

where  $Y_0$  is the intercept,  $C_m$  represents the percentage of carbon mineralized at time t,  $C_0$  is the potentially mineralizable carbon, k is the mineralization rate (day<sup>-1</sup>), t is the time (day).

## 2.5. Plant test

The organic products (D1, C1, D2, C2), the organic references (MSWC and BD) were added to the soil and mixed thoroughly to achieve 300 kg available N ha<sup>-1</sup> as reported in the soil incubation. The chemical fertilizer (NPK) was added at the same rate 300kg available N ha<sup>-1</sup> (at 100% of N availability; considering a ploughing layer of 30 cm and a density of 1.3 §Table 1S). Unamended soil was also compared (Control). Then the amended soil (1kg on TS basis) was added to 2L pots (ø 16 cm), previously half filled (1 L) with expanded agricultural clay (with controlled pH). A completely randomized design with three replicates was adopted. Ryegrass (Lolium multiflorum subsp. *italicum*) seeds, cv. Sprint were sown  $(1 \text{ g pot}^{-1})$  and then covered with a thin layer of sand to prevent dehydration. Pots were placed in a growth chamber with a 14/10h light/dark photoperiod and respective temperatures of 23/13 °C. The lighting was assured by Master Tld 58 W-840 tubes (Philips, Amsterdam, The Netherlands). The pots were regularly watered with tap water to keep constant moisture level (60% WHC). At 4-week intervals (28, 56 and 84 days after sowing), the plants were harvested by cutting the stems 2 cm above the soil, and the fresh tissue was dried in a forced-air oven (60 °C for 72 h). On the last harvest (day 84), the roots were also recovered by watering way the soil, and they were dried as described above. Total nitrogen (TN) was determined on plant tissue and root via elemental analyser (EA 1110, Thermo Electron, Germany).

# 2.6. Statistical analysis

Two-way ANOVA was run for the factors fertilizer treatments and harvests, and their interaction, addressing the shoot parameters (DW; N content; N uptake). One-way ANOVA was run on cumulated harvests of shoots (28–84 d), and root at final harvest (84 d), addressing the same parameters. Tukey's HSD test at p < 0.05 was used to separate levels in significant ANOVA sources: The Statistica 7® package (Statsoft, Tulsa, OK, USA) was used to perform ANOVA and the Tukey test.

The cumulative plant nitrogen apparent recovery fraction (N-ARF) (%) was calculated according to Gunnarsson et al. (2010), as follows:

N-ARF (%) = 
$$\frac{\sum_{28}^{84} \left( N_{treatment}(tn) - N_{control(tn)} \right)}{N_{applied}} \times 100$$

where:  $N_{treatment (tn)}$  is the N uptake (mg pot<sup>-1</sup>) from the tissue of treated pots (digestates, composts, and NPK) at time t (28; 56; 84 etc.);  $N_{control (tn)}$  is the N uptake (mg pot<sup>-1</sup>) from the tissue of control pots at time t (28; 56; 84);  $N_{applied}$  is the N applied with treatments (mg pot<sup>-1</sup>).

# 3. Results

#### 3.1. Organic products

The four organic products compared in this work showed very different characteristics (Table 1). To a deeper insight, the slurries from anaerobic digestion  $(D_1 \text{ and } D_2)$  showed similar pH (ranging between 8.4 and 8.9), and staged a limited variation also in TS (25 and 35% in the two respective products). In this frame, the VS and C content ranged very close, attaining about 50-60% the former, and  $\approx 300 \text{ mg g}^{-1}$  the latter in D<sub>1</sub> and D<sub>2</sub>, respectively. A similar stability (OUR) was also achieved (54 and 64 mmol  $O_2 \text{ kg}^{-1} \text{ VS h}^{-1}$ ). Conversely,  $D_1$  and  $D_2$  showed a contrasting N content (35 vs.  $16 \text{ mg g}^{-1}$ ), leading to a very different C:N ratio (8.9 vs. 19.9). The two composts ( $C_1$  and  $C_2$ ) deriving from the respective digestates ( $D_1$  and  $D_2$ ) showed a pH ranging between 7.3 and 10 ( $C_1$  and  $C_2$ ). Compared to C<sub>2</sub>, C<sub>1</sub> featured lower TS (63 vs. 76%), VS (39 vs. 43%), and C (242 vs.  $255 \text{ mg g}^{-1}$ ). Oppositely, C<sub>1</sub> (from wet digestate) attained a higher N (25 vs.  $18 \text{ mg g}^{-1}$ ), reflecting in a lower C:N ratio (9.7 vs. 14.2). The two composts ( $C_1$  and  $C_2$ ) showed much higher stability than the original slurries; they also showed slight differences between them (3 and  $12 \text{ mmol } O_2 \text{ kg}^{-1} \text{ VS } \text{h}^{-1}$ ). The content of nutrients other than C and N outlines a variable picture (Table 1). P was comprised between 5 and  $9 \text{ mg g}^{-1}$  in the four studied products (C<sub>1</sub>, C<sub>2</sub>, D<sub>1</sub> and D<sub>2</sub>), Ca between 40 and  $88 \text{ mg g}^{-1}$ , and the other cations (Fe, Al, Mg and K) and anions (S) never passed 10 mg g<sup>-1</sup>. There is no clear trend in any of these nutrients between digestates and composts, nor within the two products of each category. The reference products (BD and MSWC) showed similarities as well as divergences with respect to the investigated products (Table 1). The pH was close to the investigated products' average. TS were very low and high in the respective BD and MSWC, while VS were consistent with data in the two product categories, i.e. higher in digestate (BD) and lower in reference compost (MSWC). C and N were highest and lowest in the respective BD and MSWC, compared to the investigated products. The resulting C:N ratio was quite low in BD; high in MSWC. The other nutrients fitted, substantially, in the above described ranges, with the only exception of K being approximately five times as high in BD with respect to all the other products.

As reported in Table 2, the heavy metal content was similar in the two digestates ( $D_1$  and  $D_2$ ). However,  $D_1$  showed a slightly higher content than  $D_2$  in all heavy metals. The reference digestate (BD) was at or below  $D_2$  values in all heavy metals except Zn. The two composts ( $C_1$  and  $C_2$ ) also had a similar content in all heavy metals; overall, heavy metals tended to concentrate more in composts than in digestates. Lastly, the reference compost (MSWC) was substantially similar in heavy metal content to the investigated composts.

#### 3.2. Phosphorus fractionation

The products compared in this work showed very different phosphorus fractionation (Table 3). The products of anaerobic digestion (D<sub>1</sub> and D<sub>2</sub>) performed similar H<sub>2</sub>O-P (0.70 vs. 0.59 mg g<sup>-1</sup>) while revealed very different NaHCO<sub>3</sub>-P (3.18 vs. 0.62 mg g<sup>-1</sup>), thus performing very different labile-P (42 vs. 24%). These products showed also important differences in the NaOH-P (0.81 vs. 0.08 mg g<sup>-1</sup>), these representing 9 and 2% of the whole extractable P. HCl-P was the dominant fraction in both D<sub>1</sub> and D<sub>2</sub> (3.47 vs. 2.43 mg g<sup>-1</sup>) corresponding to 37 and 68% of the extractable P. The composted products (C<sub>1</sub> and C<sub>2</sub>) showed the same H<sub>2</sub>O-P (0.36 mg g<sup>-1</sup>) corresponding to  $\approx$ 5% of recovery while performed very different NaHCO<sub>3</sub>-P (2.13 vs. 0.63 mg g<sup>-1</sup>), thus showing 37 and 13% of labile-P. The alkali ex-

tractable P was in  $C_1$  and  $C_2$  at 0.60 and  $0.15\,mg\,g^{-1}$  (9 and 2%), while the HCl-P was the dominant fraction being detected at 2.43 and  $5.83 \text{ mg g}^{-1}$  (36 and 75%). Reference digestate BD had the highest  $H_2O-P$  (2.7 mgg<sup>-1</sup>) attaining also to the highest bicarbonate extractable P (NaHCO<sub>3</sub>-P,  $2.5 \text{ mg g}^{-1}$ ), thus showing 84% of labile-P. In BD NaOH-P was at  $0.8 \text{ mg} \text{g}^{-1}$  (13%), being very low also the HCl-P  $(0.9 \text{ mg g}^{-1}; 15\%)$ . Oppositely the reference compost (MSWC) showed the lowest H<sub>2</sub>O-P and NaHCO<sub>3</sub>-P (0.16 and  $0.54 \text{ mg g}^{-1}$ ) attaining to 16% labile-P. In MSWC the NaOH-P was registered at  $0.58 \text{ mg g}^{-1}$  (14%), while HCl-P was at  $2.82 \text{ mg g}^{-1}$  thus representing 66% of the whole extractable-P. The residual P was negligible in all the compared products, being relevant only in MSWC (0.57 mgg<sup>-</sup> 14%). Amongst the Ads and composts the whole P fractionation (%) was in D<sub>1</sub>: HCl-P (37)>NaHCO<sub>3</sub>-P (34)>NaOH-P (9)>H<sub>2</sub>O-P (8); D<sub>2</sub>; HCl-P (68)>NaHCO<sub>3</sub>-P (12)=H<sub>2</sub>O-P (12)>NaOH-P (2); C<sub>1</sub>: HCl-P (36)>NaHCO<sub>3</sub>-P (32)>NaOH-P (9)>H<sub>2</sub>O-P (5); C<sub>2</sub>: HCl-P (75)>NaHCO<sub>3</sub>-P (8)>H<sub>2</sub>O-P (5)>NaOH-P (2); BD: NaHCO<sub>3</sub>-P (44)>H<sub>2</sub>O-P (40)>HCl-P (15)>NaOH-P (13); MSWC: HCl-P (66)>NaOH-P (14)>NaHCO<sub>3</sub>-P (13)>H<sub>2</sub>O-P (4).

#### 3.3. Soil Olsen-P

As reported in Fig. 2, the Olsen-P in control soil (Ctrl) was detected at the lower level throughout the incubation ( $\approx 5 \text{ mg kg}^{-1}$ ), being the reference compost (MSWC) in the same range. Amongst the organic products the digestates and compost from wet process (D<sub>1</sub> and C<sub>1</sub>) attained the best since the beginning ( $\approx 15 \text{ mg kg}^{-1}$ ), thus showing a common reduction down to about 11-13 mg kg<sup>-1</sup> after the second sampling (day 14), performing fairly constant to the end. The products from the dry-batch process (D2 and C2) performed the worst Olsen-P from the beginning  $(7-9 \text{ mg kg}^{-1})$ , these showing a similar reduction down to 7mgkg<sup>-1</sup> after the second week and performing constant value to the end (day 84). The chemical P source and the reference digestate (P-chem and BD) showed higher Olsen-P since the beginning (20 and 23 mg kg<sup>-1</sup>) performing a rapid decrease within two-three weeks, thus behaving like wet process products in the following incubation stages. The RPE (%) at day 0 generally confirmed the Olsen-P outcomes: BD  $(128) > D_1 = C_1 (71) > D_2 (36) > C_2 (18) > MSWC (11).$ Throughout the incubation D<sub>1</sub> and C<sub>1</sub> showed constant RPE increase up to 142 and 102% in the end (day 84), BD showed constant decrease in the same timeframe down to 53% at day 84. Oppositely  $D_2$  and  $C_2$ showed fairly constant RPE during the soil test, these ranging at about 30% (on average). MSWC showed the worst in this context, decreasing down to -35%. As reported in Fig. 3 the relationship between the Olsen-P determined in soil at the beginning of the incubation (day 0) and the theoretical amount of labile-P (from SCE) added with the different organic products is described by a linear relationship of the type Olsen-P=1.1374 Labile-P - 3.9734 (R<sup>2</sup>=0.83\*).

# 3.4. Soil CO<sub>2</sub> emission

As reported in Fig. 4, the control soil performed low CO<sub>2</sub> instantaneous emission throughout the incubation test, attaining about 6 mg CO<sub>2</sub> kg<sup>-1</sup> soil h<sup>-1</sup> on average. In this frame, amongst the organic products the anaerobic digestates D<sub>1</sub> and D<sub>2</sub> showed the greatest CO<sub>2</sub> emission since the beginning of the soil test, doubling the control soil during the first days of incubation, and performing in the same range as reference digestate (BD  $\approx$  13 mg CO<sub>2</sub> kg<sup>-1</sup> soil h<sup>-1</sup>, on average). Later, all the products from anaerobic digestion (D<sub>1</sub>, D<sub>2</sub>, BD) showed reduced CO<sub>2</sub> emission attaining background level within 2–3 weeks (Fig. 4). In this framework, at the beginning of the incubation the investigated composts attained a very different  $CO_2$  emission rate:  $C_1$  showed the lowest while  $C_2$  the highest emission rate (6 vs. 10 mg  $CO_2$  kg<sup>-1</sup> soil h<sup>-1</sup>), being the reference compost intermediate (8 mg  $CO_2$  kg<sup>-1</sup> soil h<sup>-1</sup>). The total net  $CO_2$  emission (Fig. 4) amongst anaerobic digestates exhibits  $D_2$  at the highest total  $CO_2$  emission, 2-folds higher than  $D_1$  (3800 vs. 1300 mg kg<sup>-1</sup> soil), being BD intermediate (1700 mg kg<sup>-1</sup> soil). At the same time, composts performed lower total  $CO_2$  emission compared to digestates, in this frame  $C_1$  reached  $\approx$ 500 mg kg<sup>-1</sup> soil, while  $C_2$  was at about 700 mg kg<sup>-1</sup> soil, very close to the reference compost (MSWC: 780 mg kg<sup>-1</sup> soil).

As reported in Table 4, the carbon mineralization (%) outlined the following ranking:  $D_2(29) > D_1(21) = BD(20) > MSWC(11) = C_1$ (7)=C<sub>2</sub>(7). The C mineralization kinetics indicates that  $D_1$  attained a much faster C mineralization rate (k) than  $D_2$  (0.2093 vs. 0.0546 day<sup>-1</sup>), being BD intermediate (0.0847 day<sup>-1</sup>). C<sub>1</sub> and C<sub>2</sub> scored a similar k (0.0625 and 0.0455 day<sup>-1</sup>), resulting in a kinetics slower than the reference compost (MSWC, k=0.1419 day<sup>-1</sup>).

# 3.5. Plant test

The potted plant test showed that ryegrass dry biomass was affected by both fertilizer treatment (p < 0.05) and harvest (p < 0.05). As shown in Table 5, the best shoot dry weight was registered at the first harvest (day 28, 1.62 g pot<sup>-1</sup> on average). At the subsequent harvests, a progressive DW reduction was registered (at the respective day 56 and 84, 1.07 and 0.41 g pot<sup>-1</sup>, on average). More specifically, the unamended control (Ctrl) showed the worst DW throughout the pot test  $(0.81 \text{ g pot}^{-1})$ , on average), in the same range as the products from dry-batch digestion  $D_2$  and  $C_2$  (0.89 and 0.85 g pot<sup>-1</sup>, on the respective averages), which in turn were slightly lower than reference compost MSWC (0.92 g pot<sup>-1</sup>, on average). The products from wet digestion ( $D_1$  and  $C_1$ ) showed higher DW, attaining 1.11 and 1.10 g  $pot^{-1}$  (on average), in the same range of reference digestate BD (1.06 g pot<sup>-1</sup>). In this context the chemical fertilizer (NPK) showed the best performance attaining 1.54 g pot<sup>-1</sup> (on average). The cumulated biomass over 84 days showed similar pattern: Ctrl, D<sub>2</sub> and C<sub>2</sub> performed the worst, in the same range of MSWC ( $2.65 \text{ g pot}^{-1}$ , on average). At the same time D<sub>1</sub>, C<sub>1</sub> had higher dry biomass production in the same range of BD ( $3.26 \text{ g pot}^{-1}$ , on average). In this frame NPK showed the best results ( $4.61 \text{ g pot}^{-1}$ ). The root performed a different pattern: D<sub>2</sub> and C<sub>2</sub> attained the worst biomass, in the same range as MSWC  $(1.37 \text{ g pot}^{-1}, \text{ on average})$ , i.e., lower than  $D_1$ ,  $C_1$ , BD and Ctrl (1.75 g) $pot^{-1}$ ); the chemical reference performed the best (2.50 g pot^{-1}). The total (shoot+root) DW outlined the following ranking (g  $pot^{-1}$ ): NPK  $(7.12) > BD = C_1 = D_1 (5.02) > MSWC = Ctrl = D_2 = C_2 (4.05).$ 

The shoot N content was the highest at the first harvest at day 28 ( $35.4 \text{ mgg}^{-1}$ , on average), showing a steep decrease at the second harvest at day 56 ( $16.6 \text{ mgg}^{-1}$  on average), then flattening at the last harvest at day 84 ( $15.9 \text{ mgg}^{-1}$  on average). The chemical reference (NPK) performed the best from the first cut (day 28), showing the highest N content throughout the pot test ( $39.1 \text{ mgg}^{-1}$ , on average). Amongst the organic products, D<sub>1</sub> performed best since the first harvest (day 28), showing the highest N content throughout the pot test ( $23.1 \text{ mgg}^{-1}$ , on average), in the same range as BD ( $22.0 \text{ mgg}^{-1}$ , on average), D<sub>2</sub> and C<sub>1</sub> attained a lower shoot N content ( $20.1 \text{ mgg}^{-1}$ , on average), while C<sub>2</sub> performed worst, in the same range of the reference compost and unamended control ( $18.5 \text{ mgg}^{-1}$ , on average). The root N (at the final harvest at day 84) was the highest in MSWC ( $14.3 \text{ mgg}^{-1}$ ); all the investigated products (D<sub>1</sub>; D<sub>2</sub>; C<sub>1</sub> and C<sub>2</sub>) attained a similar N content ( $11.6 \text{ mgg}^{-1}$ , on average), in the same

range of BD and NPK  $(11.3 \text{ mg g}^{-1})$ , on average), while Ctrl showed the worst N content  $(9.3 \text{ mg g}^{-1})$ .

The shoot N uptake, was highest in NPK throughout the pot test (64.3 mg pot<sup>-1</sup>, on average), then followed the products from wet di-gestion  $D_1$  (30.1 mg pot<sup>-1</sup>, on average), and  $C_1$  (27.5 mg pot<sup>-1</sup>, on average) in the same range of reference digestate BD (28.1 mg pot<sup>-1</sup>, on average), while the products from dry-batch digestion (D<sub>2</sub> and C<sub>2</sub>) attained a lower N uptake (20.3 mg pot<sup>-1</sup>, on average), close to MSWC and Ctrl (19.5 mg pot<sup>-1</sup>, on average). The cumulative shoot N uptake over 84 days was best in NPK (193.0 mg pot<sup>-1</sup>), then followed  $D_1$  and  $C_1$  in the same range of BD (85.8 mg pot<sup>-1</sup>, on average).  $D_2$ and  $C_2$  exhibited the lowest ranking (55.4 mg pot<sup>-1</sup>), in the same range of MSWC and control soil  $(58.5 \text{ mg pot}^{-1})$ . The root N uptake was the best in NPK (28.9 mg pot<sup>-1</sup>), while all the investigated products and the two references attained similar results  $(18.3 \text{ mg g}^{-1})$ , on average), in the same range as the control soil  $(16.2 \text{ mg g}^{-1}, \text{ on average})$ . The total (shoot + root) N uptake portrayed the following ranking (mg pot<sup>-1</sup>): NPK (222)> $D_1 = C_1 = BD$  (105)> $D_2 = C_2 = MSWC = Ctrl$  (74). The apparent N recovery was highest in NPK since the first harvest at day 28 (ARF  $\approx$  35%), showing this treatment constantly increasingly ARF up to 75% at the end of the experiment at day 84. Amongst the studied products, D1 performed the best, showing 10.9% ARF at the first cut (day 28), performing a constant increase up to 16.8% and 17.4% at the second and third harvest (day 56 and 84). C1 attained a slightly lower ARF ranging from 8.8% to 13.4% all through the pot test, in the same range of BD. The other products (D<sub>2</sub>; C<sub>2</sub>) at the first harvest staged a negative ARF (-2% on average), in the same range of MSCW (-0.5). Later, D<sub>2</sub> partially recovered during the pot test a positive final ARF (+1.2%) performing like MSWC (+1.6%), C<sub>2</sub> was always negative (-3%, on average). The total (shoot + root) ARF (%) outlined the following ranking: NPK (77.5)> $D_1$ =BD (17.7) $\geq$ C<sub>1</sub>  $(15)>MSWC (3.6)>D_2 (1.2)>C_2 (-3.1).$ 

## 4. Discussion

## 4.1. Organic products

The organic products compared in this work showed important differences as a result of the different feedstock and process conditions. In this frame both anaerobic digestates from food waste (wet and dry-batch), showed similar carbon content attaining on the contrary very different N content: the wet digestate (D<sub>1</sub>) almost doubled the dry-batch one  $(D_2)$  (35 vs.  $16 \text{ mg N g}^{-1}$ ). As mentioned in the introduction, the wet and the dry-batch process require different feeding strategies to maintain the biological/chemical equilibrium during the digestion process. In this context the wet digestion fosters the utilization of 100% pulped food waste via the utilization of big amount of water to dilute potentially inhibitory substances such as ammonium (Chen et al., 2008). On the contrary, the reduced amount of water typically utilized for the dry-batch process requires limited N loading to the system (Chen et al., 2008). To this aim, a mixture of food waste and green waste (with higher C:N) was used in the dry-batch process reported in our experiment to prevent process imbalance (Fig. 1), and this is most likely the reason of the different N content and C:N ratio between wet and dry-batch digestate.

Beside a similar carbon content, both digestates attained also similar quality of their organic fraction thus showing similar stability level (OUR). Although there is not yet a general consensus about the threshold of the OUR to be safely used when digestates are supplied to agricultural soils, it is generally acknowledged that stable products have a lower potential  $CO_2$  emission, beside lower potential N immo-

bilization (Alburquerque et al., 2012a, 2012b; Iocoli et al., 2019). In this frame, the recent proposal appeared in the EU fertilizer regulation about the OUR threshold for anaerobic digestates to be directly used for agricultural purposes is  $\leq 25 \text{ mmol O}_2 \text{ kg}^{-1} \text{ VS h}^{-1}$  (EU, 2019). In this light, both digestates analyzed in this work appeared to be not safely applicable to the soil, thus showing high potential CO<sub>2</sub> emission and potential N immobilization as further discussed in the following sections.

In this context the aerobic stabilization process (composting) appears to be strategic attaining important increase of the stability level of composted products compared to the homologous digestates (Table 1). The OM mineralization during the composting process is widely reported in literature, as the result of the microbial degradation of the easily degradable OM, in turn determining the temperature increases of the composting mass thus resulting in a sanitation of the final product, and in a low potential CO<sub>2</sub> emission once added to the soil (Bernal et al. 1998; Grigatti et al., 2011). The overall effect of composting is generally a C:N reduction. In our work only the food-waste dry-batch digestate (D<sub>2</sub>) showed a moderate C:N reduction following the composting to C<sub>2</sub> (from  $\approx$ 19 to  $\approx$ 14), the final C:N however resulted not favorable to the N mineralization and to the plant N utilization as further discussed later. As previously reported, the different process (wet and dry-batch) implies the utilization of different mixtures (food waste/green waste), in addition the digestate from the wet process requires the utilization of a bulking agent to reach ideal composting condition. In this frame, the addition of green waste (25%) to  $D_1$  (Fig. 1) showed a slight increase of C:N from the final stage of digestion to the final stage of composting (from  $\approx 9$  to  $\approx 10$ ). However, in C<sub>1</sub> (from the wet process) the C:N was still in the range favorable to N mineralization and plant N uptake, as discussed in the following sections. The addition of a bulking agent for the successful composting of D<sub>1</sub> (from the wet digestion process) affected also the P content which was reduced by 30% in the homologous compost (C<sub>1</sub>); on the contrary, the composting of D<sub>2</sub> (dry-batch) without the addition of a bulking agent resulted in a P increase (+50%). Similar was the effect on the heavy metals content that, nevertheless, remained within the normal range for this type of products (Kupper et al., 2014; Grigatti et al., 2015).

#### 4.2. Phosphorus speciation

Several studies have addressed P fractionation from organic waste such as sewage sludge, bio-solids, animal manures and slurries (Brod et al., 2015; Kahiluoto et al., 2015; Bachmann et al., 2016), while only a few have provided information about anaerobic digestates from food waste and about their composts, especially in light of their potential and actual soil behavior (García-Albacete et al., 2012; Grigatti et al., 2015). In this work, we opted for the Dou et al.'s (2000) protocol, which is designed for the investigation of P release from organic waste. The SCE technique allows the labile P (H<sub>2</sub>O+NaHCO<sub>3</sub>-P) to be described, in addition to the partially available P (NaOH-P) and the scarcely soluble P (HCl-P). Based on Liu et al.'s (2014) description, labile P (water-extractable free P) is generally ascribed to inorganic forms (mainly orthophosphate), whereas the bicarbonate-extractable P is acknowledged to be formed by mixed organic/inorganic forms. The labile P is univocally considered to be easily available for plants, whereas the NaOH-P that comprises the P forms involved in Fe- and Al-P (hydro-)oxide complexes is assumed to be available in the medium term (Hedley et al., 1982). Lastly, the scarcely soluble P that is extractable only in a strong acid (HCl) is recognized to be unavailable for plants as it is formed by insoluble Ca-P compounds in sub-alkaline soils, as that used in this work.

The products obtained in this work following the wet and dry-batch anaerobic digestion of food waste (D<sub>1</sub> and D<sub>2</sub>) performed very different labile P ( $\approx$ 40 vs.  $\approx$ 20%), showing at the same time a consistent contribution of the acid extractable P ( $\approx$ 40 and 70%). These outcomes are in agreement with the Ca:P ratio of the tested products, being this at 5 and 14 in the wet  $(D_1)$  and dry-batch digestate  $(D_2)$ , respectively. BD showed notably lower Ca:P ratio at <2. The occurrence of Ca-P aggregates with low solubility in manure compost and broiler litter with Ca:P>2 has already been observed in the literature (Frossard et al., 2002; Toor et al., 2005; Grigatti et al., 2015; Grigatti et al., 2017). In the digestates compared in this work, food waste was the main source of feeding (100% in wet- and 90% in dry-batch), while BD was obtained from energy crops (mainly) and animal slurry. The energy crops are generally recognized to show very low Ca:P (Grigatti et al., 2015), like animal slurries while green and food waste are generally found with higher Ca:P.

To a deeper insight, it appeared that the amount of labile P in BD was similar to that reported in the literature for similar products (Bachmann et al., 2016), while  $D_1$  showed generally higher P in the labile fraction, compared to what is reported in the literature for anaerobic effluents from urban wastewater, being at the same time in the same range of what reported for food waste anaerobic digestates by Tampio et al. (2016). The alkali extractable P was found in digestates in a similar range of what reported by Tampio et al. (2016) for similar products, being this detected at the higher level in the wet digestate  $(D_1)$  in comparison to the dry-batch one  $(D_2)$  (9 vs. 2%), showing the former slightly higher Fe and Al, being this the very likely reason of this fractionation. As previously said the NaOH-P is recognized to become available in the medium-long term, in this context D<sub>1</sub> performed slight and constant Olsen-P relative release in time (RPE), as further discussed in the soil section. In this framework the composting process differently affected the P fractionation from the wet and the dry-batch digestion path. The labile-P was reduced by 10 and 50% in compost ( $C_1$  and  $C_2$ ) with respect to their ingestates ( $D_1$  and  $D_2$ ), this decrease in labile-P was confirmed by the increased scarcely soluble P (HCl-P). This phenomenon was widely described in literature (Frossard et al., 2002; Eneji et al., 2004); in this case it appeared therefore relevant the Ca:P ratio of the different materials, performing C<sub>2</sub> a P fractionation very similar to that of the reference compost (MSWC). The reference compost (MSWC), showed modest labile P associated with a very high Ca:P ratio (as well as very high Fe:P and Al:P ratios). High ratios of Ca, Fe and Al with P are responsible for strong reductions of P extractability, in accordance with the findings reported in the literature for compost (Grigatti et al., 2015; Tampio et al., 2016; Grigatti et al., 2019).

# 4.3. Soil Olsen-P and RPE course

Assessing OW suitability to supply the requested amounts of nutrients (namely, N and P) at the right times with respect to plant peak demand is a fundamental premise for successful replacement (partial/total) of mineral fertilizers. Phosphorus may be subjected to many different fates once added to the soil (Khan and Joergensen, 2012; De La Fuente et al., 2013), as  $PO_4^{3-}$  rapidly fixed to soil components (e.g., CaCO<sub>3</sub> in calcareous soils) resulting in scarcely soluble compounds (i.e., Ca-phosphates) that are substantially unavailable for root uptake. OM mineralization (Houben et al., 2019) in association with the slow nutrient release from the medium available fraction (alkali-extractable P) described by Hedley et al. (1982) increases the level of plant available P form recycled organic waste (Grigatti et al., 2015; Kahiluoto et al., 2015; Mackay et al., 2017; Grigatti et al., 2019). During soil incubation, standard temperature and moisture

stress the P fixation/mineralization processes in order to better asseverate the progress of potentially available P from organic products under simulated field conditions. In this light, we chose for this experiment a slightly calcareous soil with very low Olsen-P in order to minimize the background soil status, as premise for the most reliable assessment of P availability from the investigated products. The Olsen protocol to determine plant potentially available P was specifically adopted because of the characteristics of the selected soil (pH and CaCO<sub>3</sub> content). In fact, in soils with a mildly alkaline pH, use of the Olsen method is normally advised (Watanabe and Olsen, 1965).

In this work a digestate from animal slurry and energy crop (BD), and a compost from food waste, sewage sludge and green waste (MSWC), were selected to be used as organic references, in addition to a soluble chemical P source (P-Chem) as inorganic reference. The two references combined (organic and inorganic) at different P solubility constituted valuable benchmarks for evaluating the performance of the investigated products in the P-fixation/mineralization test.

The compared products were added at the same P rate (30 mg P  $kg^{-1}$  soil); in this frame, the product from the wet digestion path,  $D_1$ and to a lesser extent C<sub>1</sub>, showed the best results in terms of Olsen-P from the beginning of the incubation attaining notably higher available P, in comparison to the control soil and to the reference compost, being lower only in comparison to BD and to the chemical P source (P-chem). On the contrary the products from the dry-batch digestion path (D<sub>2</sub> and C<sub>2</sub>) performed slightly higher only in comparison to the reference compost (MSWC), thus showing  $3-5 \text{ mg kg}^{-1}$  higher Olsen-P in comparison to the background from control soil. These outcomes were in agreement to the P fractionation results which outlined the products from the wet digestion path  $(D_1 \text{ and } C_1)$  as highly performing in terms of available-P (≈40%). Indeed the Olsen-P determined in soil at the beginning of the incubation was in agreement with the total amount of theoretical labile-P (obtained from SCE) added with the different organic products as reported in Fig. 3, being this described by a linear function of the type: Olsen-P=1.1374 Labile-P -3.9734 (R<sup>2</sup>=0.83\*). These outcomes are in agreement to what exposed by Grigatti et al. (2019), relating the theoretical labile-P added with compost to the Olsen-P in soil following the addition of various Ads and animal slurries, thus showing SCE can be a valuable tool to the study of potentially available P from recycled OW.

Soil test showed also that later on, most of the tested products performed notably high fixation within two weeks, although the different products featured slightly different outcomes. As previously said soil fixation is widely reported in literature following the utilization of both chemical and recycled organic P sources (De La Fuente et al., 2013; Grigatti et al., 2019). In this framework, the study of the relative P efficiency (RPE; % of P-Chem) from the different treatments that was proposed by Leytem and Westermann (2005), offers a valuable insight into the performance of P from the compared sources. In fact, at the beginning of the incubation (at day 0), the RPE (%) overall confirmed the Olsen-P data, i.e. BD attained the best result  $(128 \text{ mg kg}^{-1}) > D_1 = C_1 (71 \text{ mg kg}^{-1}) > D_2 (36 \text{ mg kg}^{-1}) > C_2 (18 \text{ mg kg}^{-1}) > MSWC (11 \text{ mg kg}^{-1})$ . These results are in general agreement with those obtained using pig and cattle slurry in a P-deficient soil by Leytem and Westermann (2005). Fewer is the information on this topic about food-waste anaerobic digestates and their composts, which makes the comparison arduous. The study of the relationship between the theoretical labile-P added with the recycled organic waste and the Olsen-P determined in soil showed that the different products underwent a moderate adsorption/fixation immediately after their addition to the soil used in this test. Besides, during

the whole incubation (84 days), the different organic products outlined different patterns. The RPE trend in time showed that the products from the wet digestion ( $D_1$  and  $C_1$ ) determined an almost linear increase in the available P (+90 and+50%, respectively, at the end of the incubation), thus performing a trend similar to that of P-chem (140–100%). This was in agreement with the SCE outcomes outlining that the products from the wet digestion path had higher alkali extractable P. As previously discussed, this fraction is recognized to become available in the medium-long term (Hedley et al., 1982).

### 4.4. Soil CO<sub>2</sub> emission

The rate of  $CO_2$  emissions from control soil was moderate throughout the soil test, this demonstrates that a suitable pre-incubation stage had occurred, resulting in a very low background emission throughout the incubation (84 days). In this context the  $CO_2$  emission from the soil treated with the organic products showed important differences: the compared anaerobic digestates (wet and dry-batch), showed the greatest emission rates from the beginning of the soil incubation, in the same range of BD. All these products ( $D_1$ ,  $D_2$  and BD) showed also appreciable  $CO_2$  emission in the later stages of incubation, up to the third-fourth week. Oppositely both the compost derived from digestates ( $C_1$  and  $C_2$ ) performed notably lower emissions, ranging close to the reference compost (Fig. 4).

These outcomes are in agreement with the results obtained via the respirometric assay showing that both anaerobic digestates still presented relevant amounts of easily degradable OM, thus resulting very unstable on the basis of the classification proposed by Veeken et al. (2007). In contrast to this, the tested composts resulted very stable (C<sub>1</sub>) and stable (C<sub>2</sub>), on the basis of the same classification (Veeken et al., 2007). Although there is no general agreement about the way of measuring the stability of digestates (OUR; Biochemical Oxygen Demand, Dynamic Respiration Index, residual biogas production), the results that we obtained are in agreement with the literature about digestates, thus showing intense CO<sub>2</sub> emission from soil treated with unstable products (Bernal et al., 2008; Alburquerque et al., 2012a, 2012b).

Beside the instantaneous emission, the cumulative  $CO_2$  emission can give a valuable insight to the potential agronomical and environmental risk related to the utilization of unstable products. In this frame, the digestate from the dry-batch process of food-waste (D<sub>2</sub>) showed the greatest net-CO<sub>2</sub> emissions ( $\approx$ 4000 mg CO<sub>2</sub> kg<sup>-1</sup> soil), doubling both the wet digestate (D<sub>1</sub>), and also the reference from animal slurries and energy crops (BD). The different organic products (digestates and compost) were added on the same nitrogen basis (300 kg of available N ha<sup>-1</sup>); in this frame the different N content reflected in different amounts of products to be supplied to the soil. As a result, D<sub>2</sub> supplied twice as much C as D<sub>1</sub>.

Despite the variable  $CO_2$  emission we registered in this work, these were in the range reported by Alburquerque et al. (2012a, 2012b), on a wide range of anaerobic digestates. At the same time, the results reported in this experiment were lower than those observed in a previous study (Grigatti et al., 2014), addressing a food-waste dry-batch digestate that exhibited a quite higher OUR (>150 mmol  $O_2$  kg<sup>-1</sup> VS h<sup>-1</sup>), than the analogous product in this experiment ( $D_2$ , OUR=64 mmol  $O_2$  kg<sup>-1</sup> VS h<sup>-1</sup>), in turn leading to higher  $CO_2$  emission.

The investigated composts performed whole  $CO_2$  emissions in the same range reported in the literature for this type of products (Bernal et al., 2008; Grigatti et al., 2014). As already discussed, few is the information about the behavior of food-waste digestates following their addition to the soil; therefore, also C mineralization is scarcely

known. In this work, C mineralization showed that food-waste digestates (D<sub>1</sub>; D<sub>2</sub>) were higher C mineralizing products (21 and 29%), close to BD (20%). The wet digestate  $(D_1)$  showed a greater easily degradable C pool compared to the dry-batch digestate  $(D_2)$ , as proved by the faster C mineralization k (0.2093 vs. 0.0546 day<sup>-1</sup>). BD showed an intermediate value  $(k=0.0847 \text{ day}^{-1})$ , close to that reported for a very similar product (cattle slurry + cattle manure + maize-oat silage) by Alburquerque et al. (2012a, 2012b). Composting flattened the C mineralization kinetics; in this frame both composts (C1 and C2) attained similar k values (0.0625 and 0.0455 day<sup>-1</sup>), thus showing lower easily degradable C pool in comparison to their feedstock and also to the reference compost (MSWC). It is so perceived that the anaerobic digestion of food waste followed by composting, beside intense reduction of the whole CO<sub>2</sub> emission, reduced also the C mineralization kinetics, which represents an indubitable advantage in view of carbon storage in soil following the utilization of this type of products.

# 4.5. Pot test

Conducting a pot test with Italian ryegrass is a widely diffused approach to assess the apparent nitrogen utilization efficiency from different types of recycled organic products (Gunnarsson et al., 2010; Grigatti et al., 2011; Tampio et al., 2016). It is a fast growing species with high N requests, ensuring intense exploitation of the added N, and providing a reliable description of the fertilizing potential of the tested products. At the same time, the multiple harvest technique allows the uptake kinetics to be described, and gives a valuable insight into nutrient release during a growing season. In the pot test we ran, the control soil performed poor biomass production throughout the experiment, showing low background fertility in the selected soil. Compared to this, the soil treated with chemical fertilizer (NPK) showed the best performance during the whole test (almost +75% dry biomass compared to the control soil), thus showing a non-limiting nutritional reference for the assessing the performance of organic products. In addition to this, the apparent N recovery from NPK reached  $\approx 70\%$  of applied N, implying very poor N loss via gaseous emission or leaching, as further discussed later.

As previously said in the introduction, there is an increasing interest about the fertilizing capacity from anaerobic digestates, in this frame deeply investigated resulted those from animal slurries and agro-industrial waste, being these traits still poorly addressed in food waste Ads and compost (Beggio et al., 2019). In this context some investigations reported the food-waste and compost potential fertilizing properties on the basis of their chemical traits (Tambone et al., 2010), while some other researchers demonstrated the fertilizing equivalence of Ads from animal slurries and agro-industrial waste with FW digestates (Beggio et al., 2019). However, these approaches are based on chemical-quantitative assessment, being the assessment of agronomical response and of the bio-available fraction of nutrient more descriptive for further field application of recycled OW (Tampio et al., 2016).

In this framework amongst the organic products studied in this work, the digestate and compost from wet digestion of food waste  $(D_1 \text{ and } C_1)$ , performed a biomass production in the same range as the digestate from the animal slurry and energy crop (BD), attaining the best DW from the beginning. In this context the products from the dry-batch digestion of food waste  $(D_2 \text{ and } C_2)$  staged the lowest DW from the beginning, showing at the same time intense reduction between the first and the second harvest (-45%), as observed in MSWC. These data are in agreement with the N content in the plant tissues (Table 5). The soil treated with the chemical fertilizer (NPK)

showed the highest nutrient content since the beginning ( $\approx 60 \text{ mg N g}^{-1}$ ), associated with the lowest reduction between the first and second harvest (-40%), and performing better than all the other products till the end (22 mg N g<sup>-1</sup>). Amongst the organic products, two groups are detectable: D<sub>1</sub> and C<sub>1</sub> were good performers, as BD (37 mg g<sup>-1</sup>); D<sub>2</sub> and C<sub>2</sub> were poor performers as MSWC, and slightly worse performers than the unfertilized control. These trends are in agreement with the C:N ratio of the products, showing that the products from the dry-batch digestion (D<sub>2</sub> and C<sub>2</sub>) had a C:N ratio unfavorable to N mineralization and subsequent plant utilization.

The data of apparent nitrogen utilization (N-ARF; Fig. 5) can give a further insight into the kinetics of N release from the compared products (Tampio et al., 2016). The circumstance that NPK attained very high N-ARF proves that the pot test was run under optimal condition (temperature, moisture, light) for maximum plant N utilization, minimizing N loss (leaching, gaseous emission). In this frame, the digestate from the wet process of food-waste (D1) achieved the best N-ARF form the beginning of the pot test (day 28), also performing a significant N-ARF increase between the first and second harvest (days 28-56; +35%), and a minor one between the second and third harvest (days 56-84; +3%). The cumulative N-ARF we registered about the D<sub>1</sub> were notably lower to what reported in literature for similar products tested on ryegrass by Tampio et al. (2016). These authors reported N-ARF ranging from 30 to 50%. On the basis of the data they reported (TS) they tested the whole digestate (liquid+solid). In our experiment the liquid fraction from the wet digestion was separated and sent to the wastewater treatment plant (Fig. 1). Composting did not dramatically reduce the apparent utilization efficiency of N from D<sub>1</sub>, reflecting in a moderate N-ARF reduction in the related compost C1 (from 17 to 13%). Despite  $D_1$  and  $C_1$  showed similar initial ARF values ( $\approx 10\%$ ), C1 featured an almost linear increase in time, as BD did. This indicates that the composting process reduced the easily accessible N without deeply affecting N utilization efficiency. Conversely composting stabilized the dry-batch digestate but did not improve the negative performance of D<sub>2</sub> due to the C:N ratio unfavorable to the N mineralization and further plant uptake.

#### 5. Conclusions

The wet and the dry-batch anaerobic digestates from food waste compared in this work showed different inherent characteristics (C:N; Ca:P), these reflecting the different feeding mixtures and process. Food-waste wet digestate was the only showing a C:N favorable to N mineralization and plant N utilization, in this framework the composting process slightly affected this feature. The different C:N ratio implied the utilization of different amounts of products to ensure the same amount of N potentially available for crops, this in turn affected the whole CO<sub>2</sub> emissions showing dry-batch digestate doubled the wet one. Composting strongly reduced these emissions from both digestates being the whole emissions in the same range of the reference compost. Soil test showed that composting slightly affected the potential P fertilizing capacity from both the wet and the dry-batch digestates, being this trait mainly driven by the Ca:P ratio of parental materials. In addition to this, pot test showed that only the compost from wet digestate behaved as amendment with partial potential substitution of N chemical fertilizer and limited CO<sub>2</sub> emission, conversely the compost from dry-batch digestate negatively performed. On this basis, we can conclude that composting is a reliable strategy for management of the anaerobic digestates from food waste thus reducing their potential CO<sub>2</sub> emission without affect their N- and P-fertilizing capacity. A more accurate monitoring of C:N ratio during the composting of dry-batch digestate in view of slightly lower values can probably increase their N fertilizing value.

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